INSPIRING INNOVATION | INNOVANTE PAR TRADITION

Ecole Nationale Supérieure des Mines SAINT-ETIENNE

Améliorer la résolution angulaire en EBSD

Pourquoi ? Comment ?

Claire MAURICE

Laboratoire Georges Friedel – UMR CNRS 5307

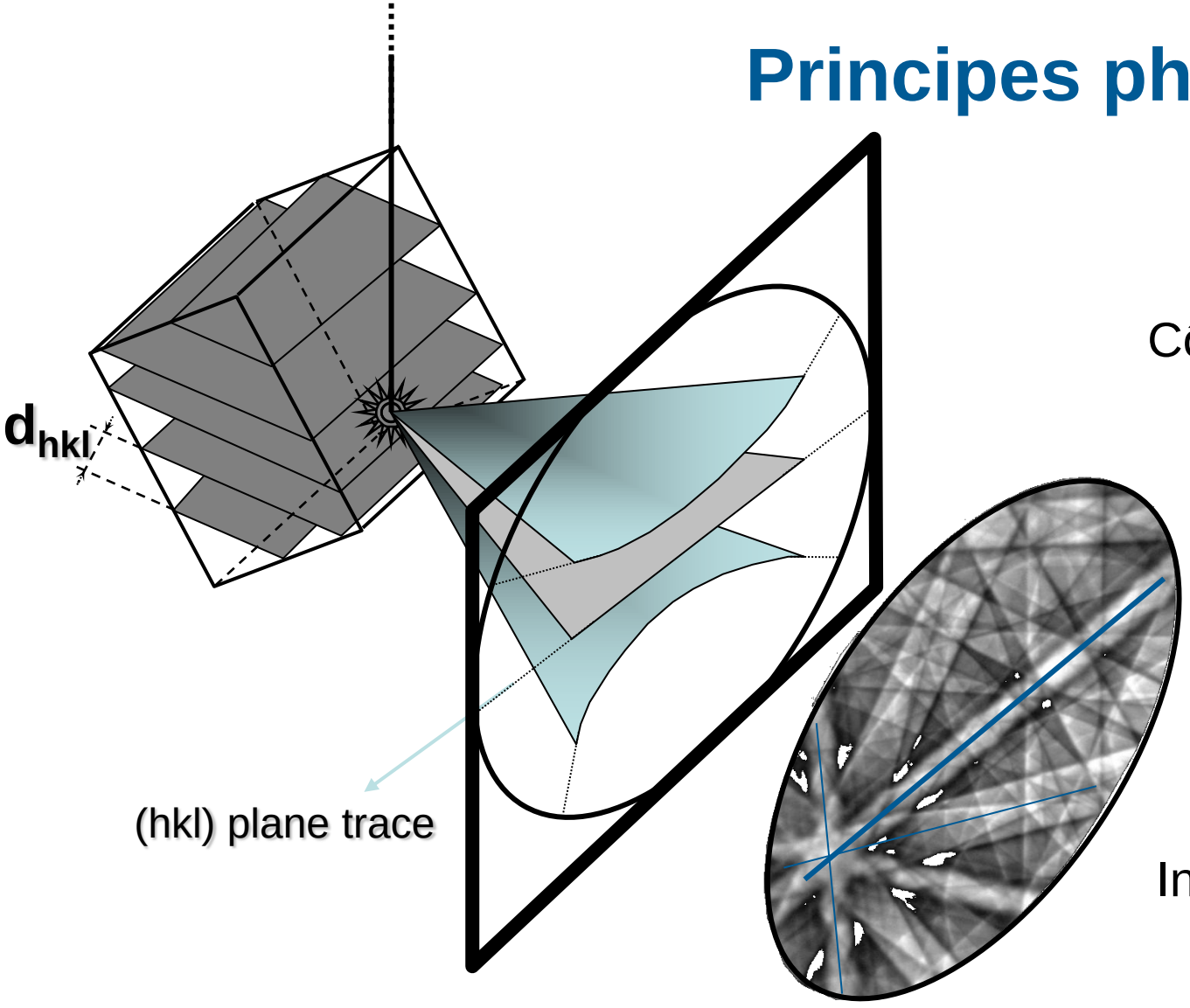
MINISTÈRE DE L'ÉCONOMIE DES FINANCES ET DE L'INDUSTRIE

SMS

INSTITUT CARNOT M.I.N.E.S

cnrs dépasser les frontières

Principes physiques de l'EBSD



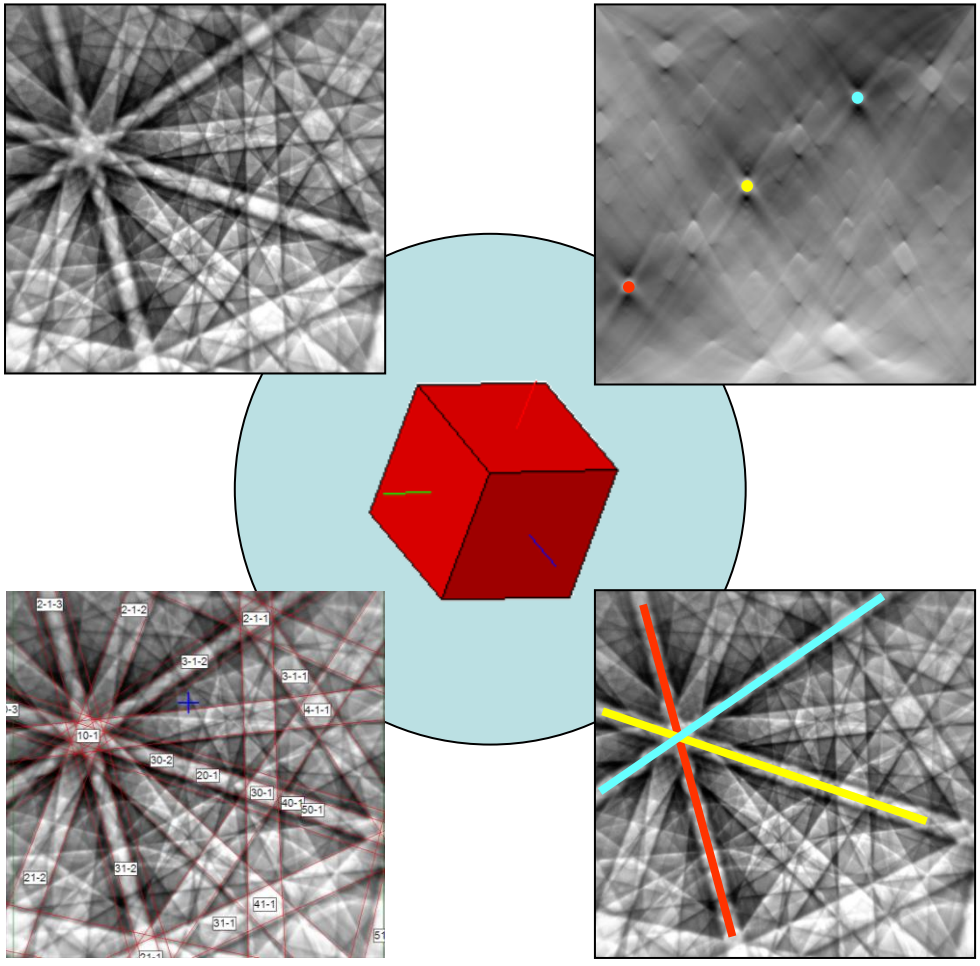
Projection gnomonique

Cônes de Kossel -> Hyperboles

1 Bande de Kikuchi
= 1 famille de plans cristallo.

Intersections
= Axes de Zone

Indexation Standard

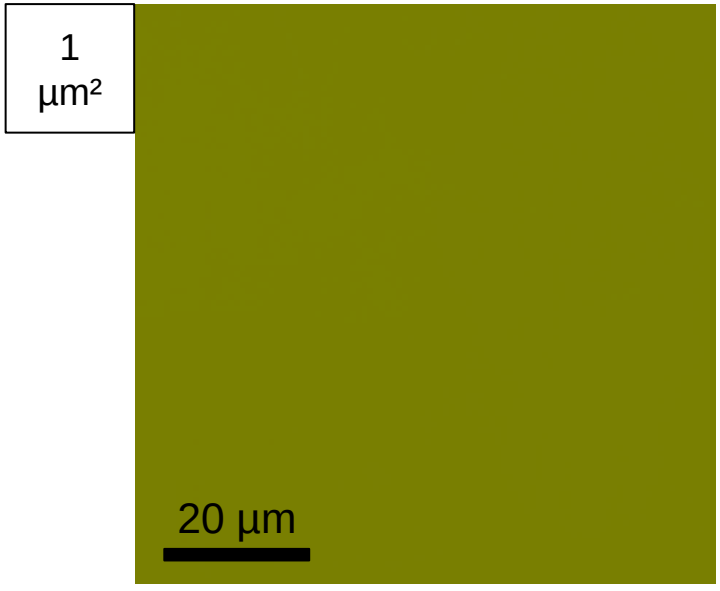


Performances

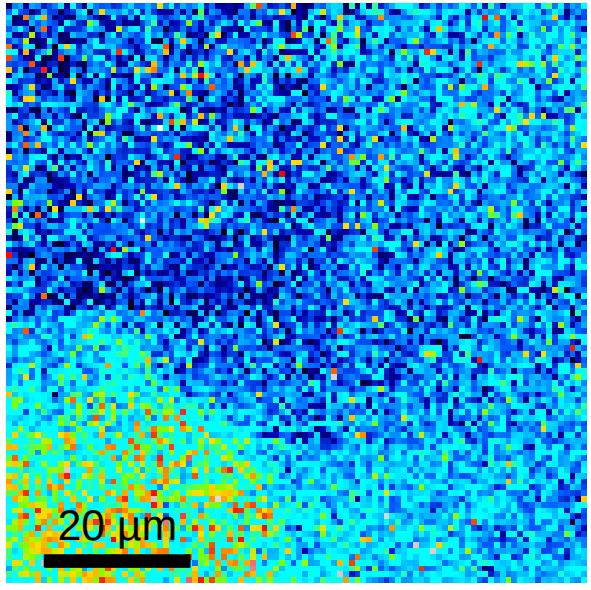
Angulaire *(relatif)*	0,1° - 0,5°	
Spatiale	W	FEG
Latérale (// axe de tilt)	*0,5 µm*	*20 nm*
Longitudinale	*x 3*	
Profondeur	qqs 10 nm	

Before we start …

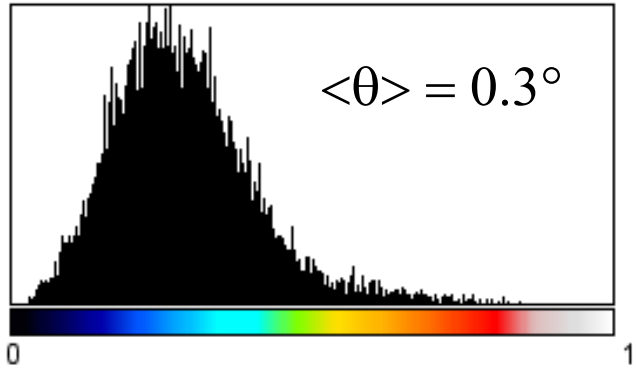
Nickel superalloy – Single Crystal



Euler angles



Deviation from mean orientation

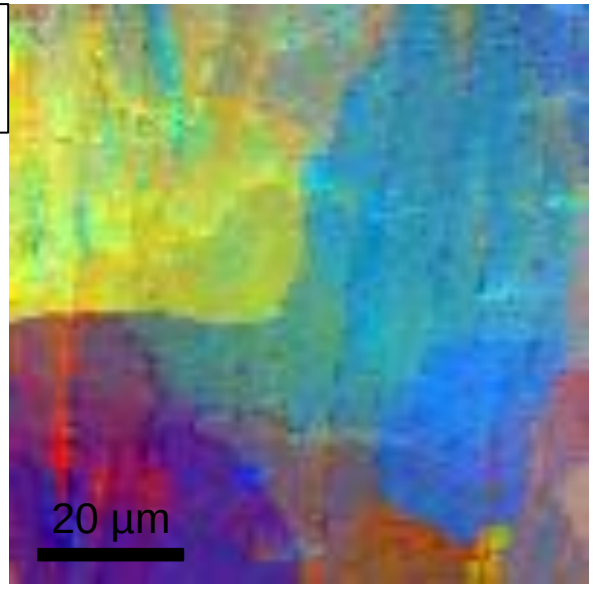


Not much to look at, except for the usual noise !

