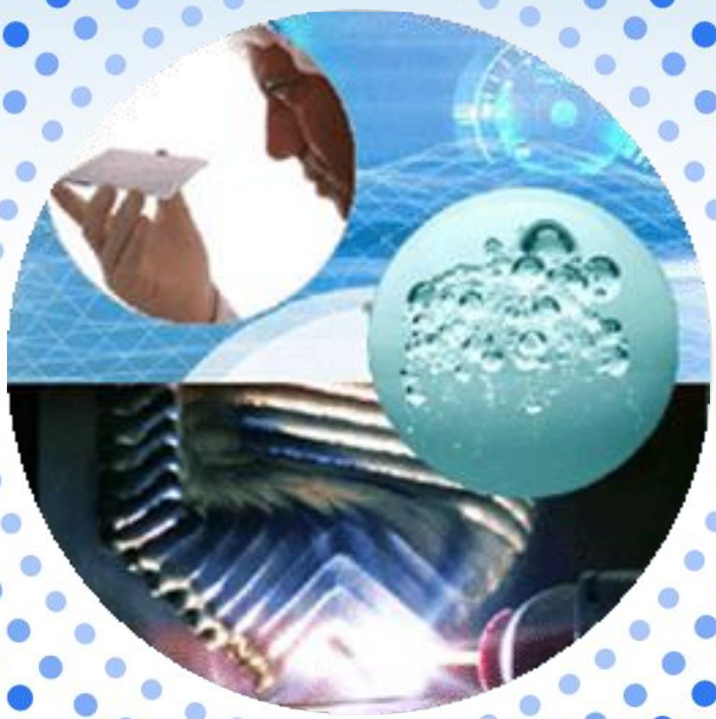
Measurements of local strain by HR-EBSD for stress and GND density evaluation.



Why go further than standard EBSD?

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Summary

1. FAQ about HR EBSD

2. Introduction to the theory

3. Some cases where a shift measurement error occurs

- **highly deformed metals**
- **wide field scan analysis**

4. Improve absolute orientation accuracy

5. Experimental conditions

6. How to process HR EBSD data using CC4 software

7. Example of application : In-situ measurements on mono and polycrystalline Tungsten sample under flexion load.

FAQ about HR EBSD

-Why stress assessment at the local level is a vital topic in the field of industry ?

-What is the application range of HR EBSD compared to standard EBSD ?

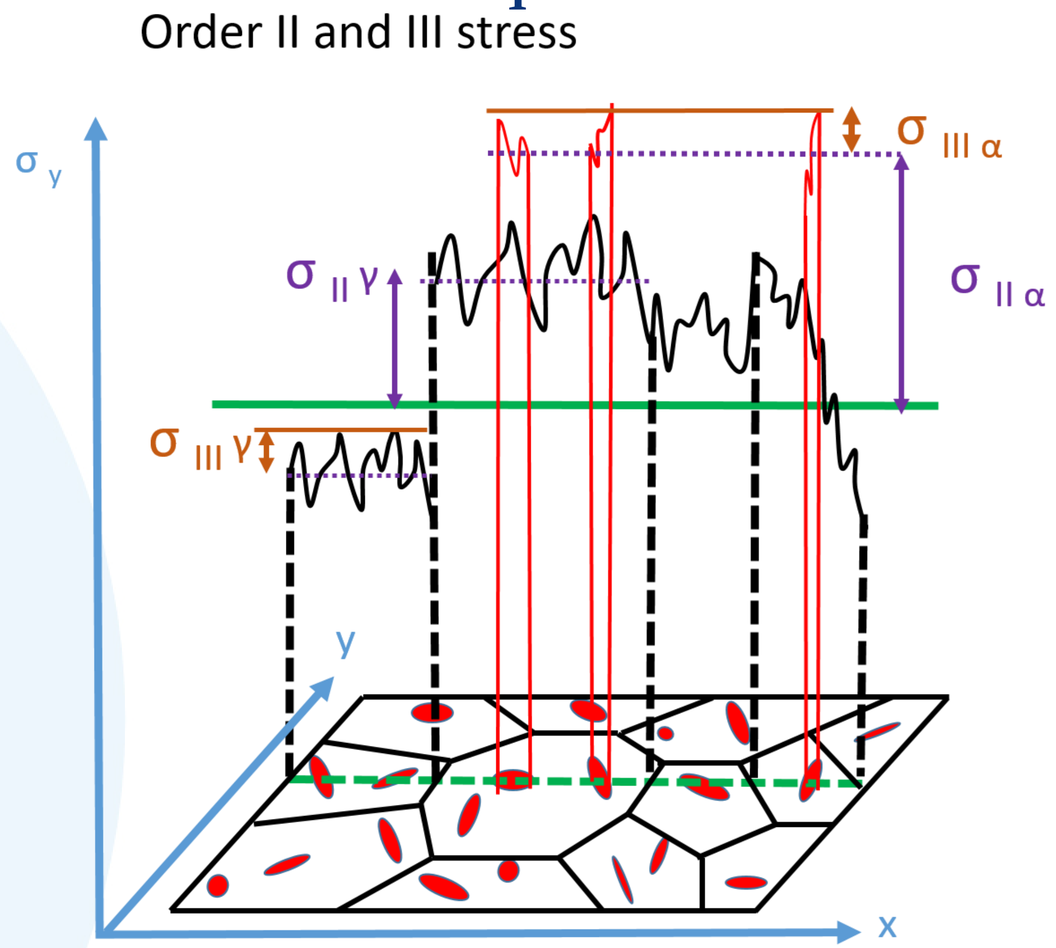
-Why Kikuchi patterns, with the highest quality, are used for HR EBSD ?

-How to optimize HR EBSD data collection ? Conditions, cautions and tips.

-Why good sample preparation is crucial for HR EBSD analysis in comparison with analysis based on orientations measurement ?

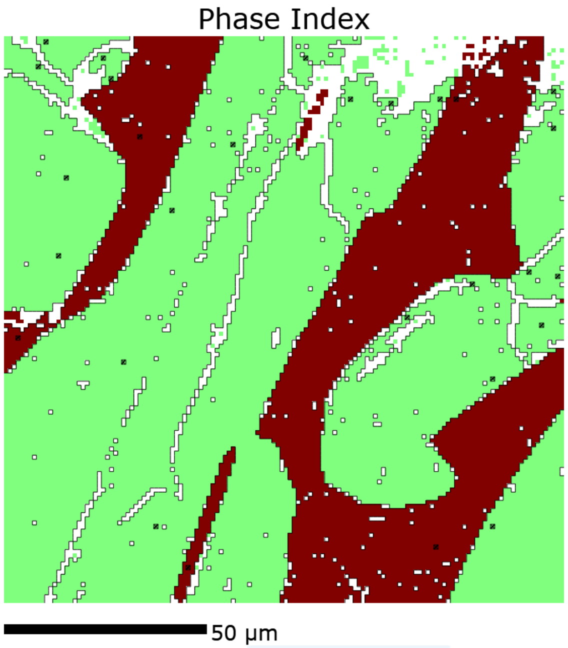
-How to process HR EBSD data ? What are the best practices for accurate results?

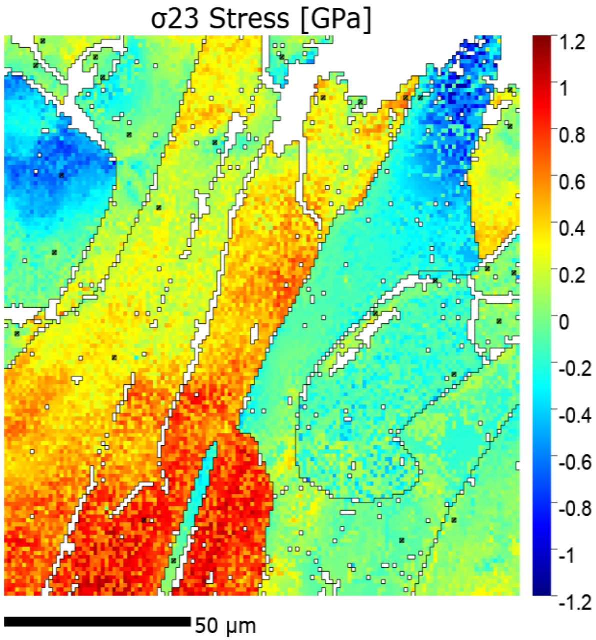
Stress order II at mesoscopic scale (from less than one square cm to few square mm).

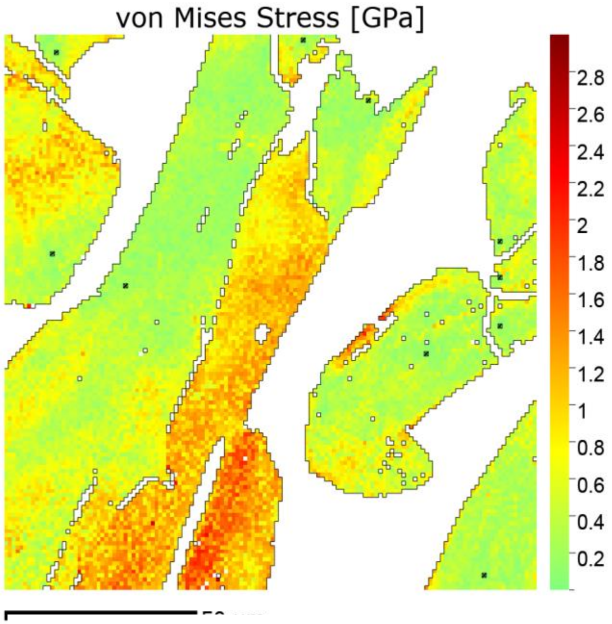


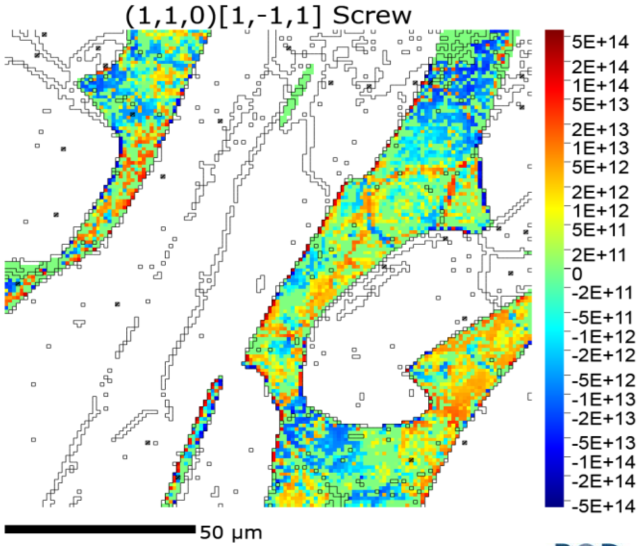
The order II stress is not identical for each grain, either because of the anisotropies of the elastic and plastic behaviors of each grain, or because of the differences in the physical properties of the phases.