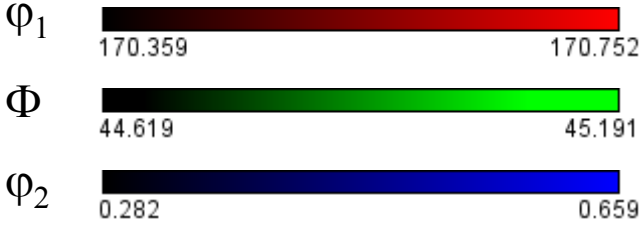
Before we start …

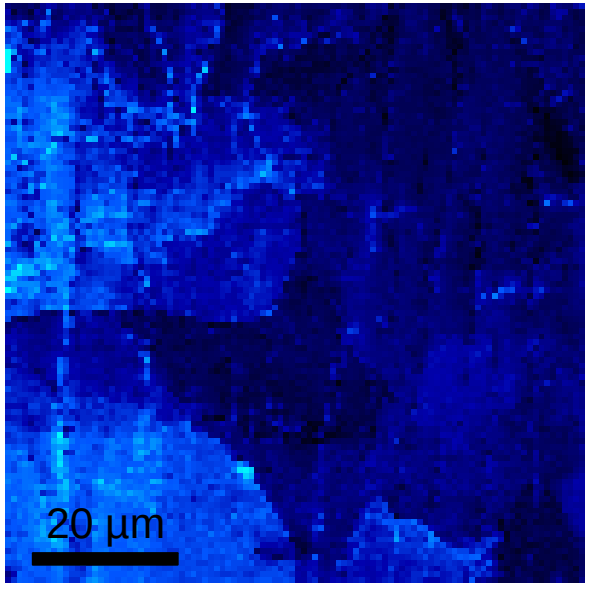
With HAR-EBSD

1 µm²

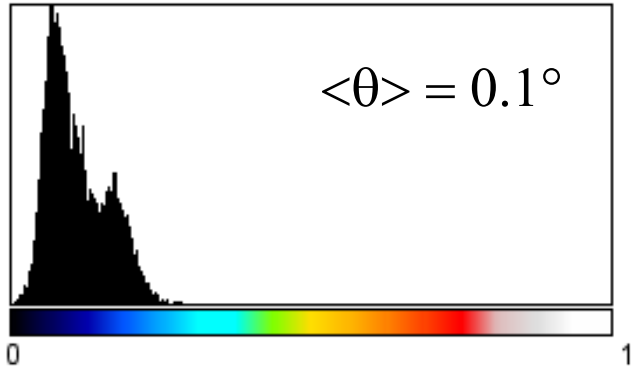


Enhanced Euler angles





Deviation from mean orientation

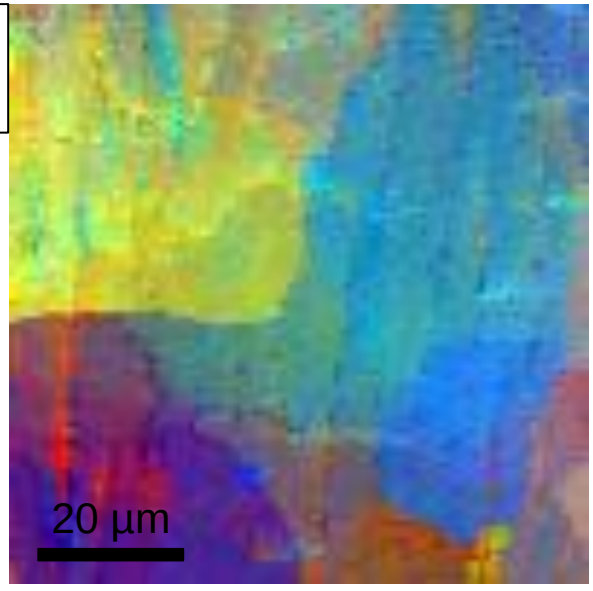


HAR-EBSD reveals slightly misoriented domains

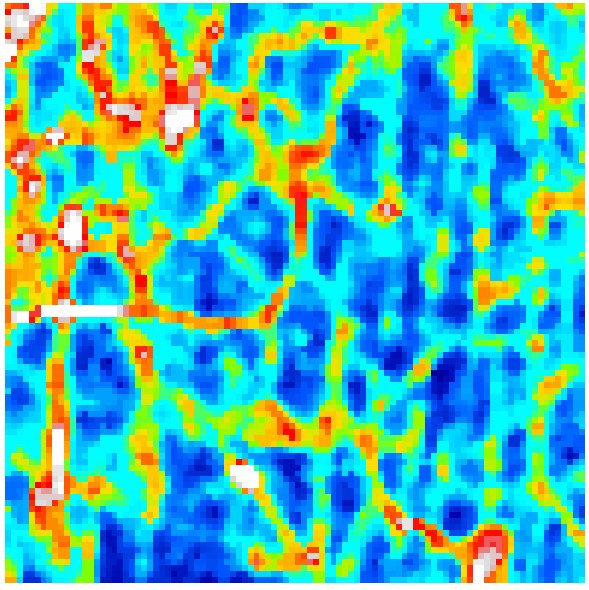
Before we start …

With HAR-EBSD

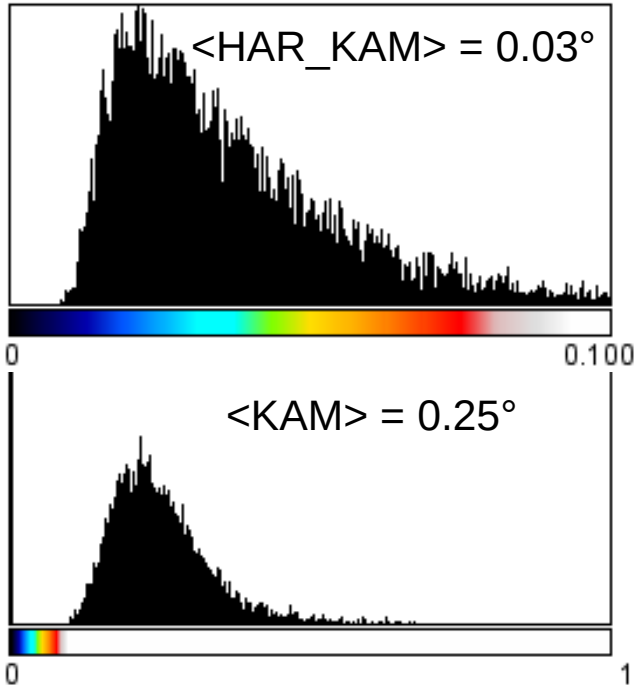
1 µm²

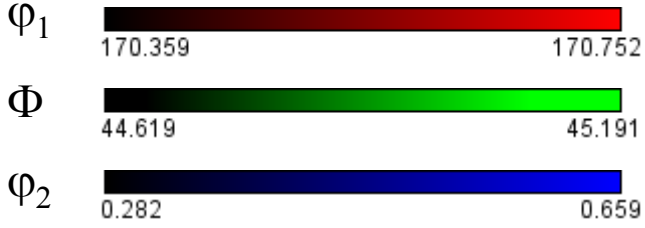


Enhanced Euler angles



Kernel Average Mis.





HAR-EBSD detects misorientations below 0.1° & improves rotation axis determination

Outline

1 – The principles of HAR-EBSD

2 – A few selected examples

elasticity – lattice defects – elasto-plastic polyX

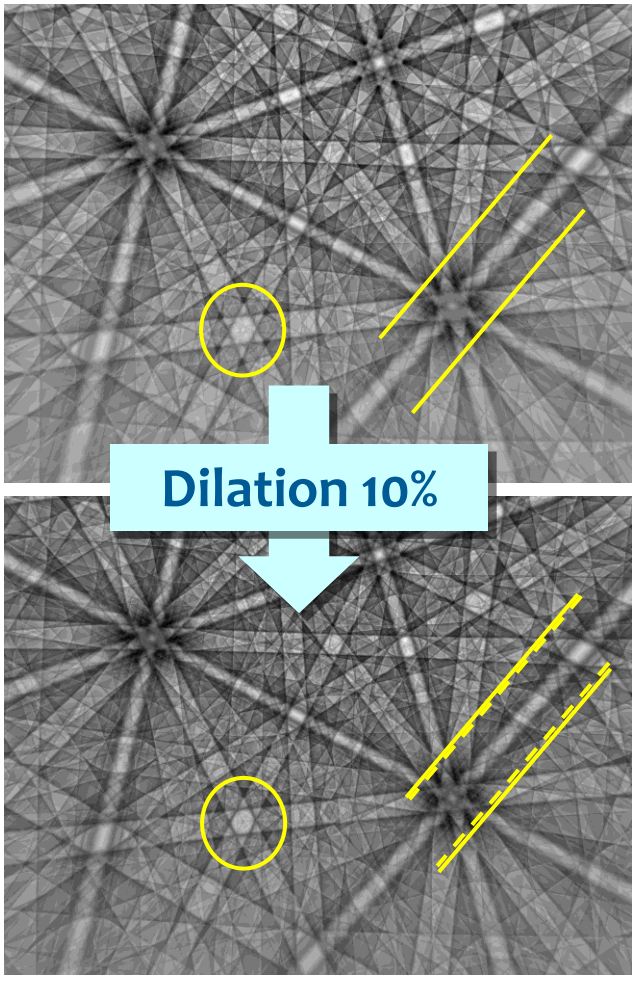
3 – Current Limitations : why not try 3D Hough ?

4 – Source Point and Elastic Strain

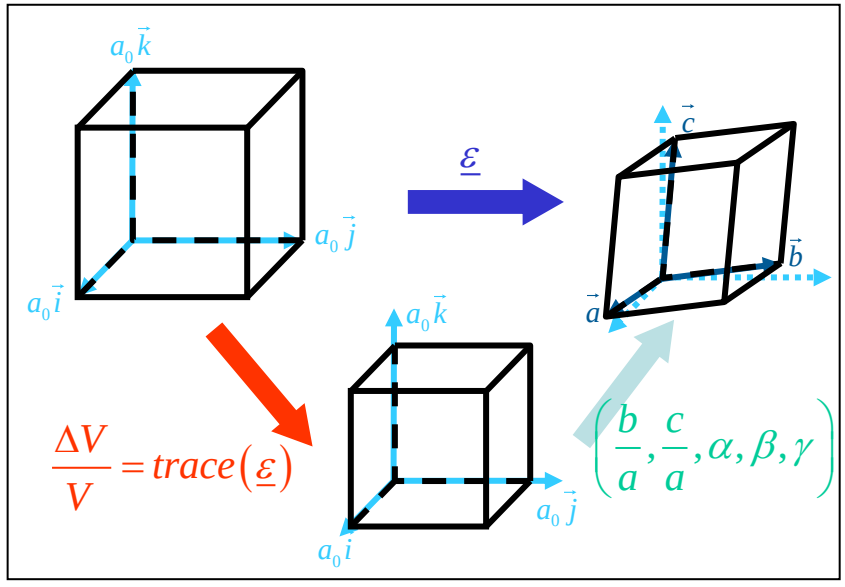
How to kill two birds with one stone ?

5 – Conclusions

Lattice elastic distortion and EBSPs



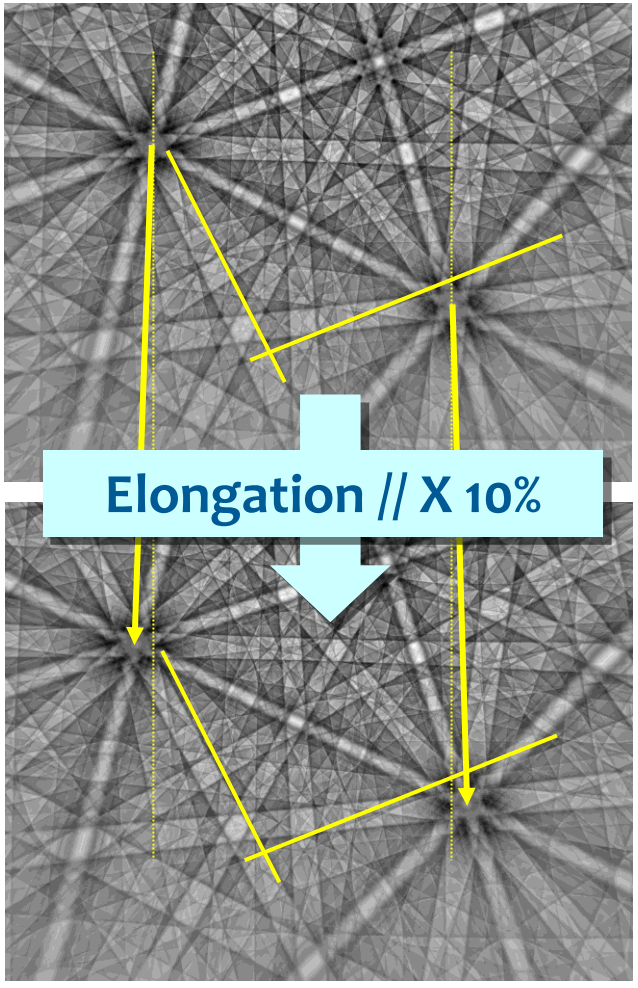
Dilation 10%



Band width change

Zone axes move

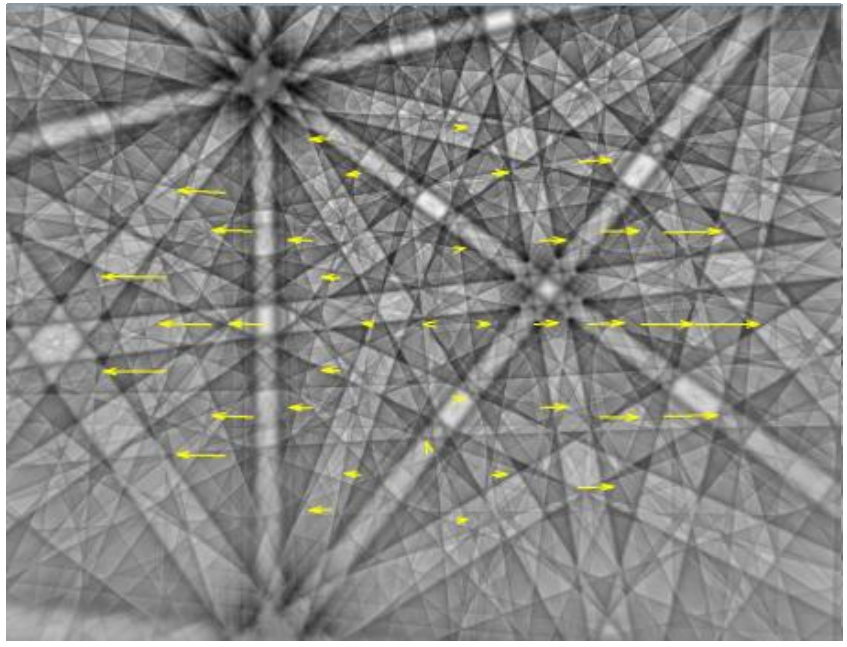
Interplanar angles change

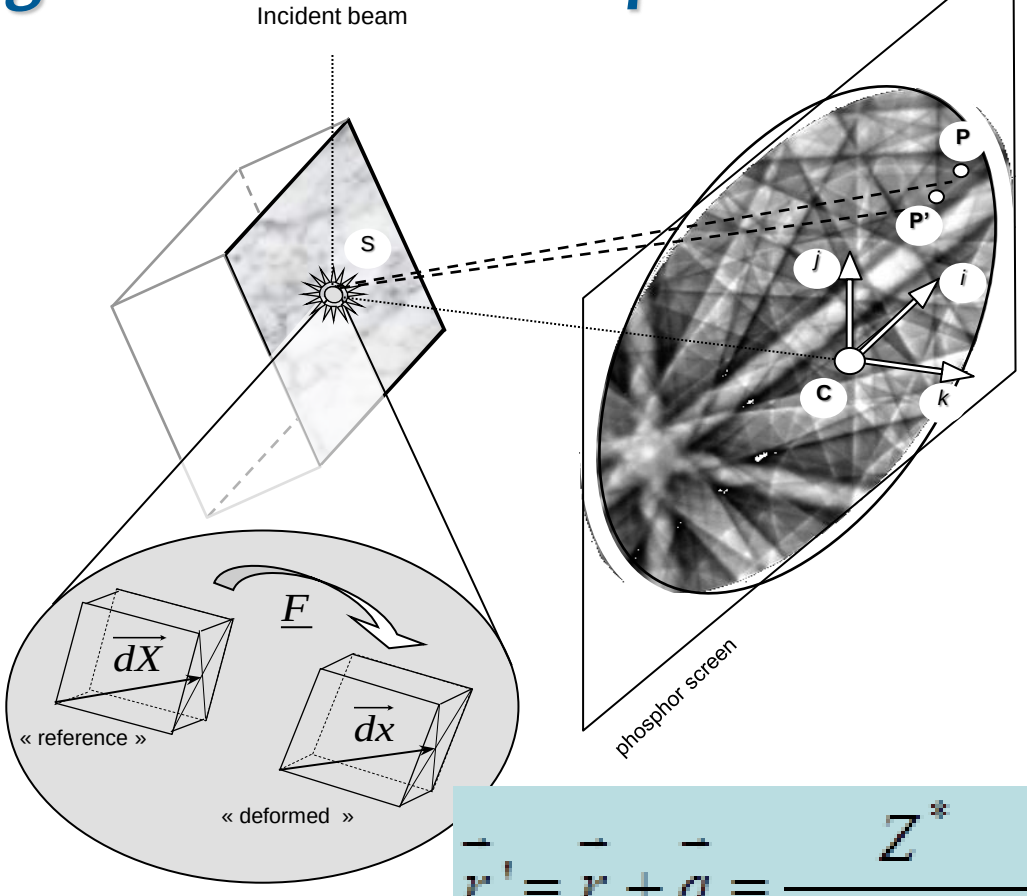


Elongation // X 10%

From shifts to strain

Link the displacement field between 2 EBSPs to the material deformation gradient between 2 probed volumes