Stress order II at mesoscopic scale : a duplex steel







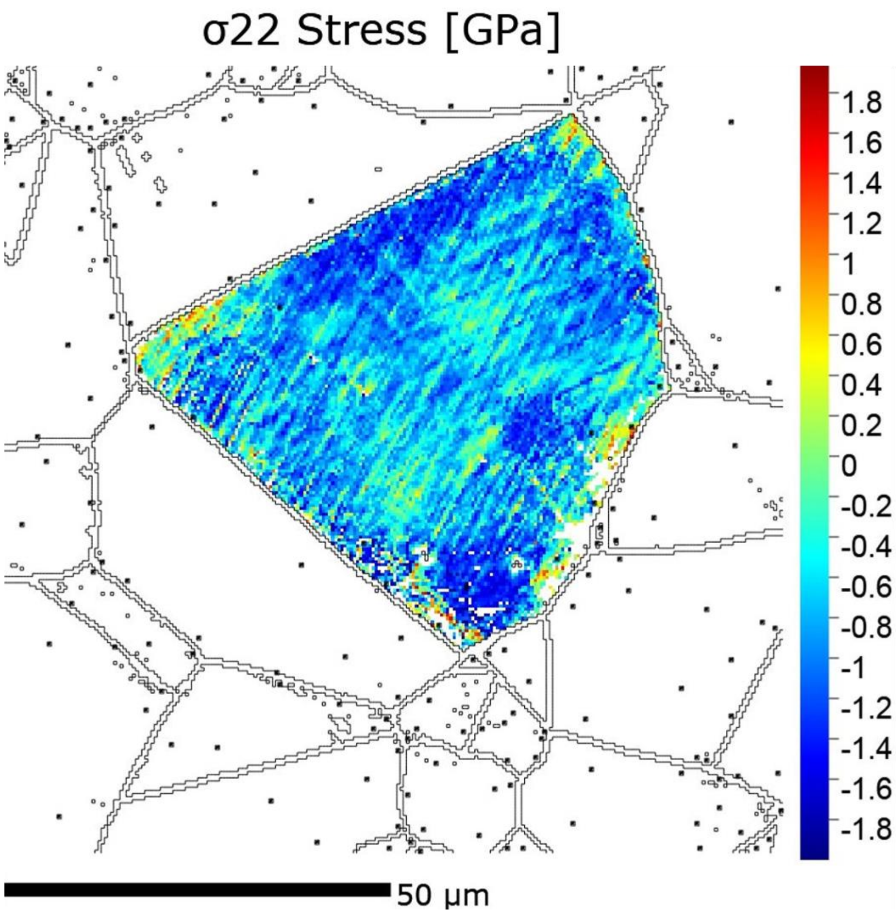


The order II stress operates at the level of the grain interfaces or between the phases.

The order II stress depends on the size and orientation of the grains.

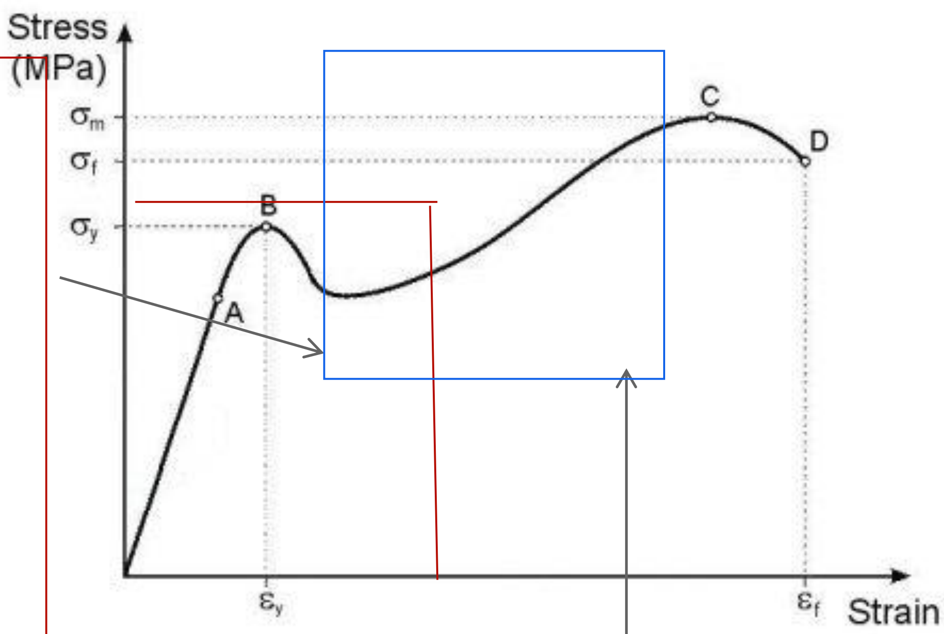
R&D

Stress order III is at microscopic scale (from less than one square mm to few square µm).



The order III stress is the result of the mechanical loading in a single grain and the effects of metallurgical parameters, like precipitation of intragranular phase or slip plans activation by dislocations. The order III stress is intragranular fluctuations.

EBSD / HR EBSD – the applications range



HR-EBSD

Measurements in the elastic domain with an accuracy of 1.10^{-4} on strain.

Very low rotations sensitivity (0.006°). But difficult to obtain reliable measurement if they are large local misorientation.

EBSD

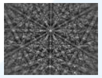
No possible realistic strain measurements in the elastic domain.

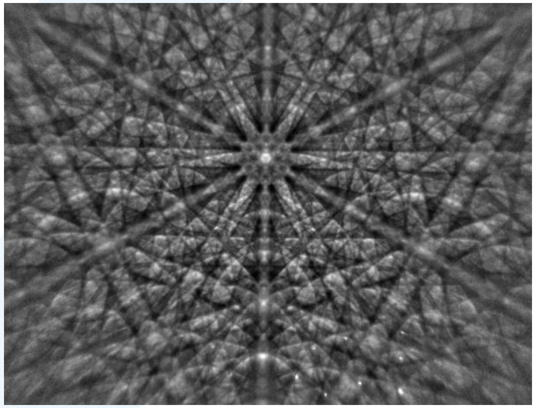
Measurements of misorientations are limited to 0.1°, that corresponds to large noise floor of GND measurement. From C to D on the curve, the patterns quality is too low for good indexing and reliable misorientations measurements.

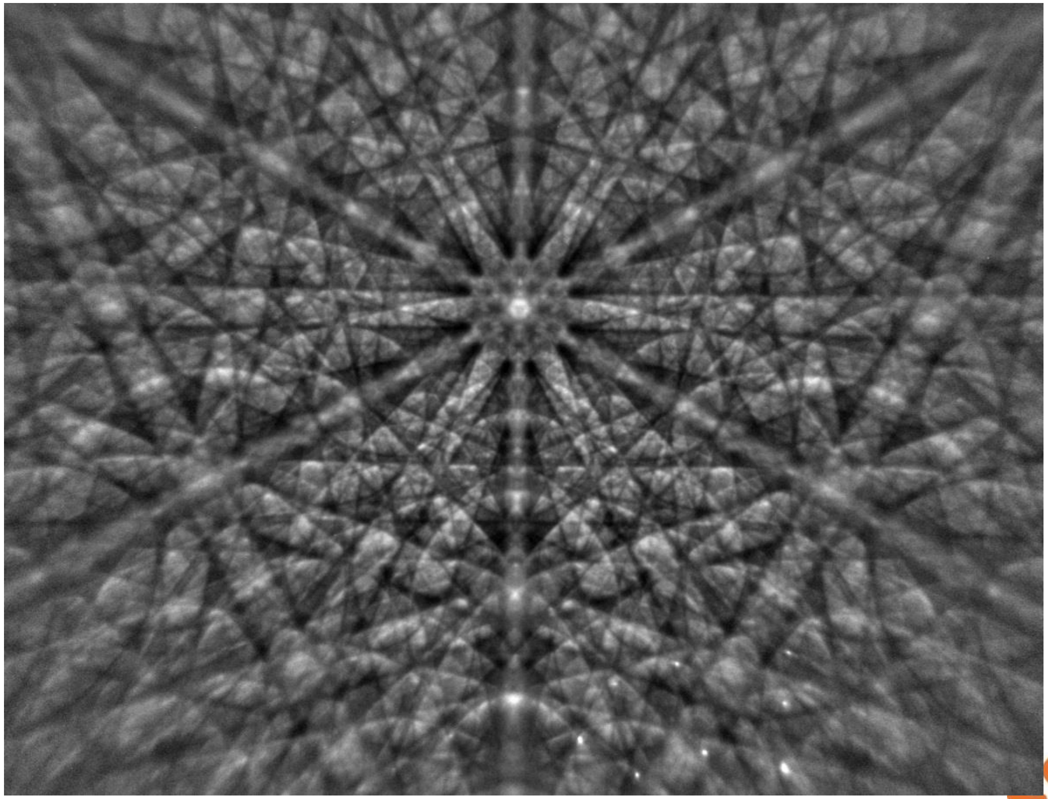
EBSD / HR EBSD – The main difference : the size of the saved patterns

EBSD fast camera, bin 8*8, pattern size 2500 pixels, 8 bits.

HR EBSD on a standard camera, pattern size 1M piexls, 12 bits.







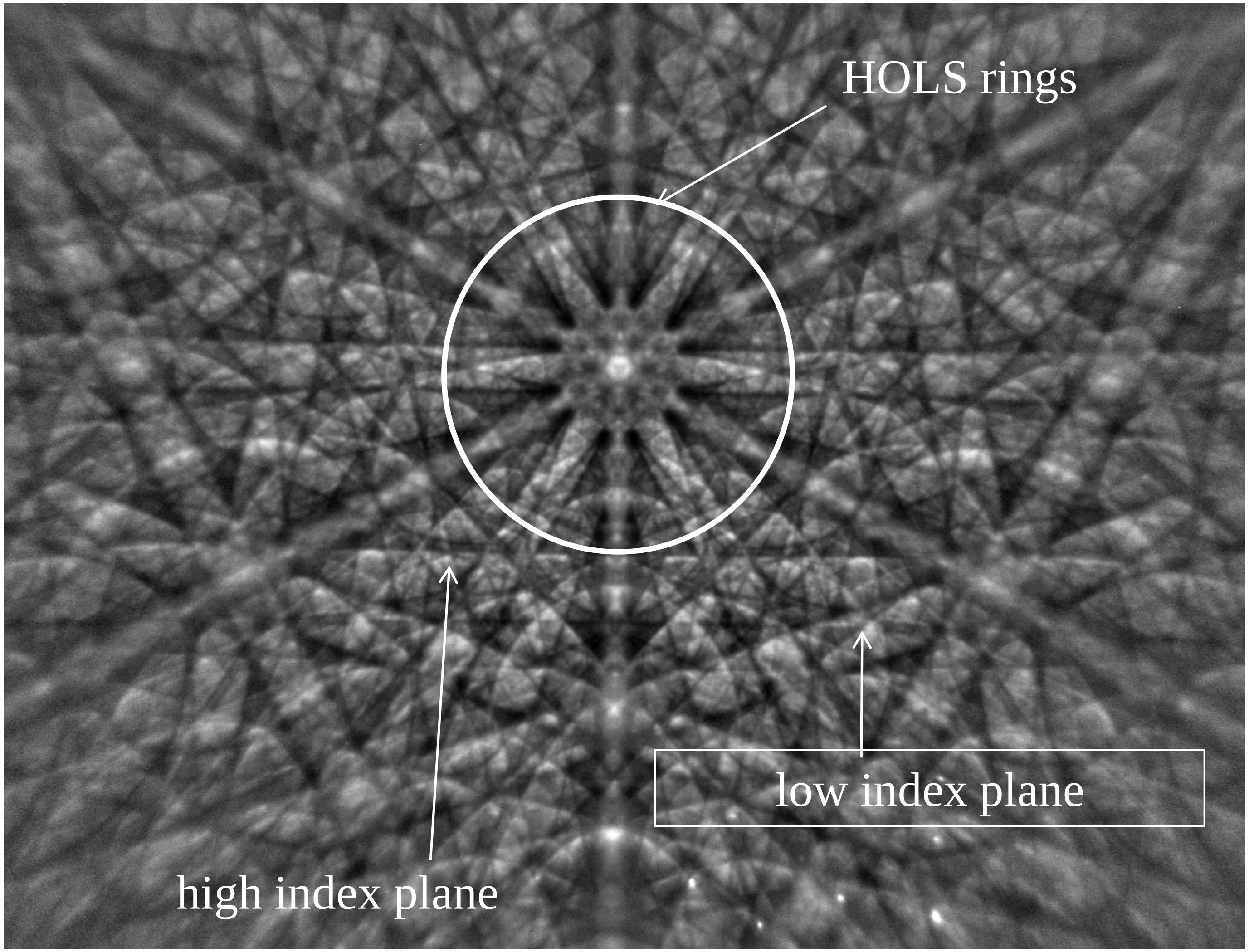
HR4M, dedicated camera to HR EBSD, pattern size 4M pixels, 16 bits.

What is useful in the HR EBSD pattern ?

All fiducial

Unlike standard EBSD, where only the low index plans are used, the HR EBSD uses all the details contained in the pattern !

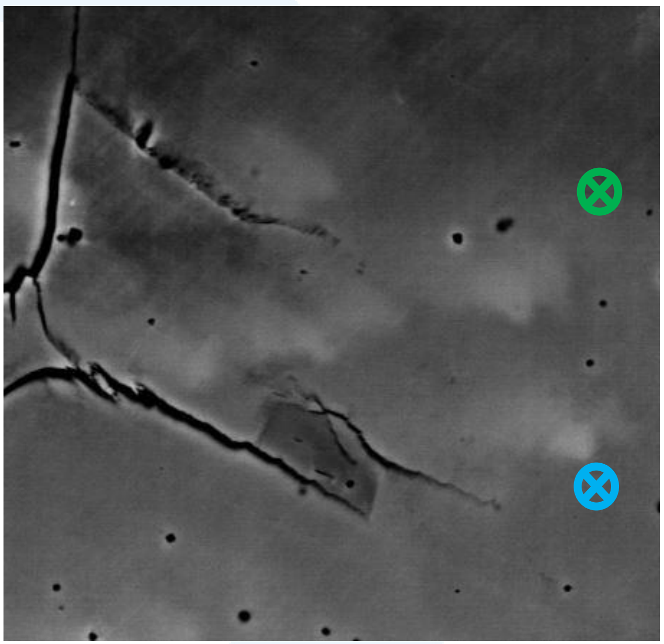
In particular, the dynamic effects on the zone axes and the contrasts within the bands are very sensitive to strain effects.

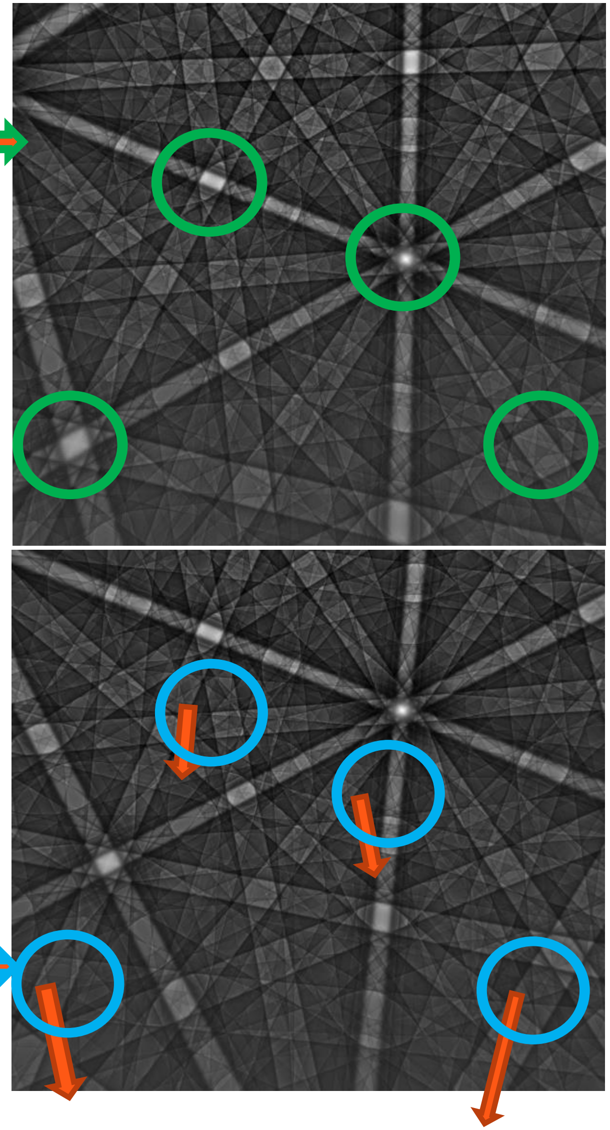


The basic of the currently used HR EBSD technique

Early idea from Troost and al 1993

Pattern coming from an unstrained part of the grain





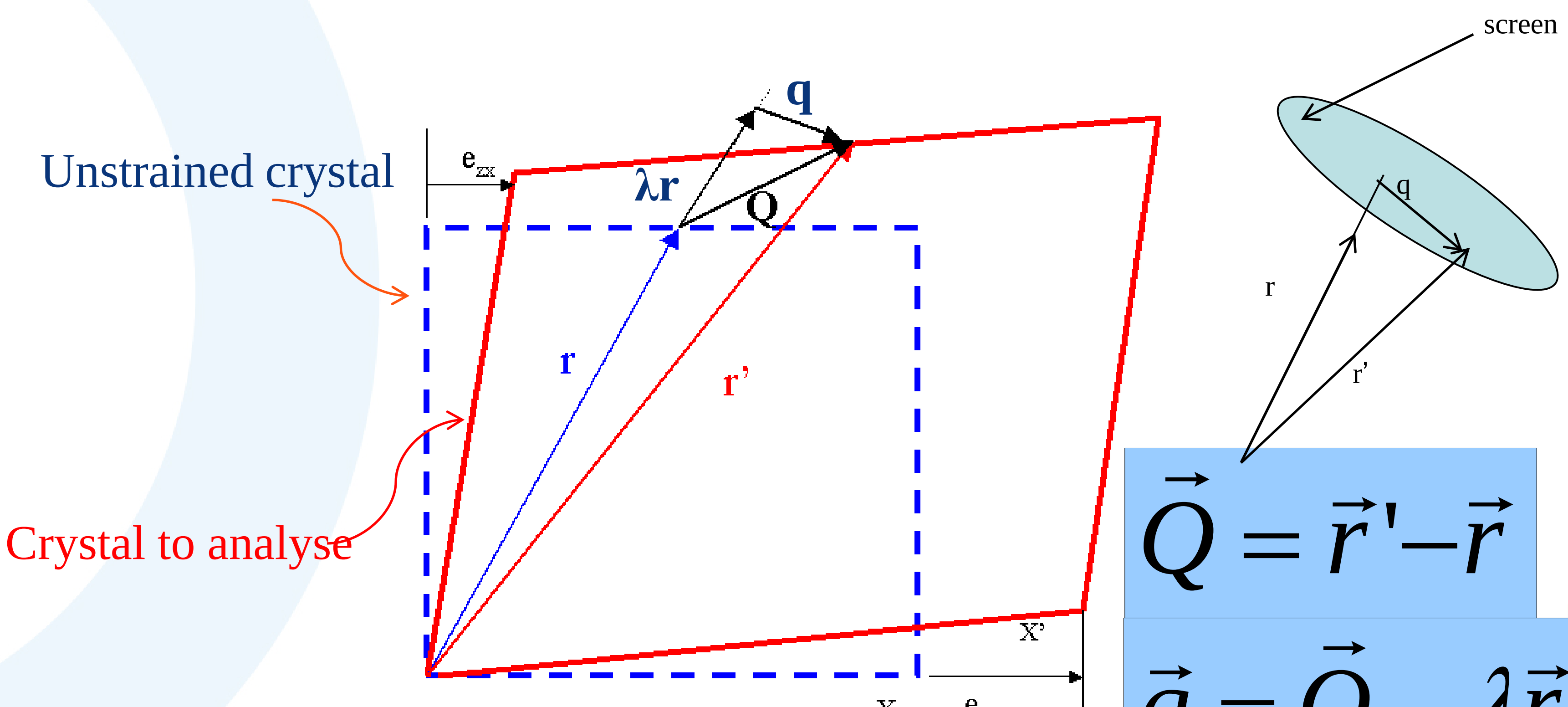
Pattern to be analysed

Apart from the GND map, all other HR EBSD measurements are obtained by using one or more references patterns per grain.