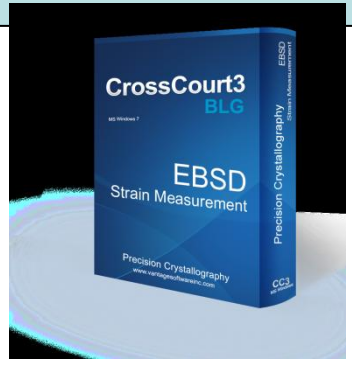


DIC used to measure shifts between 2 EBSPs

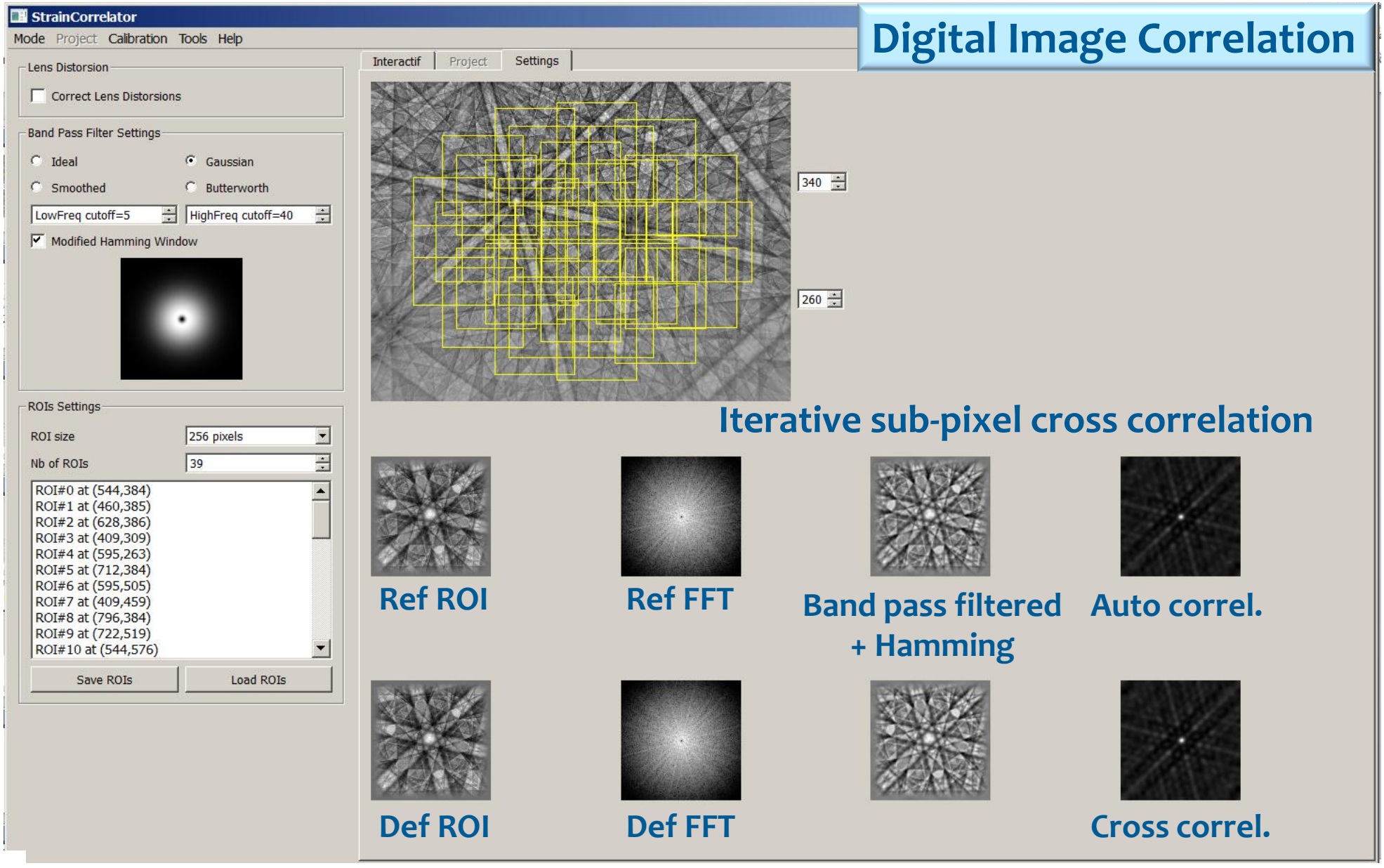
$$\vec{r}' = \vec{r} + \vec{q} = \frac{Z^*}{\left(\underline{F}\cdot\vec{r}\right)\cdot\vec{k}}\,\underline{F}\cdot\vec{r}$$

An historical perspective

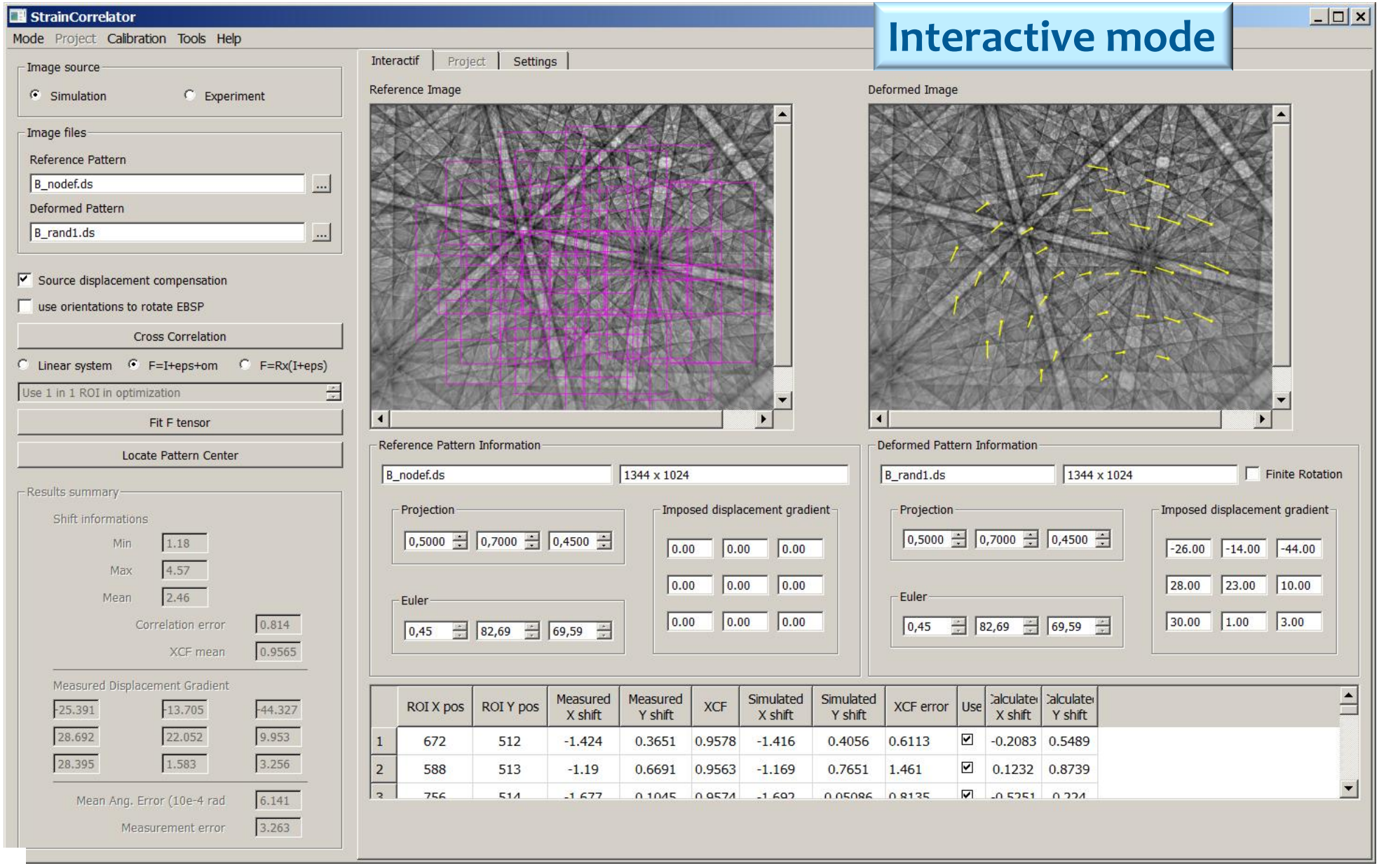
1993	Troost et al.	Microscale Elastic-Strain Determination by Backscatter Kikuchi Diffraction in the Scanning Electron-Microscope
1996	Wilkinson	Measurement of elastic strains and small lattice rotations using electron back scatter diffraction
2004	**Villert et al.**	**Début de thèse à Saint-Etienne (CEA – ST Micro)**
2006	Wilkinson et al.	High-resolution elastic strain measurement from electron backscatter diffraction patterns: New levels of sensitivity.
2007	Winkelmann et al.	Many-beam dynamical simulation of electron backscatter diffraction patterns.
2009	Villert et al.	Accuracy assessment of elastic strain measurement by EBSD.



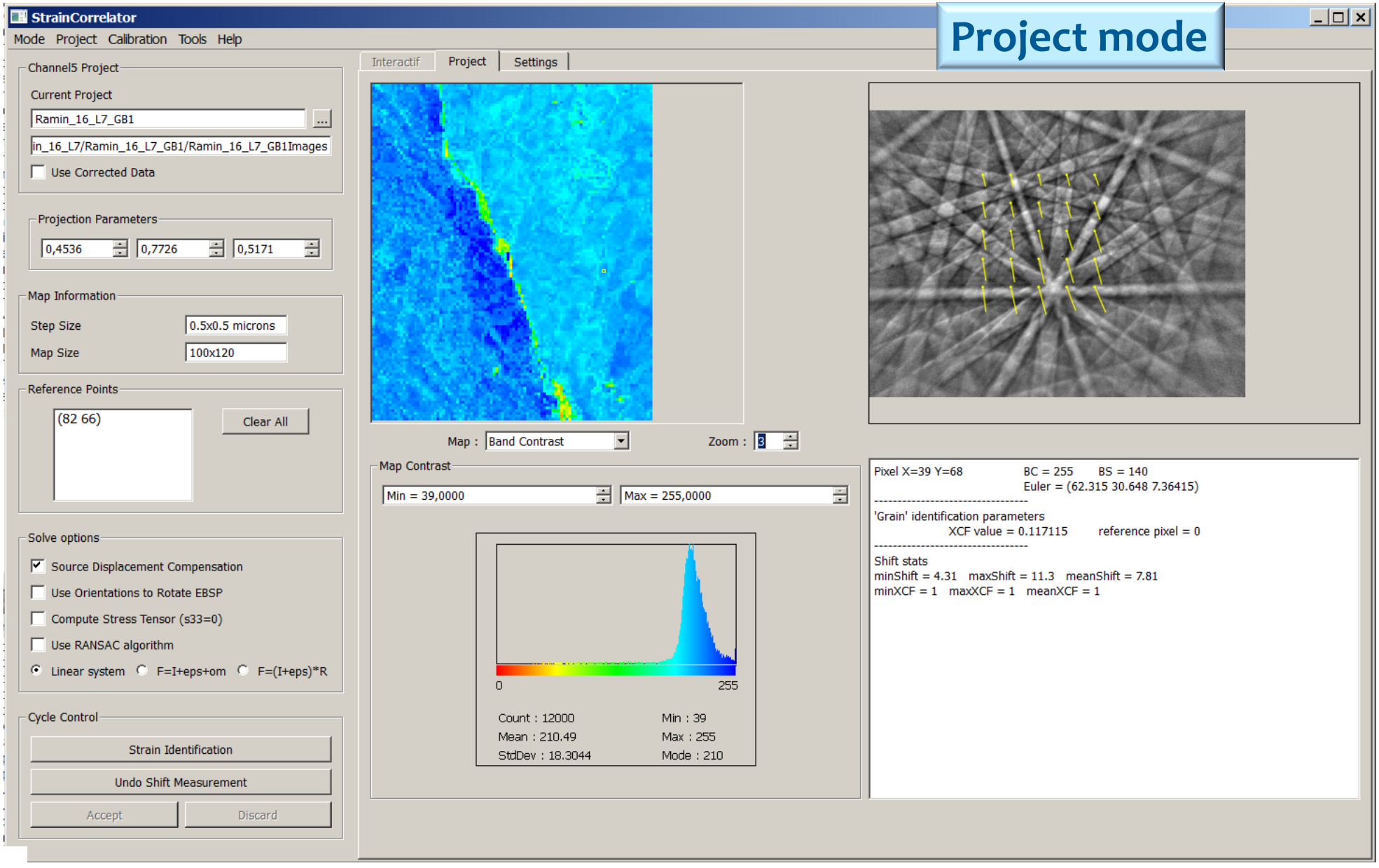
Snapshots of StrainCorrelator



Snapshots of StrainCorrelator



Snapshots of StrainCorrelator



Experimental protocol

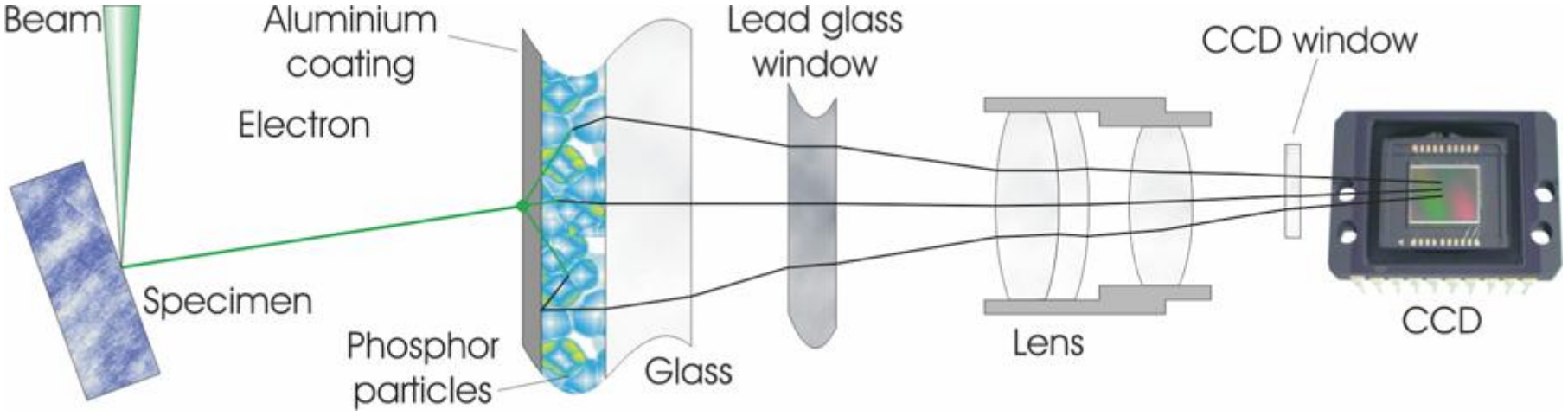
Calibration — **One time** *camera optics distortions*