HR EBSD is a relative technique (such as the GROD map with EBSD) and does not provide absolute measurements.

The basic of HR EBSD : The geometry

Displacement of vector Q is measured by cross-correlation technique between a reference pattern (chosen at an unstrained zone) and an acquired pattern to analyse.

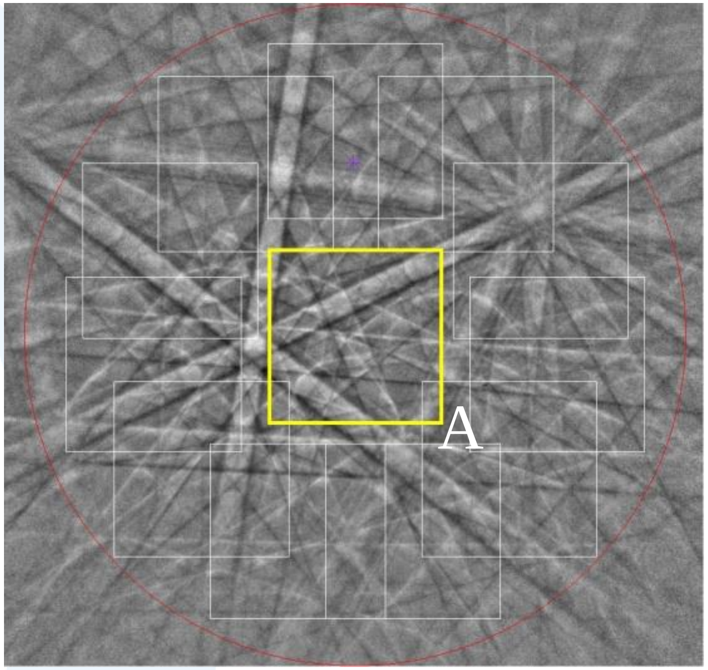


$$\vec{Q} = \vec{r}' - \vec{r}$$

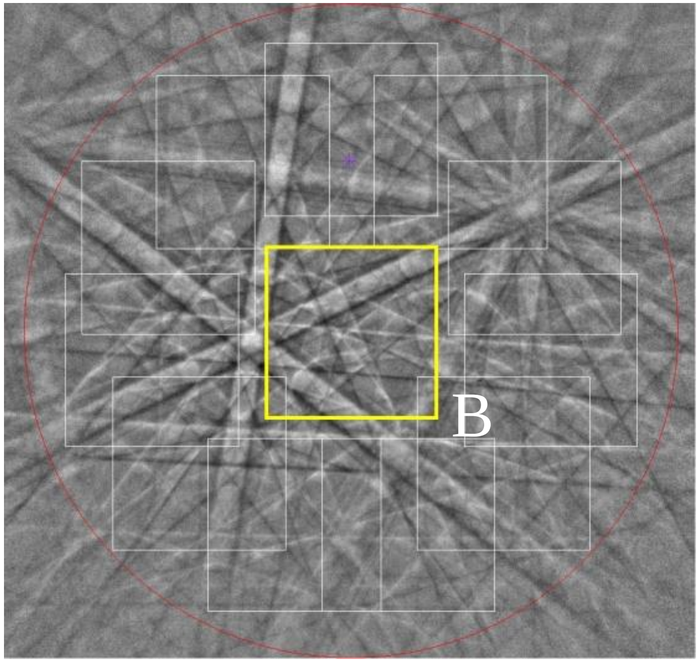
$$\vec{q} = \vec{Q} - \lambda\vec{r}$$

The basic of HR EBSD : use region of interests

Reference pattern (supposed to be unstrained)



Pattern to be analysed



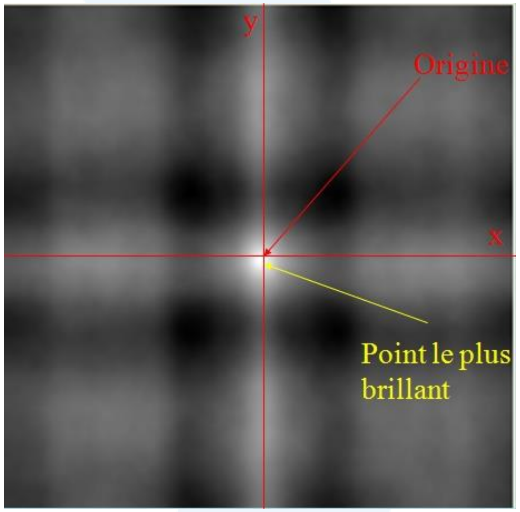
Patterns are cut into region of interests (ROIs).

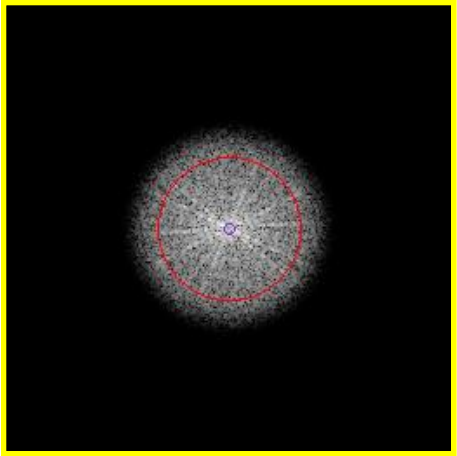
On pattern corresponding to the pixel to be measured, the ROI B is compared with the ROI A, coming from the pattern of reference.

The basic of HR EBSD : use Fourier Transform

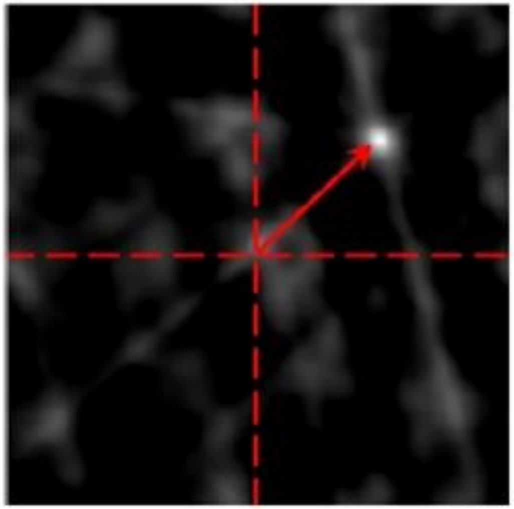
ROI (A) coming from reference pattern → **Fourier Transform / Low and high band pass filter** ← ROI (B) coming from analyse pattern

Autocorrelation of A with A (no shift of intensity)





Cross-correlation of ROI A with ROI B, inverse Fourier Transform (shift of intensity)



In the correlation image, the shift of maximum intensity point corresponds to the shift of the Kikuchi bands (vector q).

This measurement is made with 1/4 pixel accuracy.

The basic of HR EBSD : Connect shift to strain

Distortion matrix a : $\vec{Q} = \vec{r}' - \vec{r} = a \times \vec{r}$

System of 2 equations with 8 unknowns:

4 ROIs + device geometry (Pattern Center)

Crystal orientation (Euler angles) + σ33 (0)

$$a = \begin{pmatrix} \frac{\partial u_1}{\partial x_1} & \frac{\partial u_1}{\partial x_2} & \frac{\partial u_1}{\partial x_3} \\ \frac{\partial u_2}{\partial x_1} & \frac{\partial u_2}{\partial x_2} & \frac{\partial u_2}{\partial x_3} \\ \frac{\partial u_3}{\partial x_1} & \frac{\partial u_3}{\partial x_2} & \frac{\partial u_3}{\partial x_3} \end{pmatrix}$$

Where u is the shift to the x' position

Decomposition of the distortion matrix:

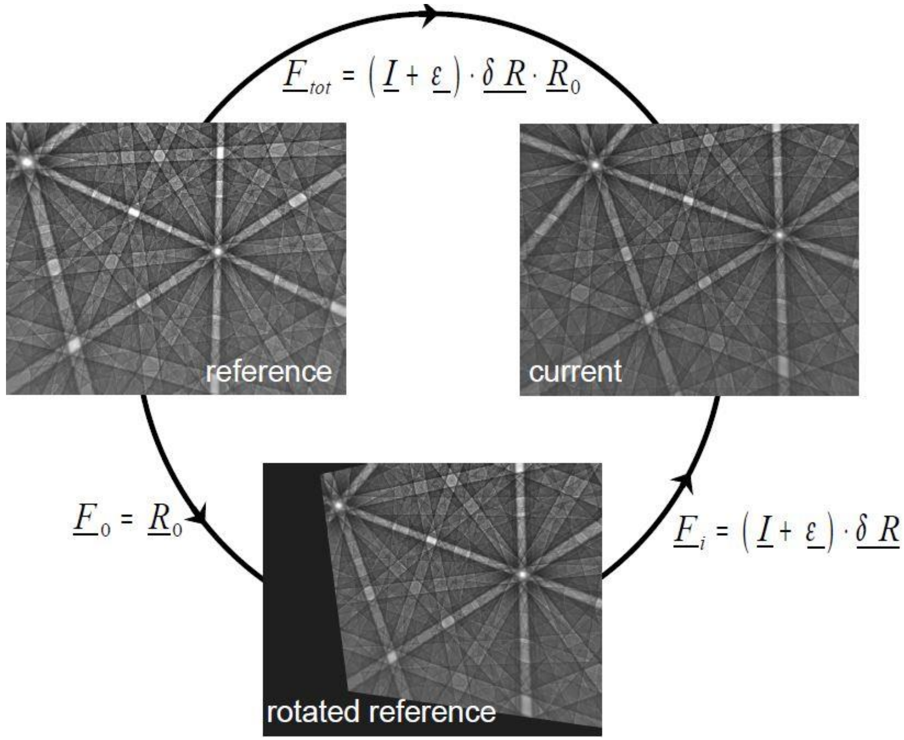
$$\begin{pmatrix} a11 & a21 & a31 \\ a12 & a22 & a32 \\ a13 & a23 & a33 \end{pmatrix} = \begin{pmatrix} e11 & 0 & 0 \\ 0 & e22 & 0 \\ 0 & 0 & e33 \end{pmatrix} + \begin{pmatrix} 0 & e12 & e13 \\ e12 & 0 & e23 \\ e13 & e23 & 0 \end{pmatrix} + \begin{pmatrix} w11 & w12 & w13 \\ -w12 & w22 & w23 \\ -w13 & -w23 & w33 \end{pmatrix}$$

Distorsion matrix **Normal strain matrix** **Shear matrix** **Rotation matrix**

Shift measurement error :
The case of highly deformed metals

The presence of large intergranular rotations causes error on the strain measurements by Cross correlation.

This can be corrected by using a choice of large numbers of reference points per grain and by using the remapping procedure.



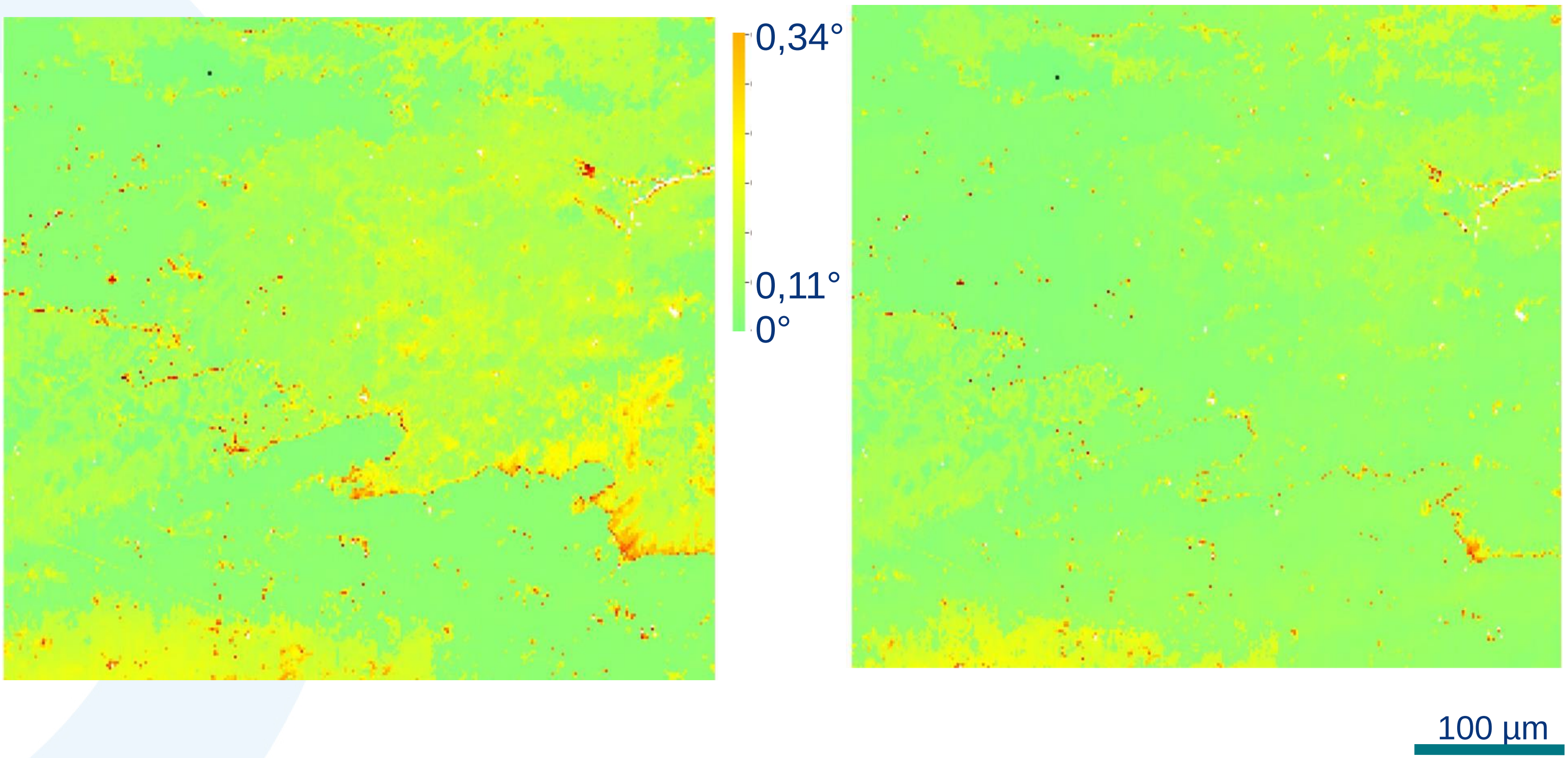
Registration procedure to correct large rotations (R_0)

C. Maurice Ultramicroscopy octobre 2011

Large intergranular rotations issue solved by remapping

0.34° average angular error before remapping

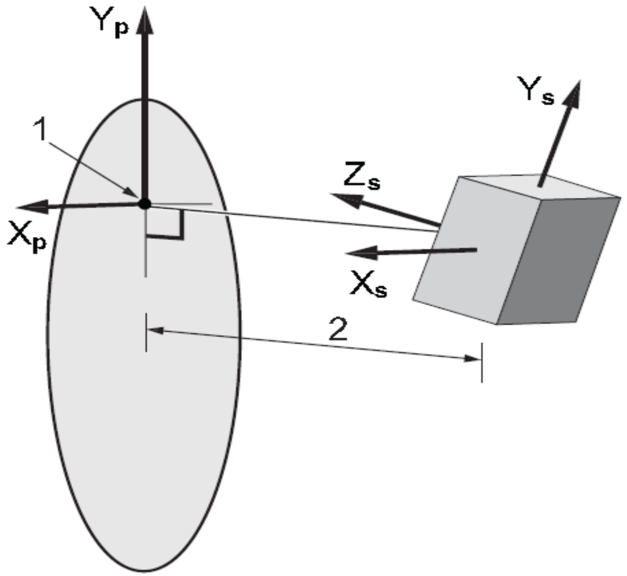
0.11° average angular error after remapping



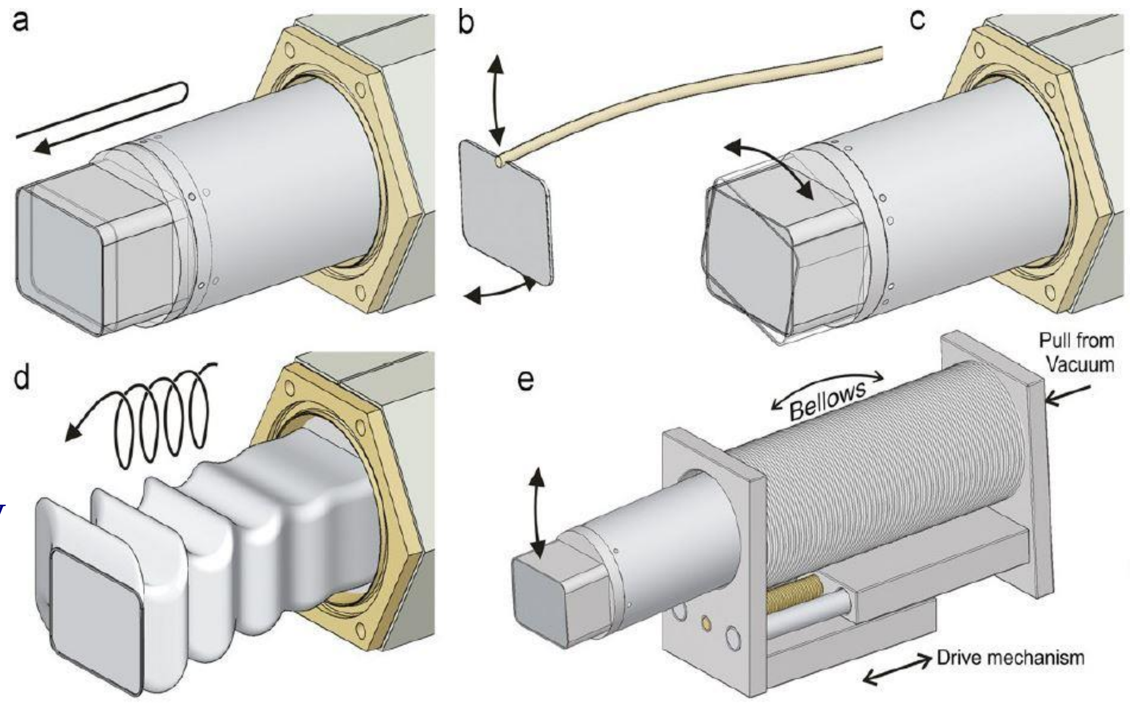
Mean angular error (residual of the minimization function) at each point of the analysis on a nickel base industrial alloy.

Shift error : The case of wide field scan analysis and reproducibility of camera setup

Precision of the determination of the projection parameters (Pattern Center PC) has a strong impact on the measurement error of the shift. It is very difficult to align the surface of the sample perfectly parallel to the screen on the camera.



Mingard, Day, Maurice, Quested, Ultramicroscopie 2011



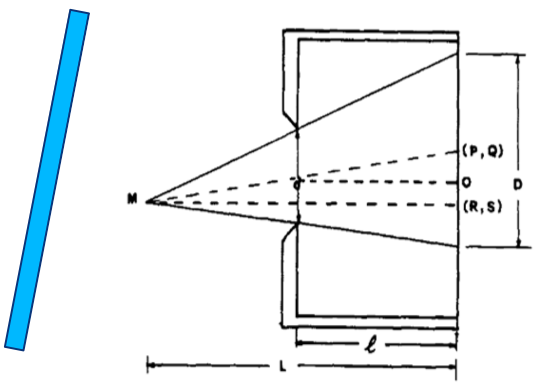
The position of the camera screen is unfortunately not always strictly reproducible.

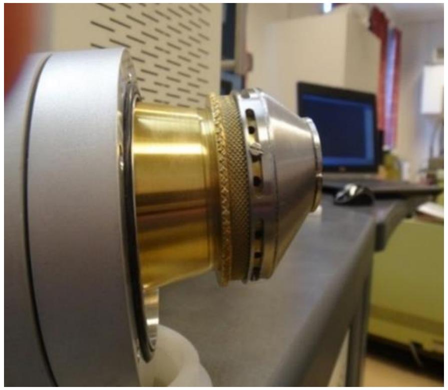
Shift error - a solution

Visualize the mark of shadows between the sample and the screen.

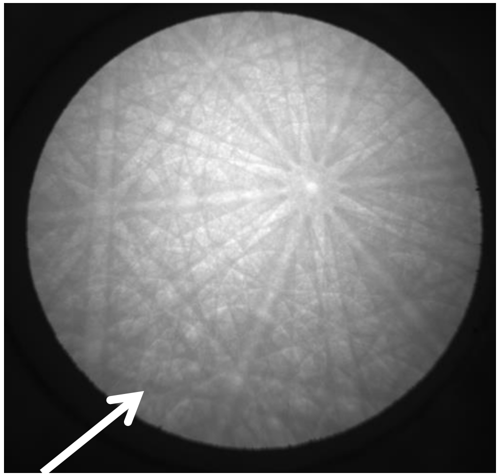
Early idea from J. A. Venables & R. Bin-jaya (1977), Philosophical Magazine.

Recently D.J Dingley and G. Meaden revised and improved the setup.





Piece drilled of a hole in front of the camera screen



Shadow coming from the edge of the hole

Improvement of the PC position by a factor of 5.

How to store HR EBSD Kikuchi patterns : conditions

Avoid scratch on the screen, shading (due to sample surface roughness) or charging effect.

Store Kikuchi patterns of the **full resolution** (1k*1k) in .tif

Never use the gain.

Use the **highest current** posible compatible with the step size of the analysis.

Do not hesitate to use a **long exposure time** but avoid over exposition.

Store the patterns by integration of a minimum of **3 raw frames**.

Check at all points of the field that the pattern corrected of the background will be undersaturated.

These conditions will allow limiting noise and the use of all the dynamics of the camera.

HR EBSD Kikuchi patterns : recommendations

High current and long exposure lead to two very important risks :

<u>The contamination of the surface of the sample.</u>

Wait until the ultimate vacuum in the microscope chamber is reached (the best is to put the sample in the microscope the day before running the scan).

Frequently perform plasma cleanings of the chamber and clean the metal sample with an oxygen-free plasma cleaner.

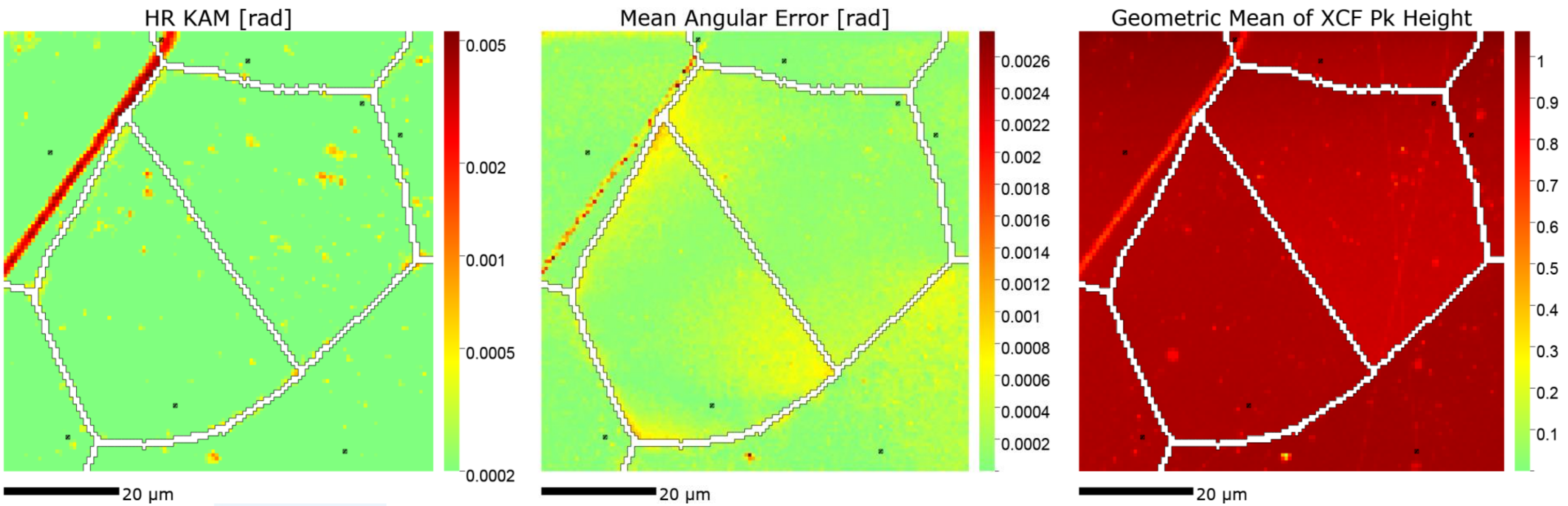
<u>The drift of the sample during the scan acquisition.</u>

Ensure a very good electrical contact. Check the stability of the probe current.

Turn on the high voltage and place the beam near the analysis area longtime before the start of the analysis. This is to establish a good thermal and electrical balance.

Start the analysis overnight to avoid the surrounding mechanical vibrations.

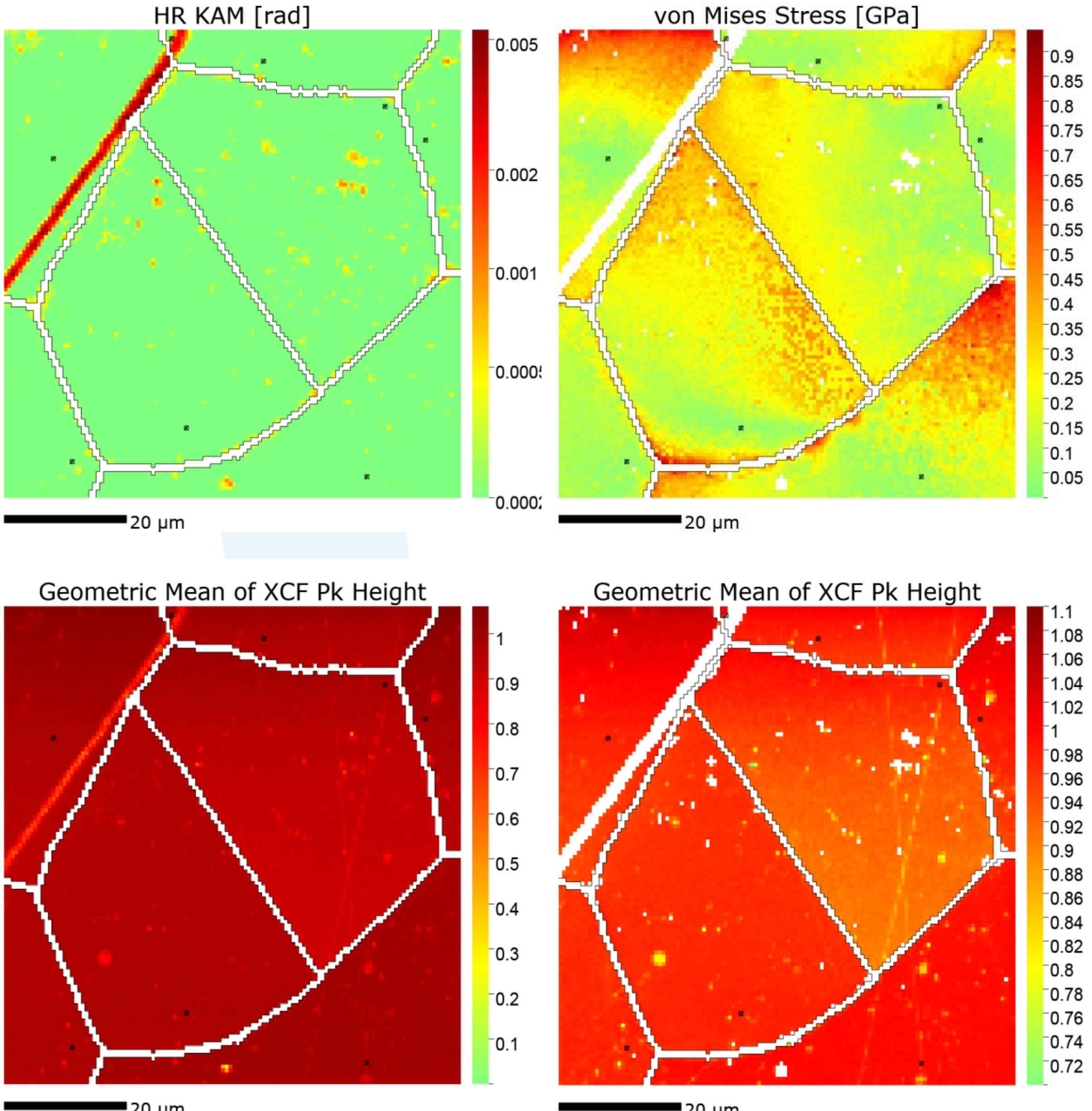
Why very good sample preparation is crucial for HR EBSD analysis !