Acquisition

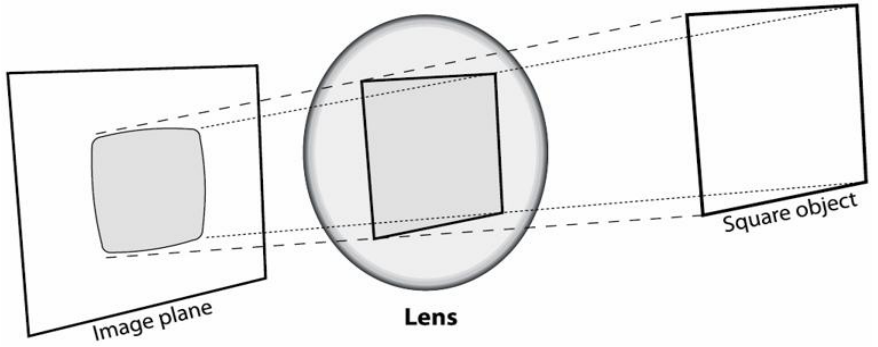
Analysis

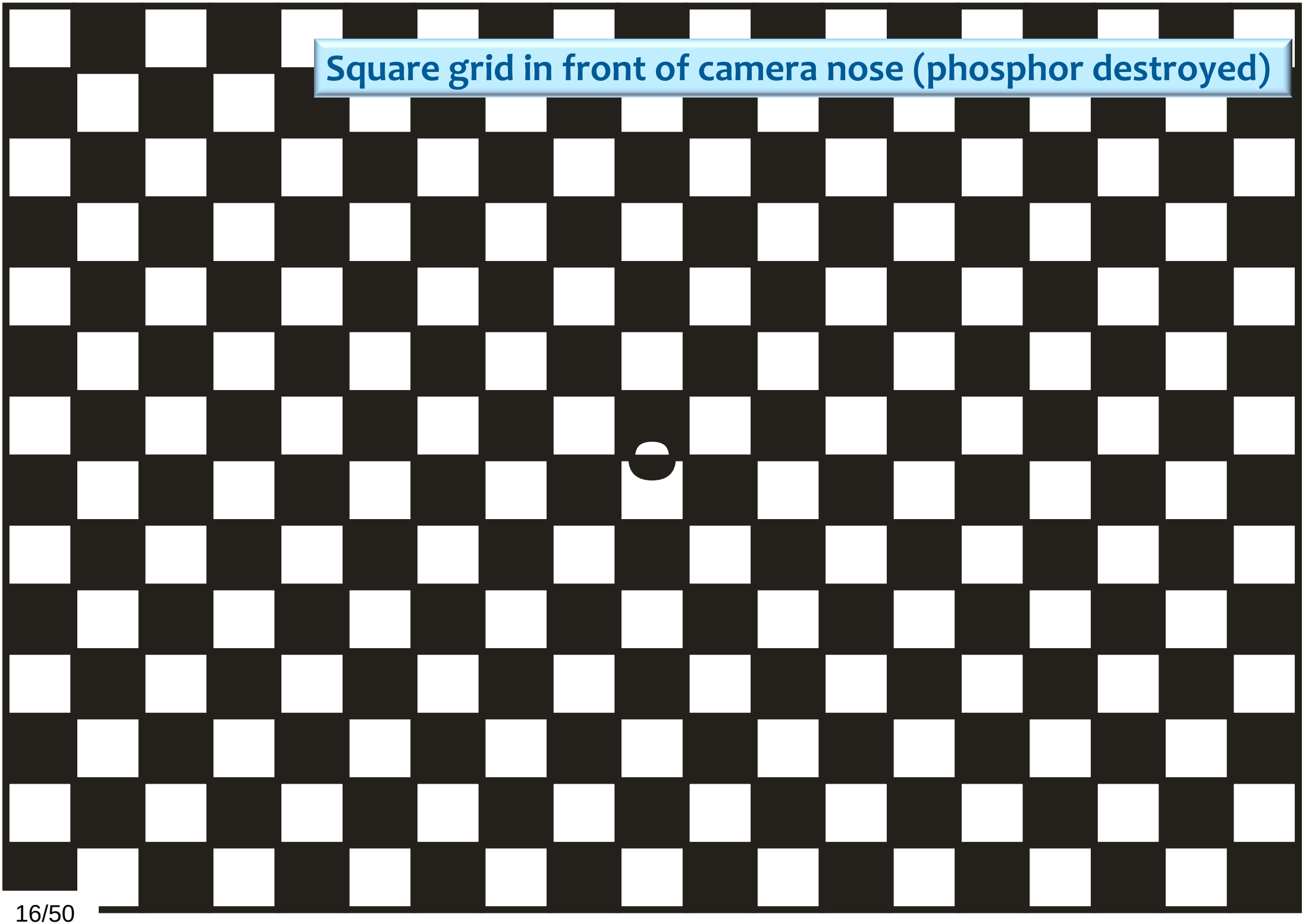
Experimental protocol

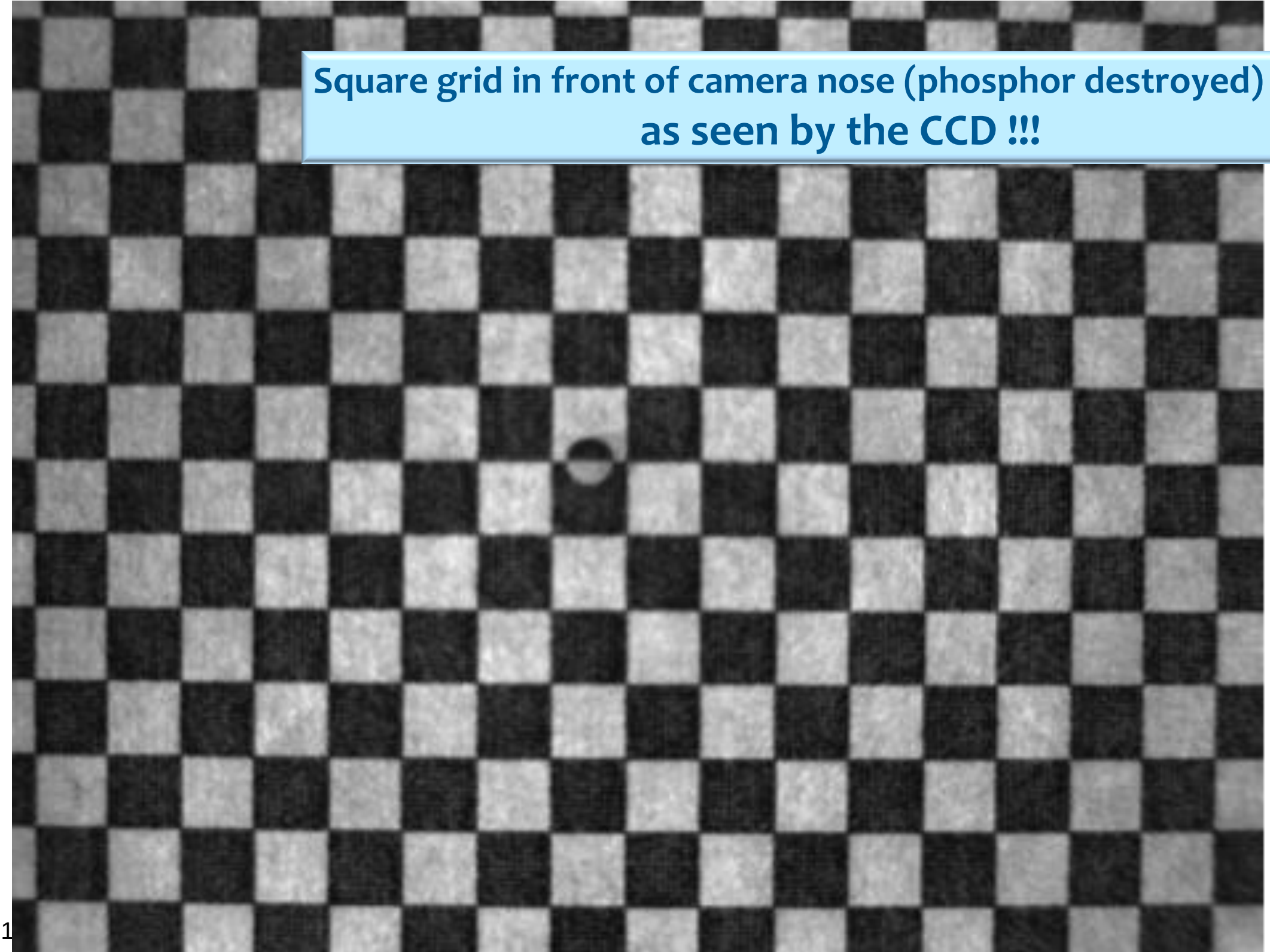
EBSP distortion



- Lens = Distortion
- CCD + Lens + Phosphor = Misalignment
- Present in all detectors
- Usually not seen by indexing

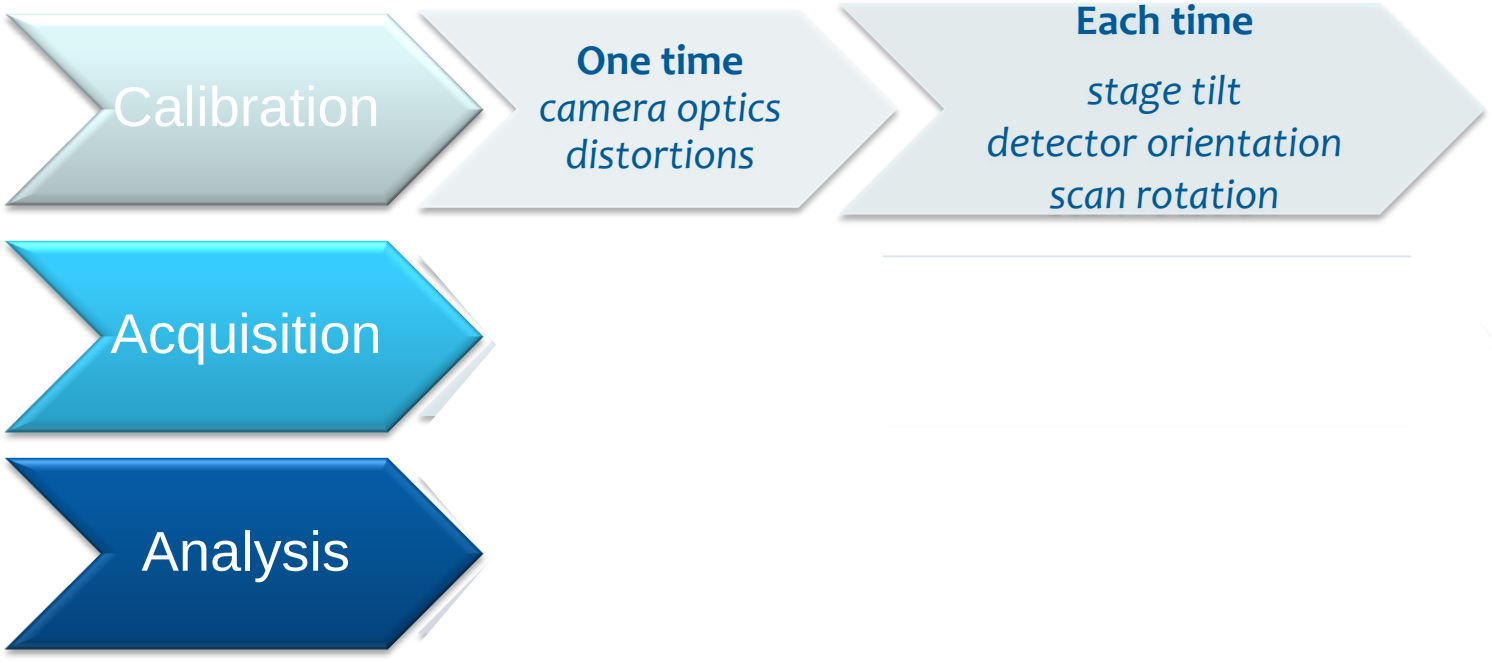



Square grid in front of camera nose (phosphor destroyed)


Square grid in front of camera nose (phosphor destroyed)
as seen by the CCD !!!


The good news : can be calibrated and corrected for…

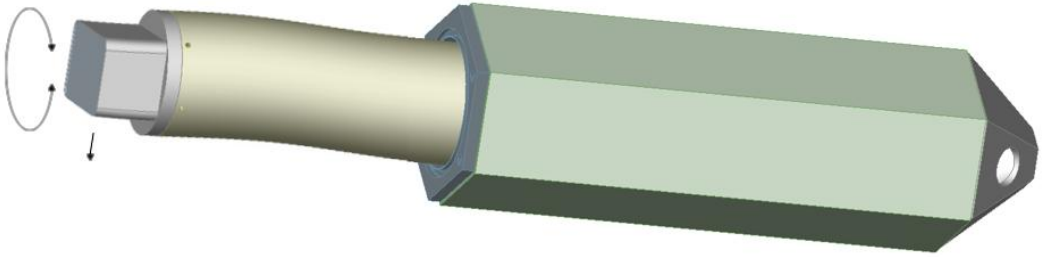
Experimental protocol

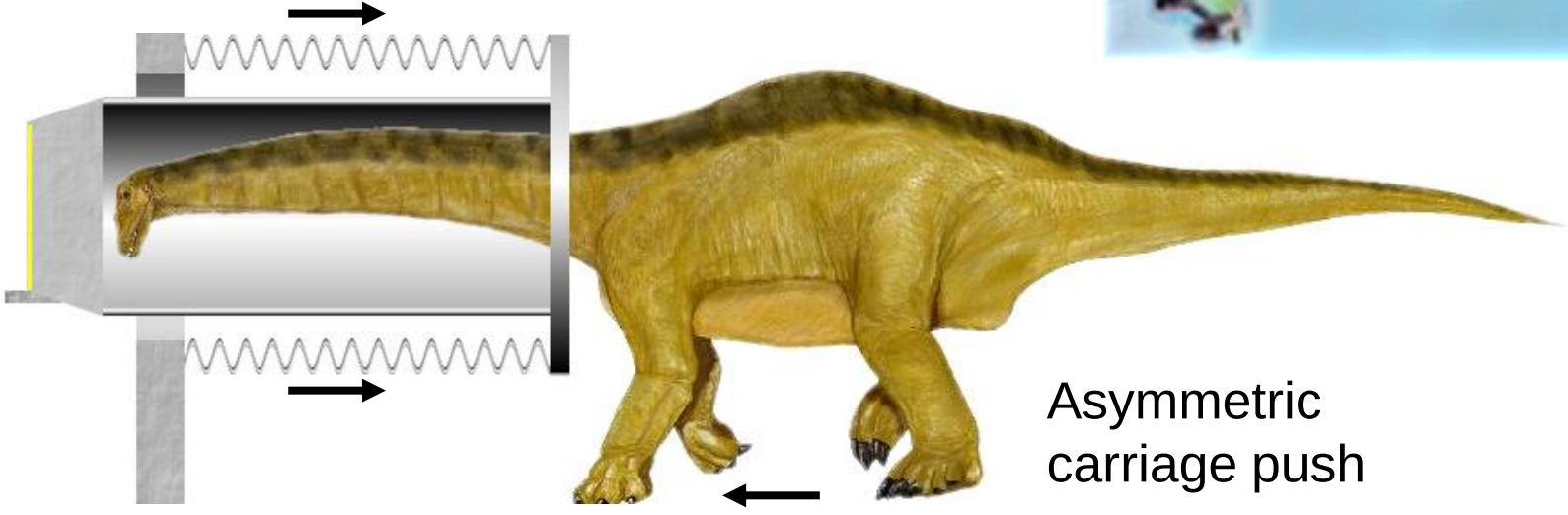


Experimental protocol

Mechanical issues (courtesy A.P. Day)

- Camera rotation & shift
- Droop, twist, boing & bounce





Asymmetric carriage push

Experimental protocol

Calibration	**One time** *camera optics distortions*	**Each time** *stage tilt detector orientation scan rotation*
Acquisition	**No binning (~1 Hz)**	**Store on hard disk (~1 Mo / per pattern)**
Analysis	**CPU intensive DIC on PC-cluster**	**GUI for shift analysis, data presentation…**