HR EBSD quality maps highlight sample preparation issues (mainly coming from strain scratchs). Those artefacs are not a problem for the indexation and invisible using standard EBSD quality map.

For HR EBSD analysis, those areas should be removed from the dataset.

Very good sample preparation is crucial for HR EBSD analysis : Filtering the data !



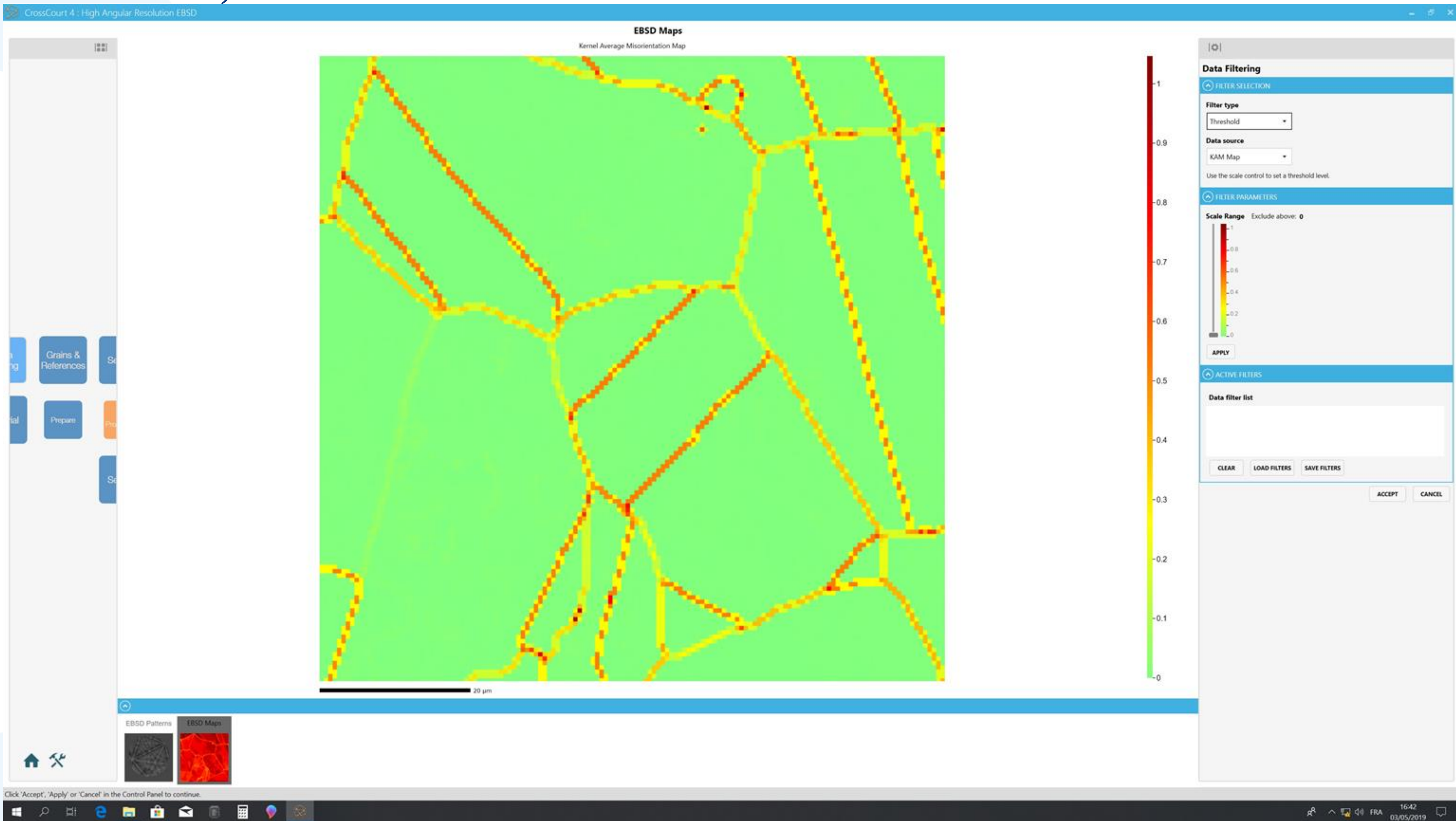
Big strain scratch removed from the dataset.

Note that some residual small strain scratchs can subsist after filtering !

Check residual artefact do not influence the stress results

How to process HR EBSD data using CC4 software. Good practice for accurate results.

Identify where the patterns, coming from two grains, do overlay (mainly at grain boundaries).

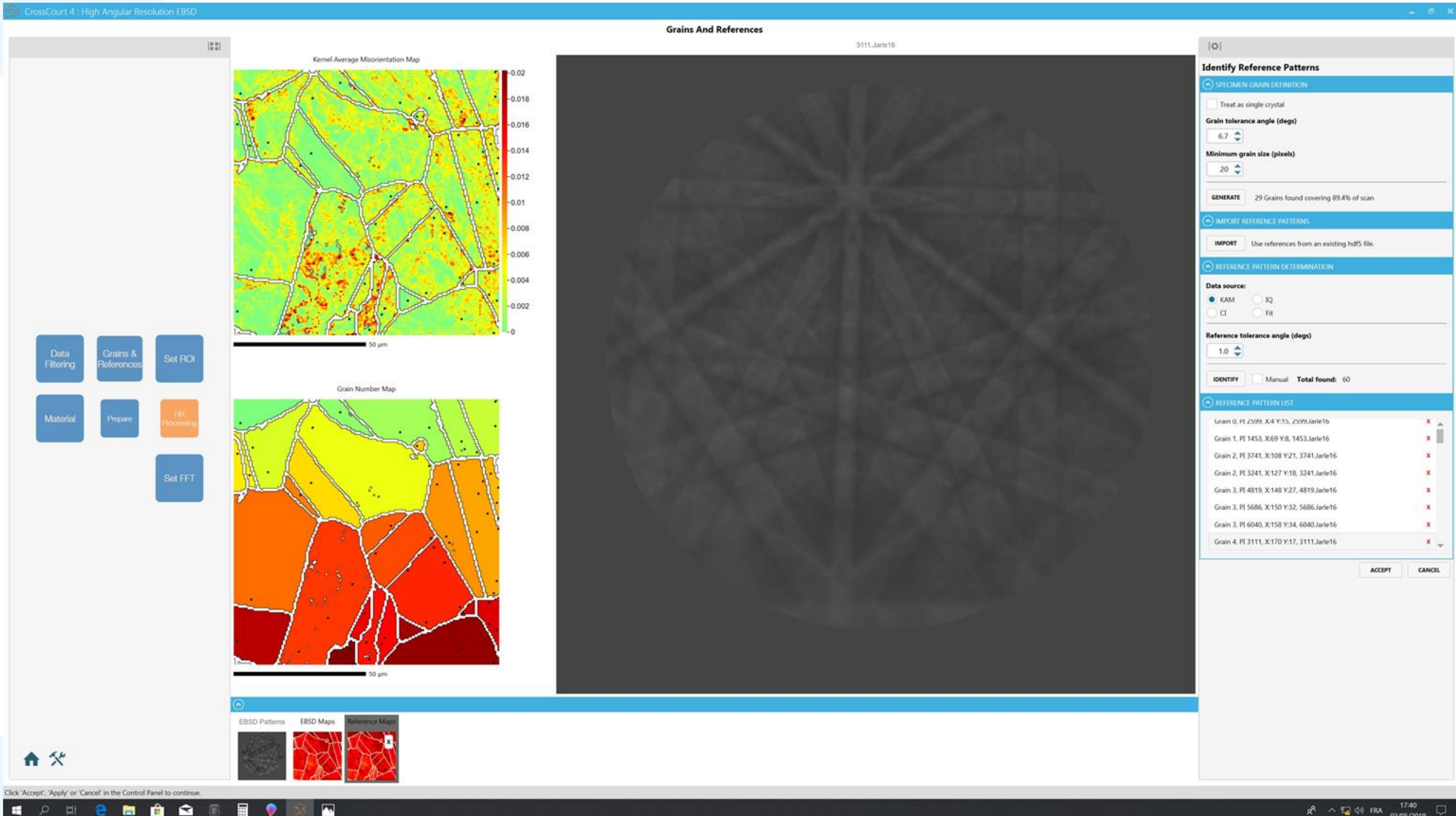


How to process HR EBSD data using CC4 software. Good practice for accurate results.