Experimental protocol

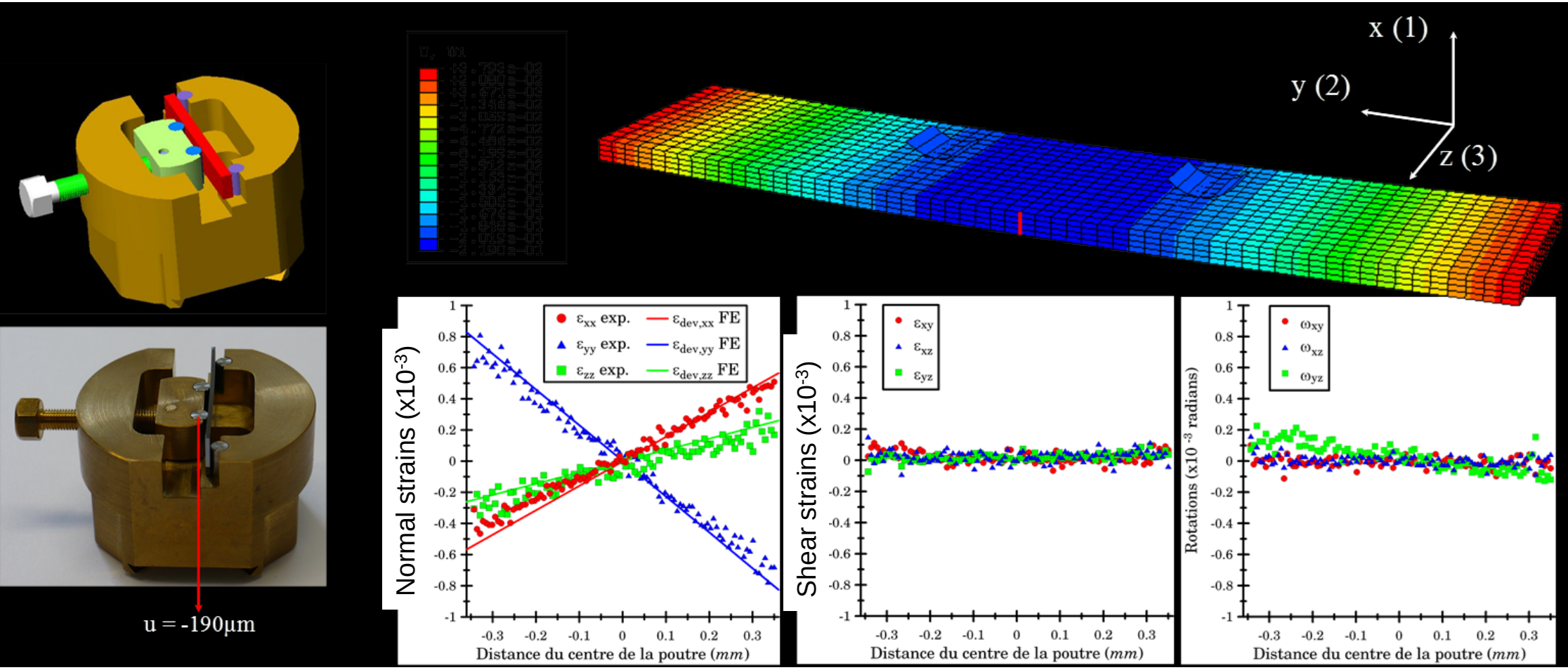
Calibration	**One time** *camera optics distortions*	**Each time** *stage tilt detector orientation scan rotation*
Acquisition	**No binning (~1 Hz)**	**Store on hard disk (~1 Mo / per pattern)**
Analysis	**CPU intensive DIC on PC-cluster**	**GUI for shift analysis, data presentation…**

Relatively small maps : 200x200 points = 50 Go !!!

More time required for data transfer than for (acquisition + analysis)

A few selected examples

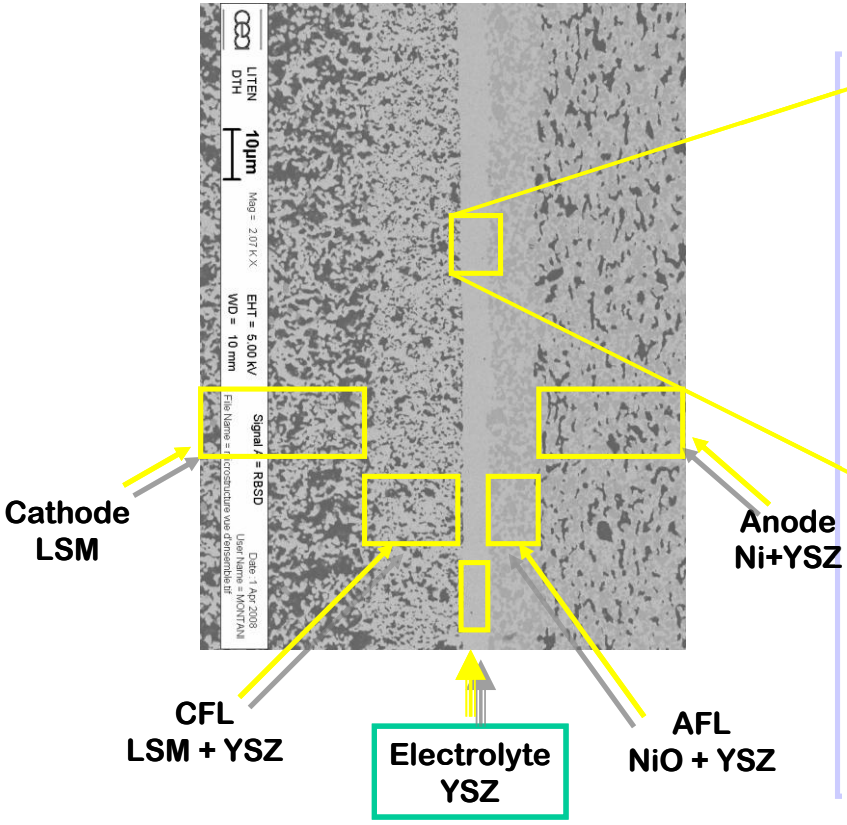
4 point bending of a Si single crystal

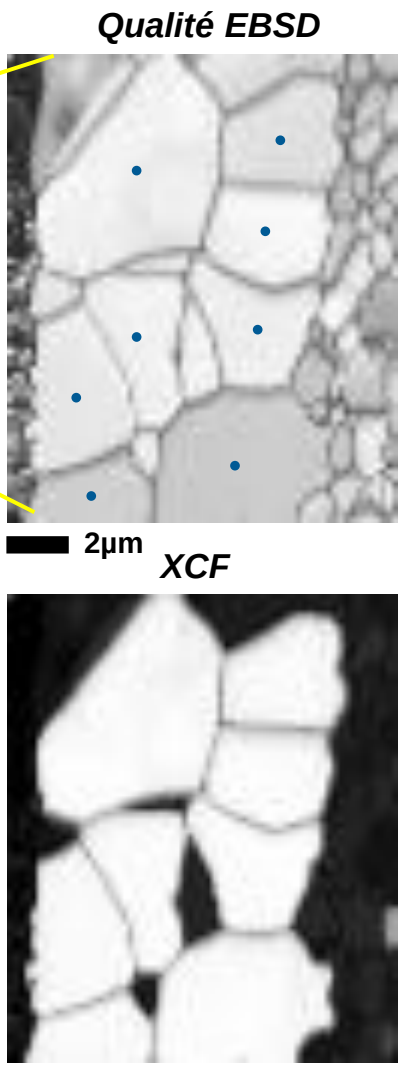


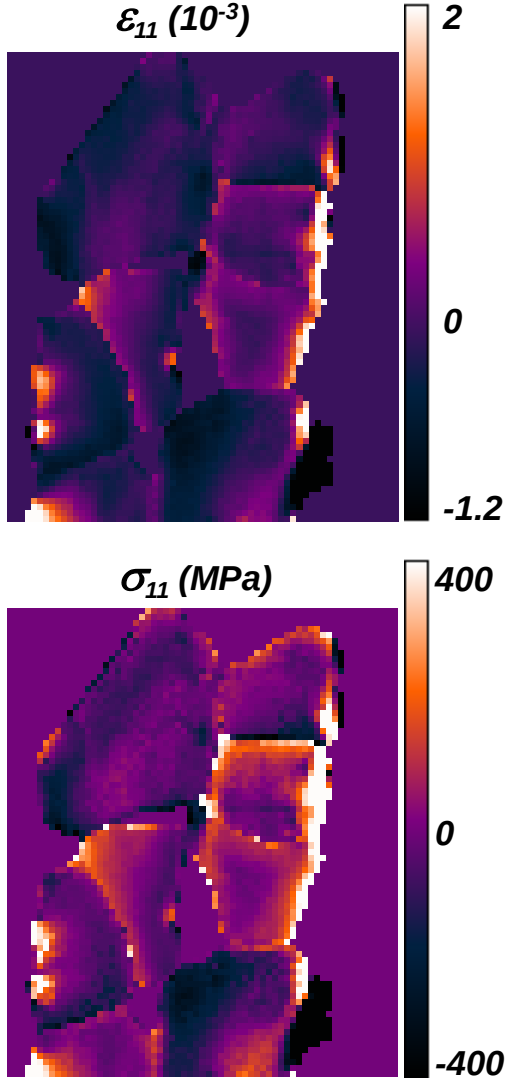
S. Villert, C. Maurice, C. Wyon and R. Fortunier,
Accuracy assessment of elastic strain measurement by EBSD, *Journal of Microscopy* 233 (2), pp 290-301 (2009)

A few selected examples

Residual strains in SOFC



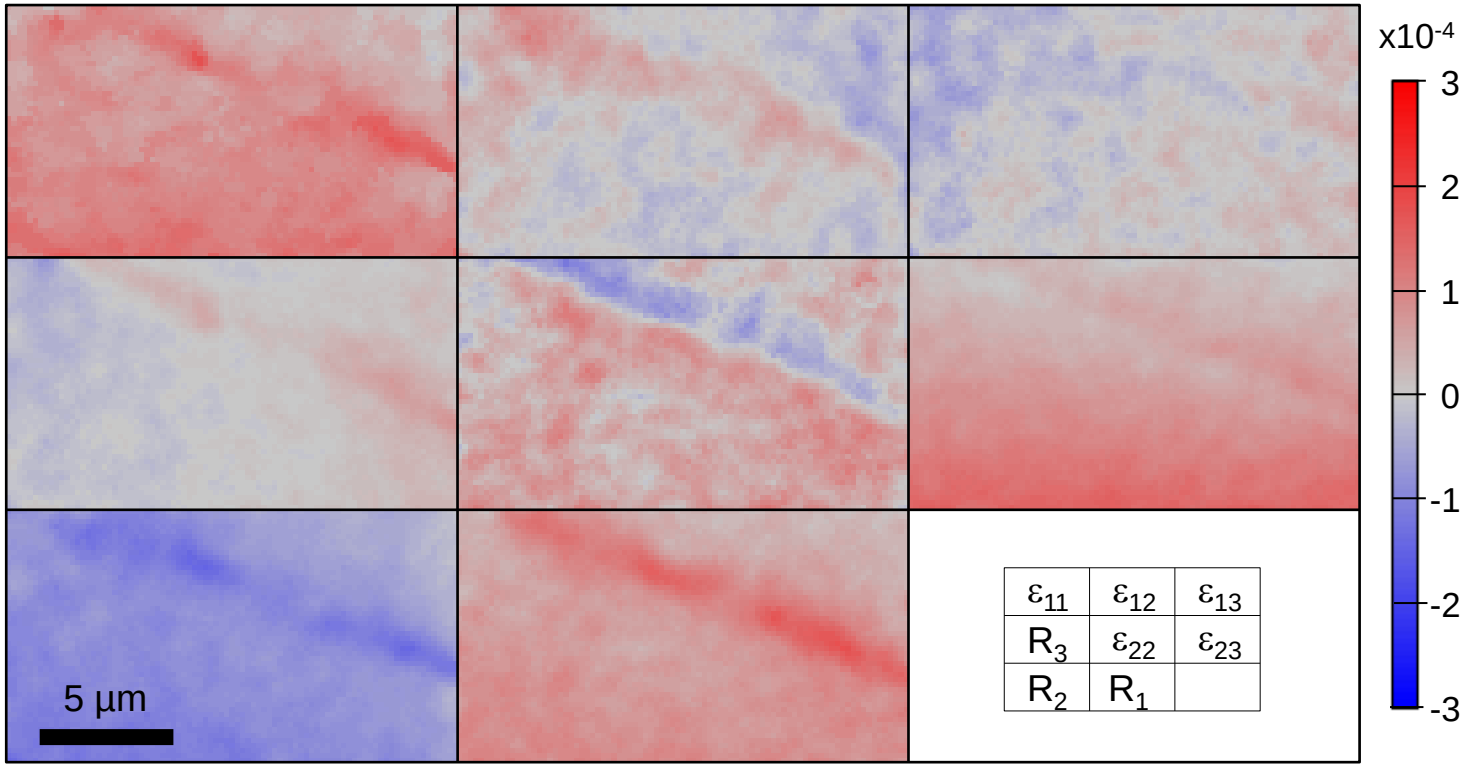




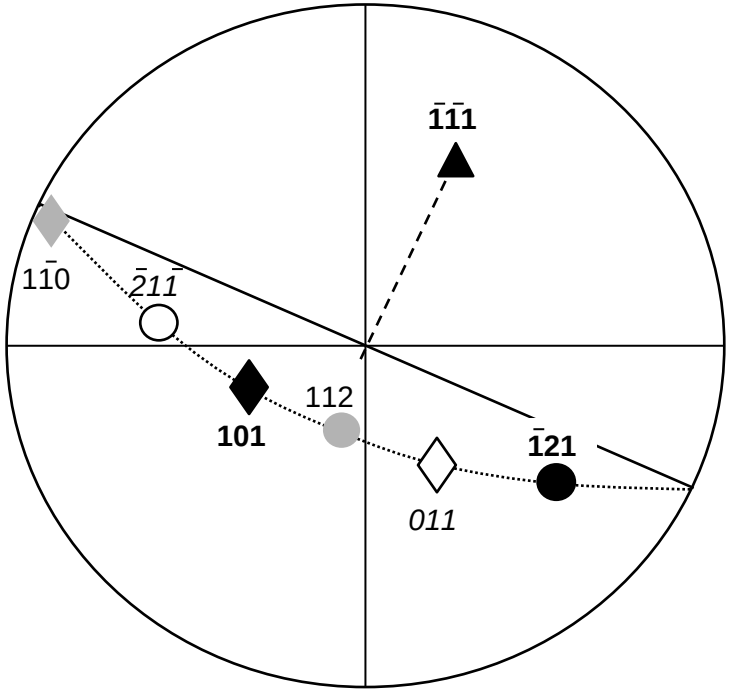
J. Villanova, «Détermination des contraintes résiduelles dans les matériaux céramiques pour SOFC », thèse de doctorat EMSE (2010)

A few selected examples

316L Single Crystal



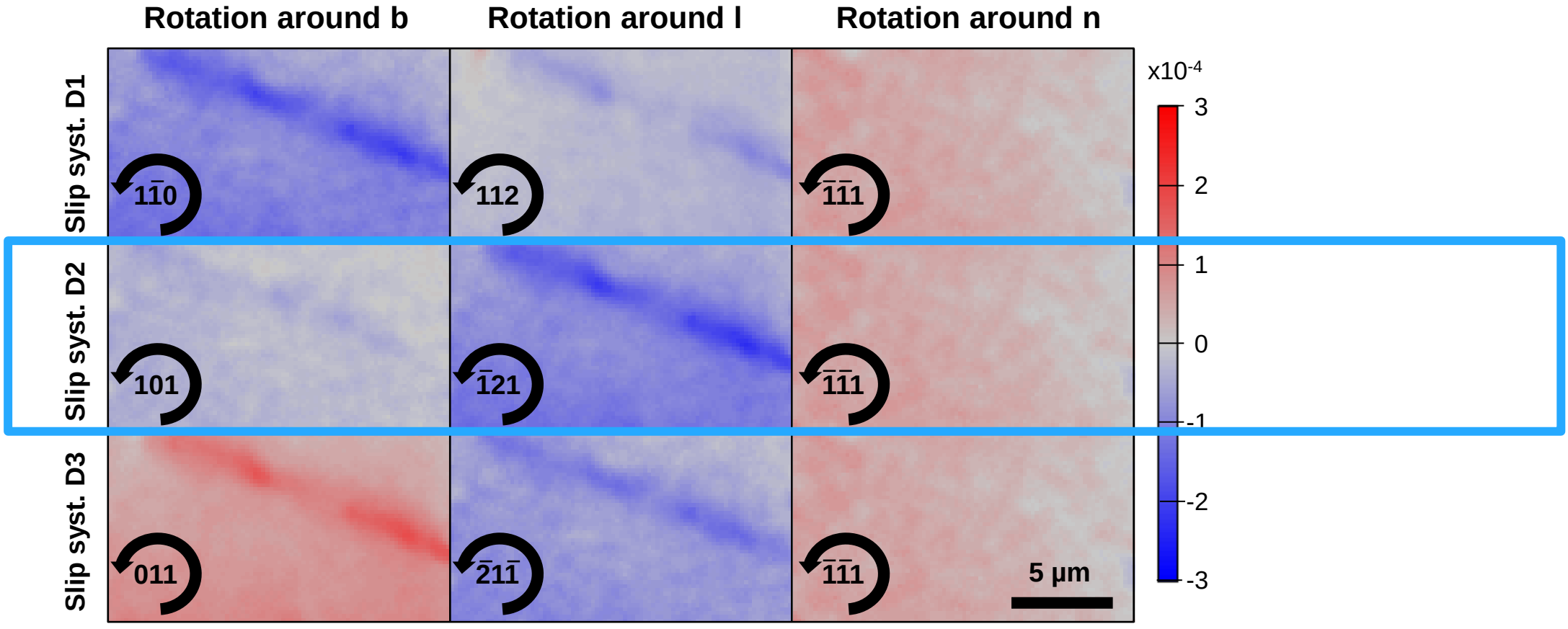
Strain and rotation tensor in Sample reference frame



Linear features // to (-1-11) plane trace

A few selected examples

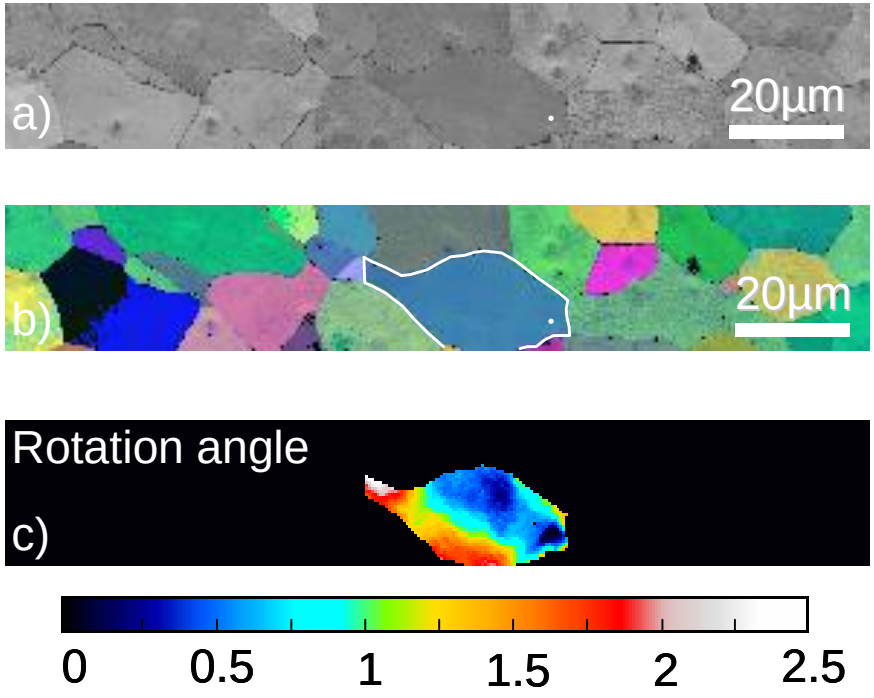
Rotation tensor projected onto the 3 possible (-1-11) slip systems

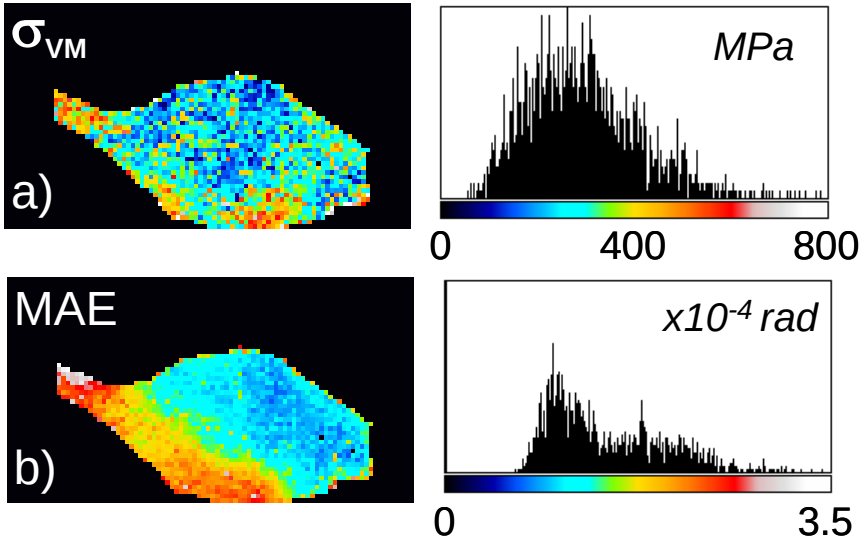


Consistent with edge dislocations on D2

A few selected examples

IF steel deformed 2.5% in traction



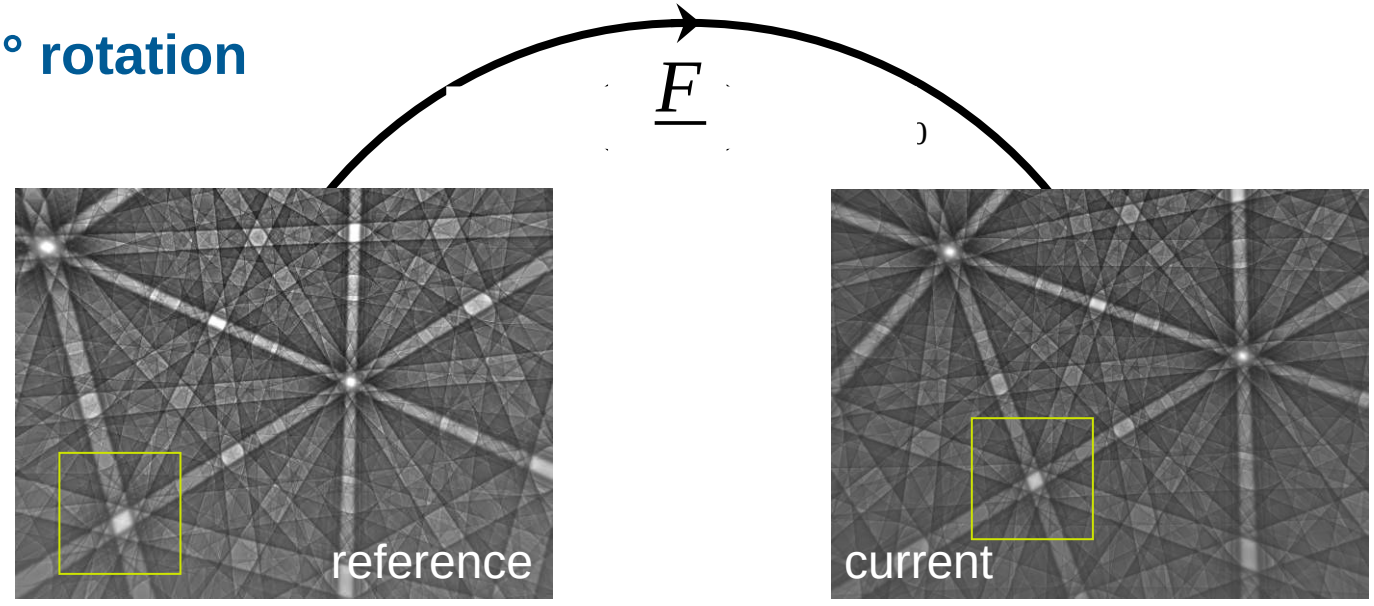


Anomalous strains and stresses are found in regions rotated by more than 1° from the reference point

Sample provided by ENSAM-Metz
ANR-SAKE collaboration

What's wrong with orientation gradients ?

15° rotation



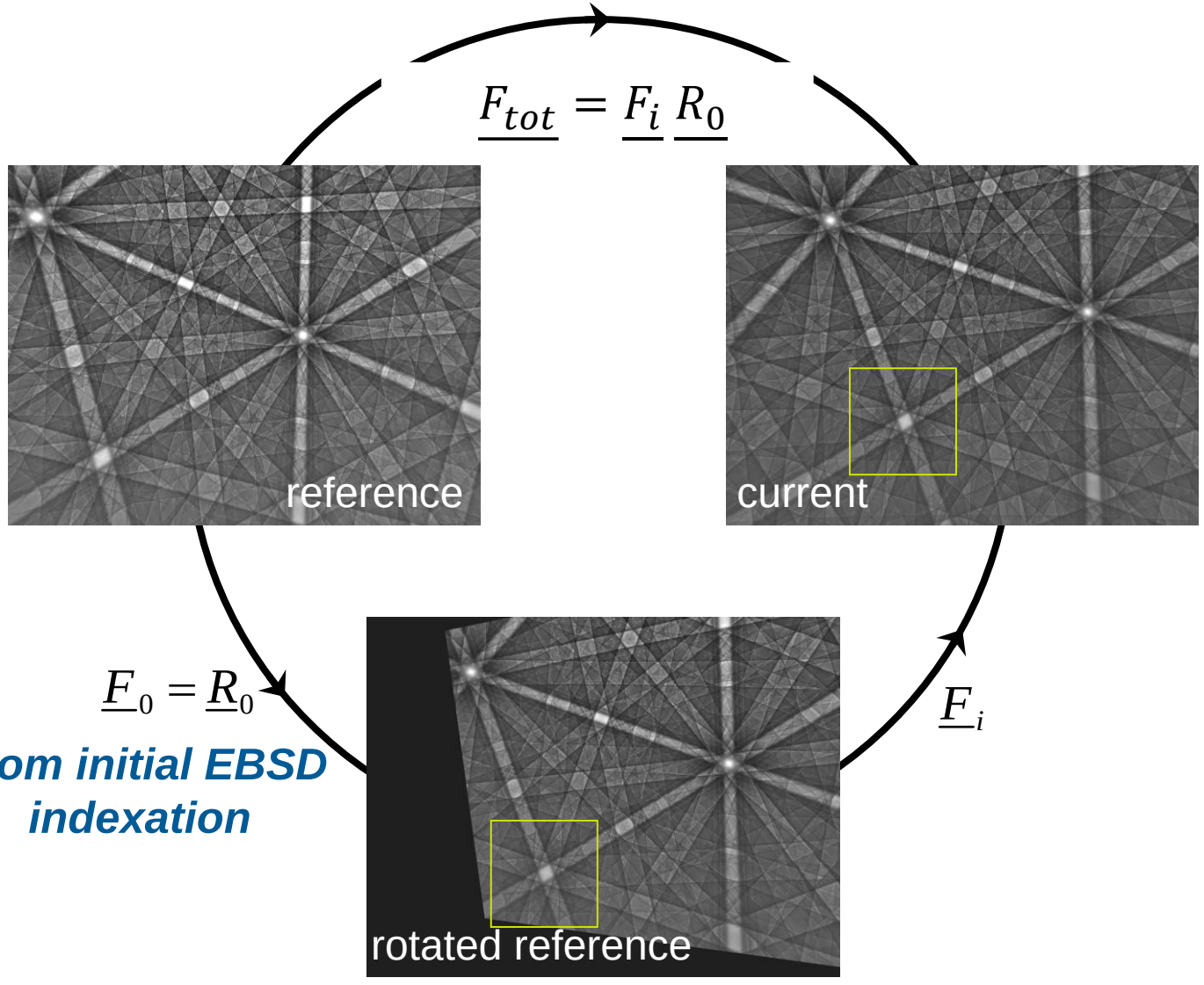
ROI patterns are stretched and rotated

Displacement field is poorly approximated by ROI shifts

Phantom strains and rotations

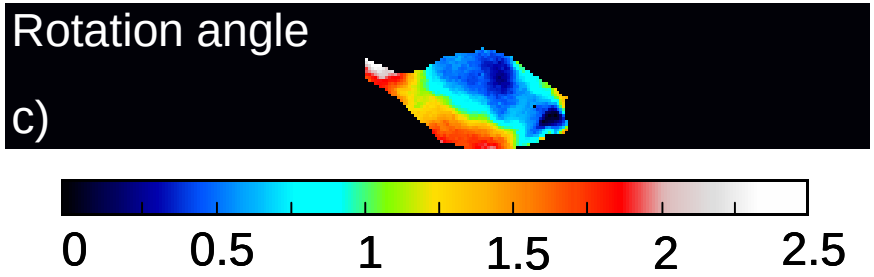
What's wrong with orientation gradients ?

15° rotation

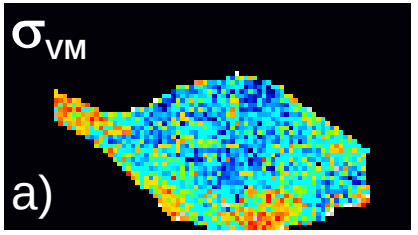


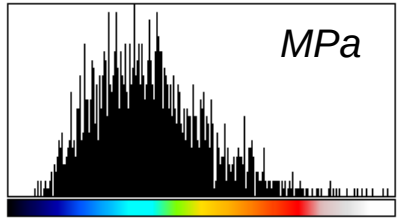
From initial EBSD indexation

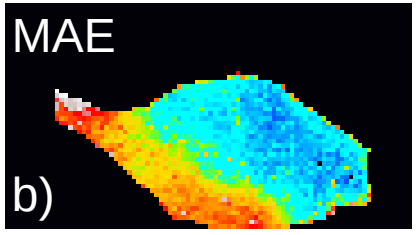
What's wrong with orientation gradients ?

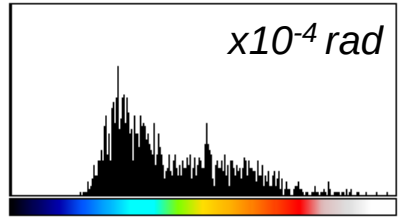


Without RRT

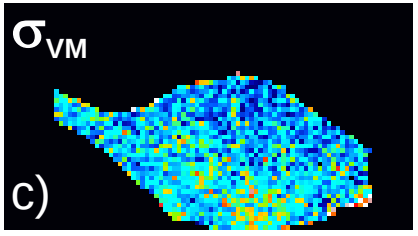


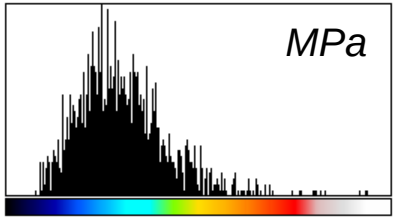


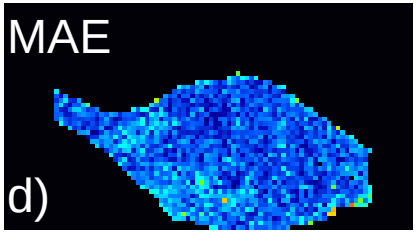


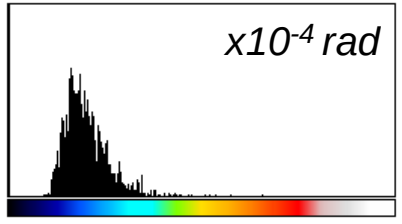


With RRT





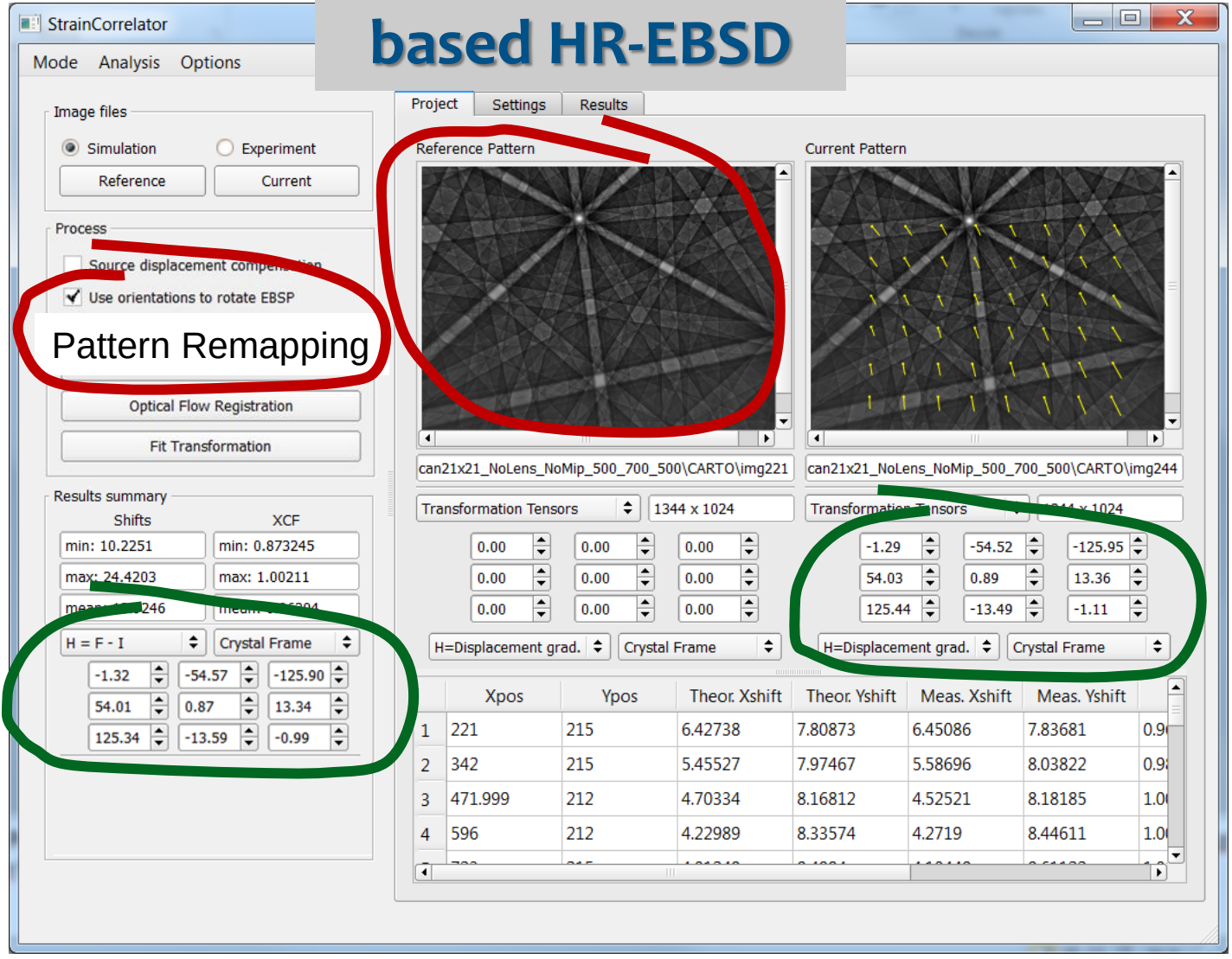




Remapping of one of the pattern solves the problem.

Residual Stresses by EBSD improving on XC-based HR-EBSD

Cross-correlation based HR-EBSD



Some success

Good agreement
« imposed / measured »
Max error ~ 0.1x10^{-4}

But a few limitations

- **Unknown mechanical state**

  Intragranular variations
  i.e. Type III Res.Stress only

- **Unknown source position**

  Possible artefacts when remapping

Limitations

A few ideas to overcome the reference pattern problem

Use a simulated pattern as reference

Attractive idea ….

… but many issues as shown by Britton et al. – Ultramicroscopy 2010

"Factors affecting the accuracy of high resolution electron backscatter diffraction when using simulated patterns"

Combine Kossel microdiffraction (mean strain) and EBSD (local gradient)

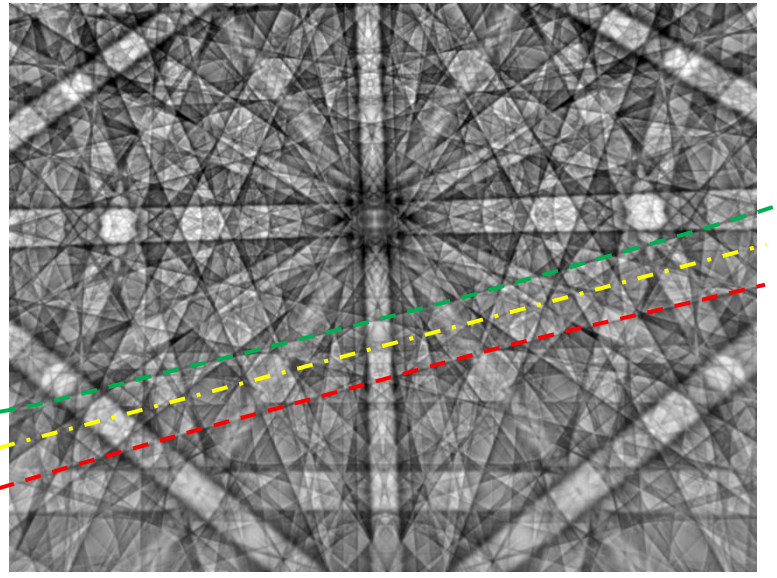
ANR Project SAKE : Strain Analysis by Kossel and EBSD
(ENSAM Metz – Mines Saint-Etienne – STMicroelectronics)

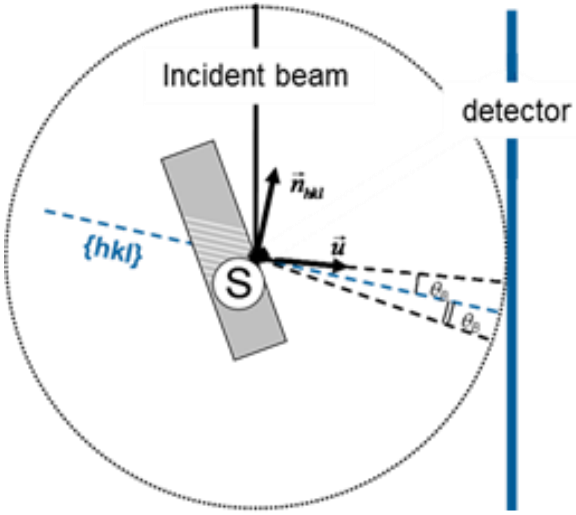
Enhance the angular resolution of direct pattern analysis

Based on accurate edge localisation by 3D Hough transform

3D Hough Transform
The Basics

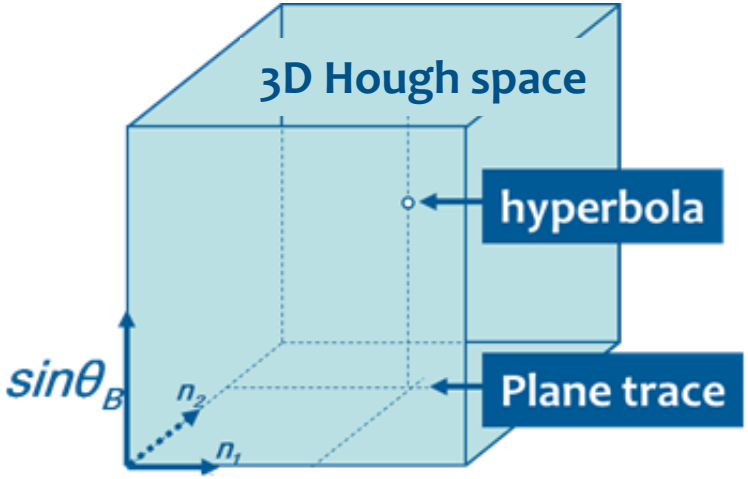
High Angular Accuracy EBSD

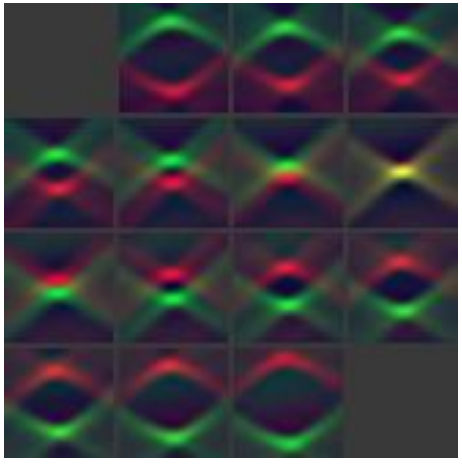




Plausible Kossel Lines

$$\vec{u}.\vec{n} = sin\theta_B$$



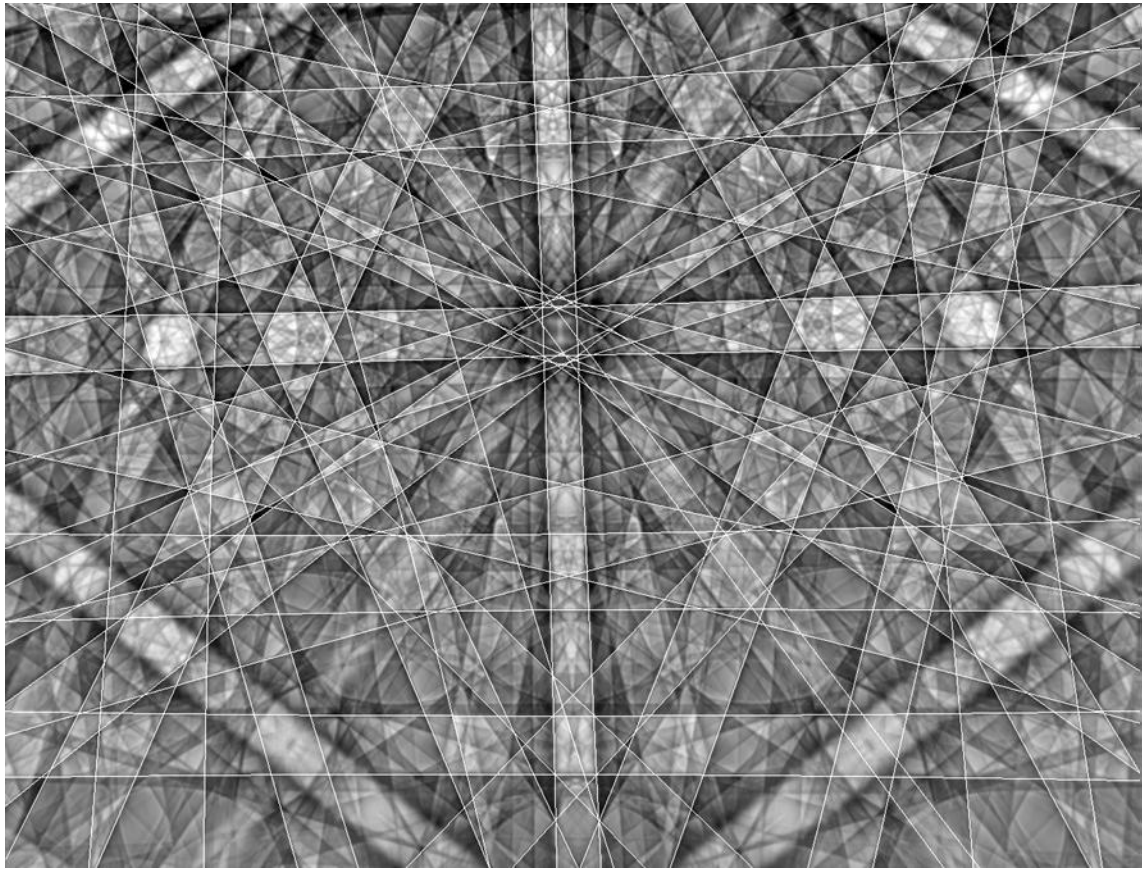


Constant $sin\theta_B$ sections

Presented at RMS EBSD 2007 – New Lanark
C. Maurice & R.Fortunier, Journal of Microscopy (2008)

Known EBSD Geometry

On unstrained germanium (simulation)



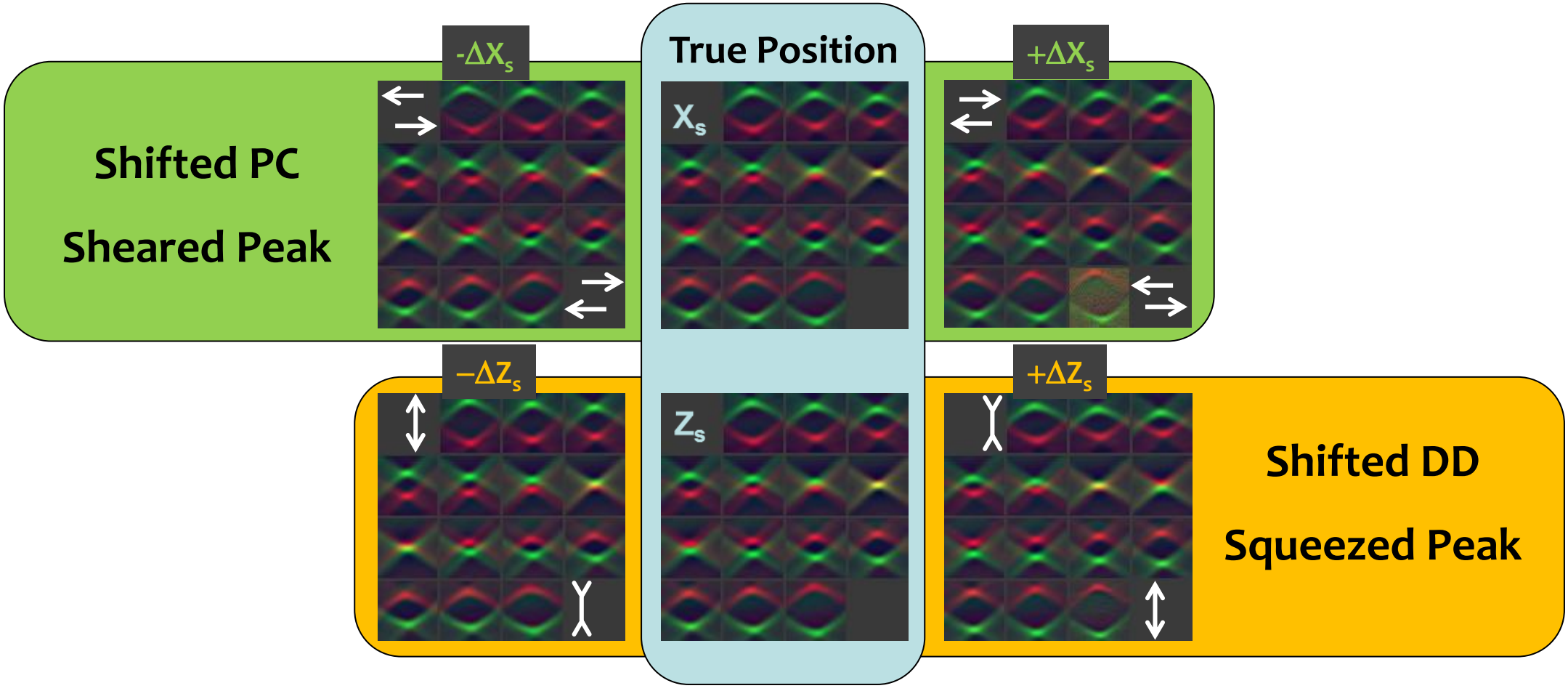
25 reflectors are detected

From these reflectors :

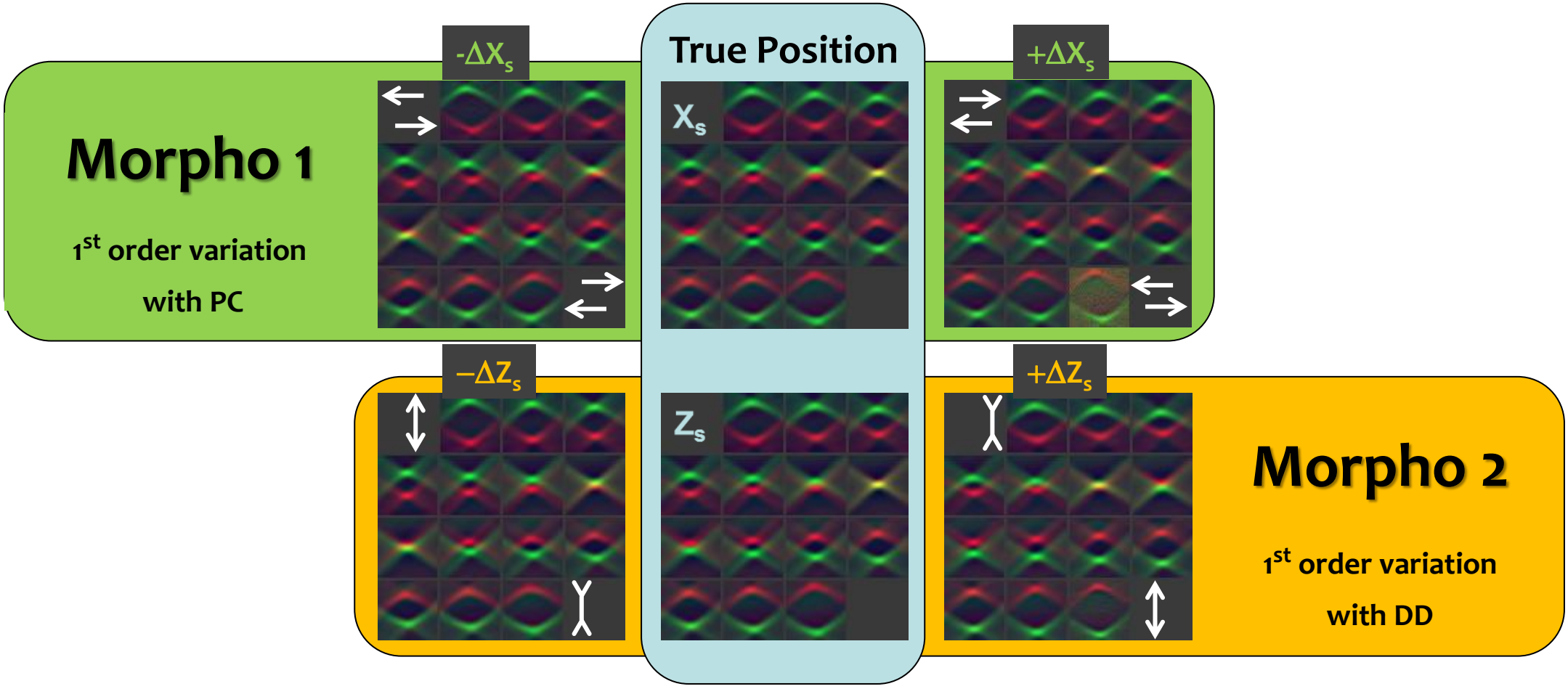
- *Refine the lattice parameters*
- *Refine the orientation*
- *Compute strains and stresses*

$$\underline{\sigma} = \begin{pmatrix} 5.4 & 6.7 & -5.8 \\ & 8.9 & 3.4 \\ & & 0.1 \end{pmatrix} \text{MPa}$$

Not so well-known EBSD Geometry

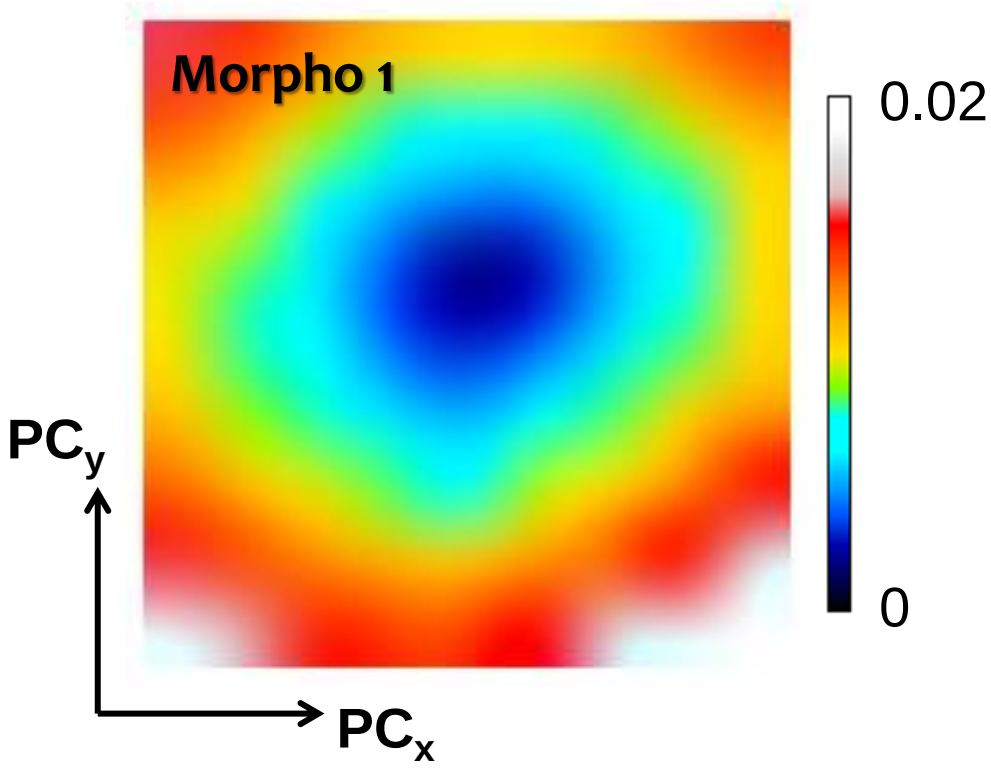


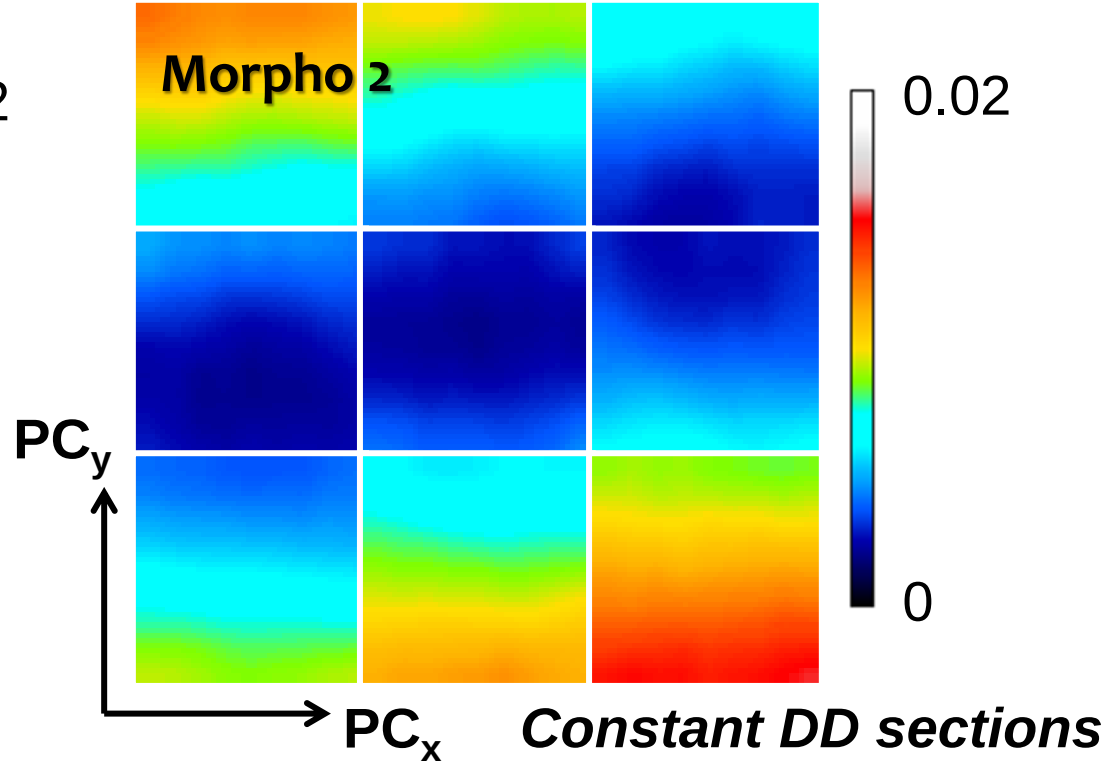
Two Morphological Descriptors to quantify the parallax error



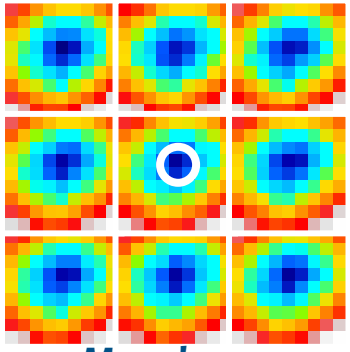
Scanning the Source Point space

Variation of the Morphological Descriptors for PC variation of ±4 pixels around true position

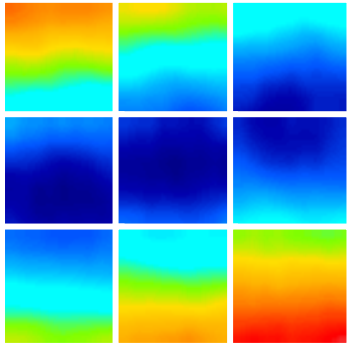




Scanning the Source Point space

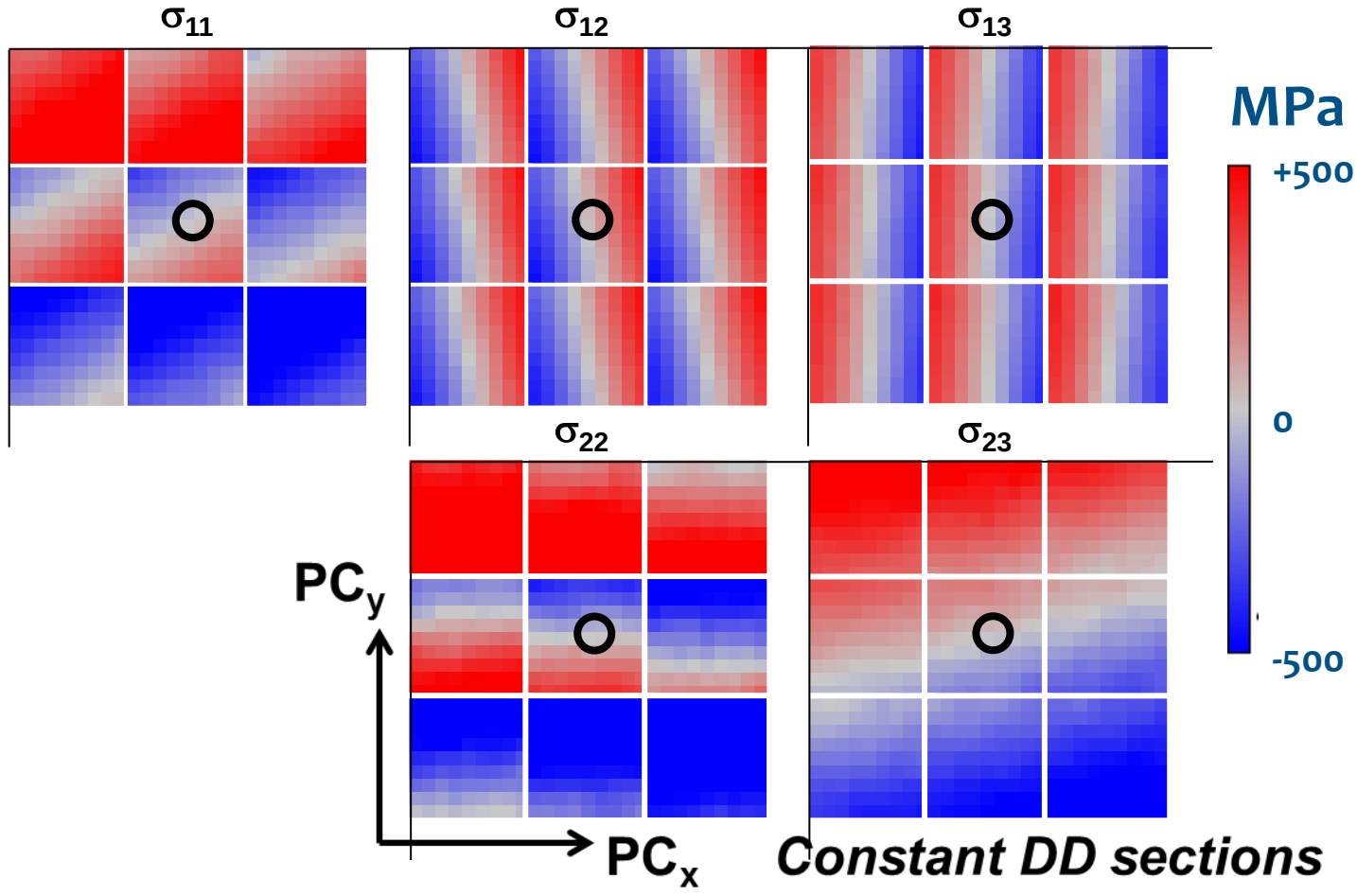


Morpho 1



Morpho 2

Mechanical state : Stress Tensor



Conclusions

Relative HR-EBSD by cross-correlation

geometric principles well established
effectively measures **low misorientations < 0.1° (check with TEM)**
remapping helps in case of orientation gradients

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« Absolute » HR-EBSD

3D Hough can yield both Source Point position and Strain

Acknowledgements

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MICROSTRESS

PNANO

Co-workers : Roland FORTUNIER, Sébastien VILLERT, Krzysytof DZIECIOL, Emeric PLANCHER (thèse EDF)

Thank you for your attention