Filter pixels of large misorientation (HR KAM)

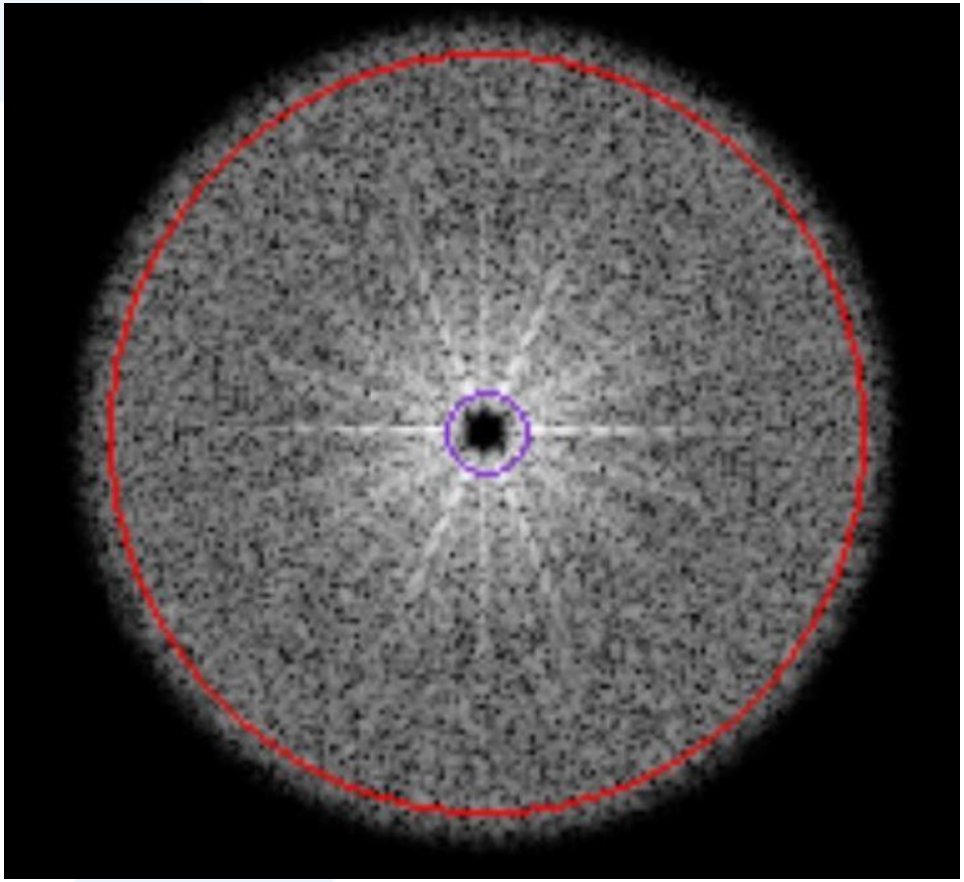
How to process HR EBSD data using CC4 software. Good practice for accurate results.

If there are misorientations higher than 1°, choose a high number of references per grain .

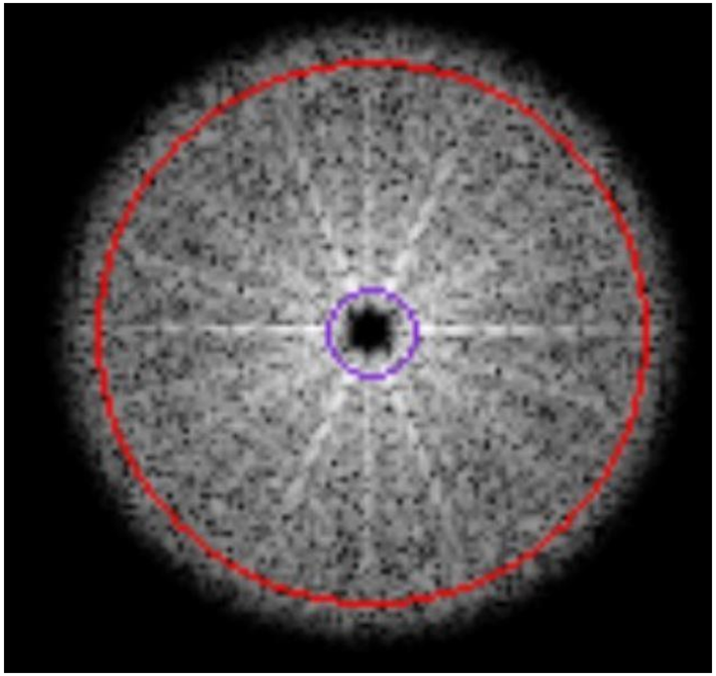


How to process HR EBSD data using CC4 software. Good practice for accurate results.

Choice of FFT filter : high frequency.



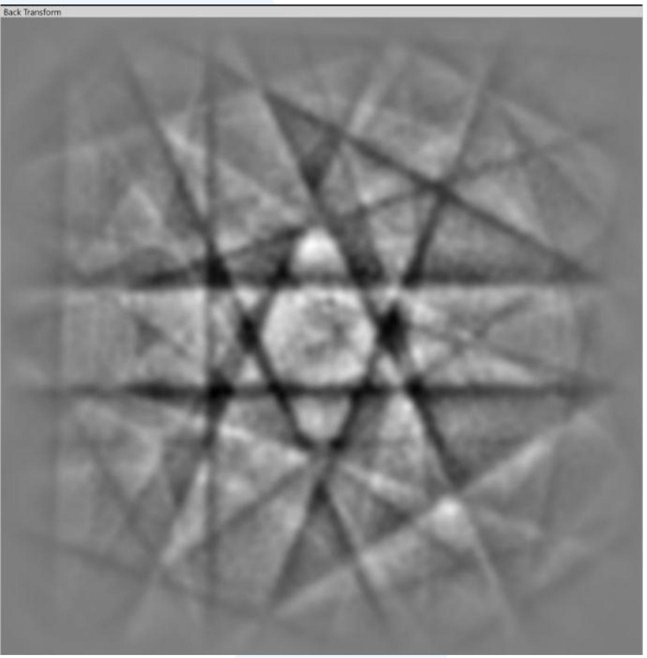
75 : too high (not necessary).



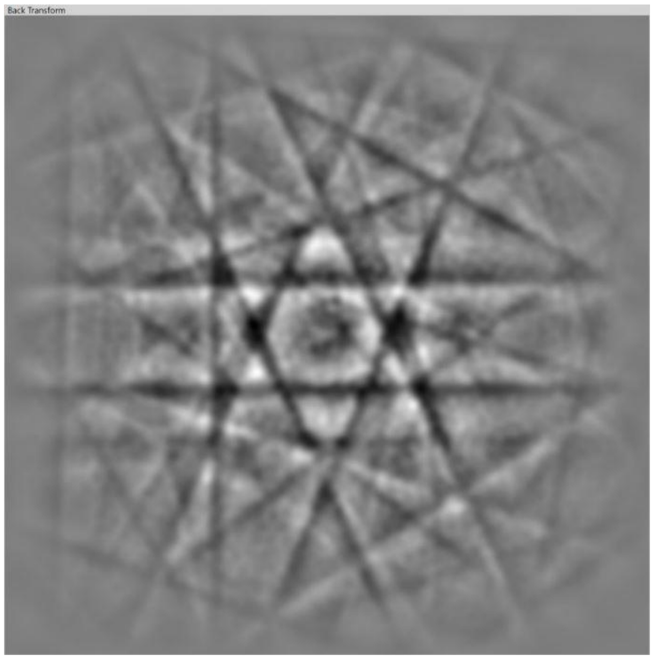
50 is sufficient.

How to process HR EBSD data using CC4 software. Good practice for accurate results.

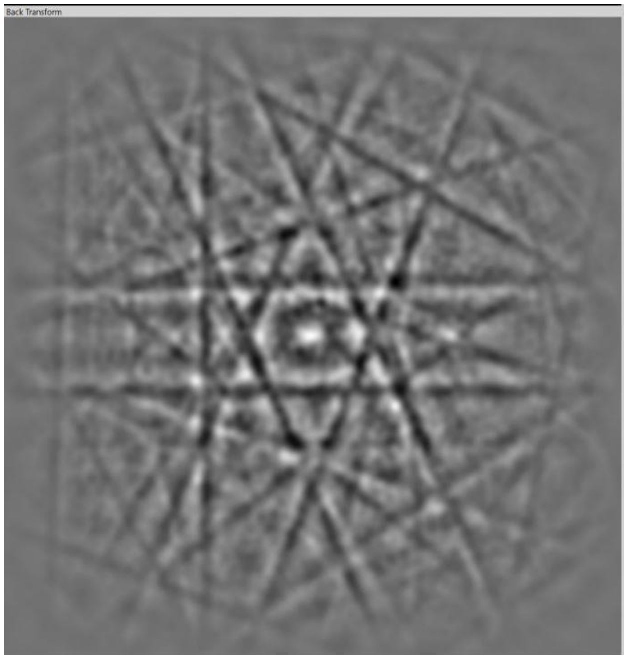
Choice of FFT filter : low frequency.



2 : too strong contrast (bad).



10: is the best value.



15: too blurry (bad).

The good value is depending on : the performance of the camera, the quality of the acquisition (noise in the pattern image) and the quality of the pattern itself (dislocations density).

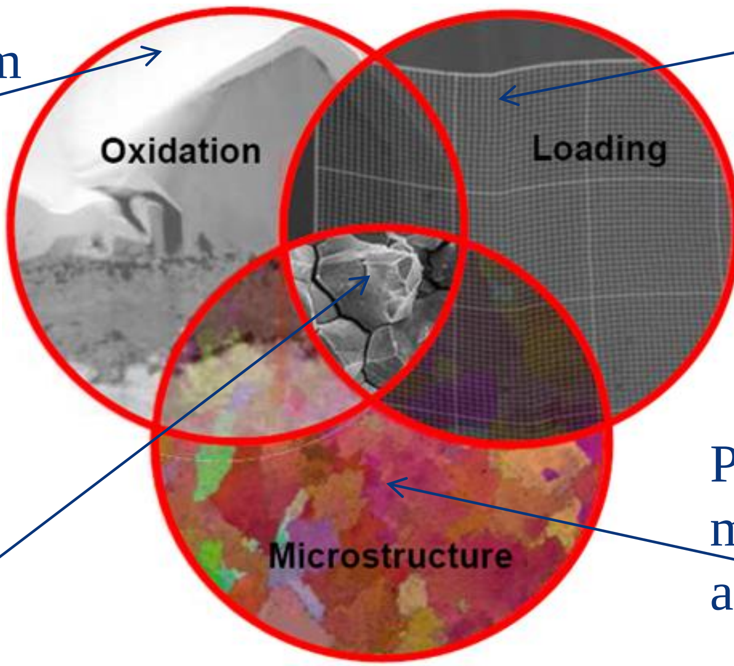
eDF R&D

Industrial context : Stress Corrosion Cracking

Stress Corrosion Cracking (SCC) is an intergranular cracking process that can lead to the failure of a component under the combined action of a corrosive environment and mechanical stress.

Mostly the applied load is less than the plastic flow stress limit.

No SCC under vacuum



the stress can come from the residual stresses due to the shaping process or be induced by the in-service loading.

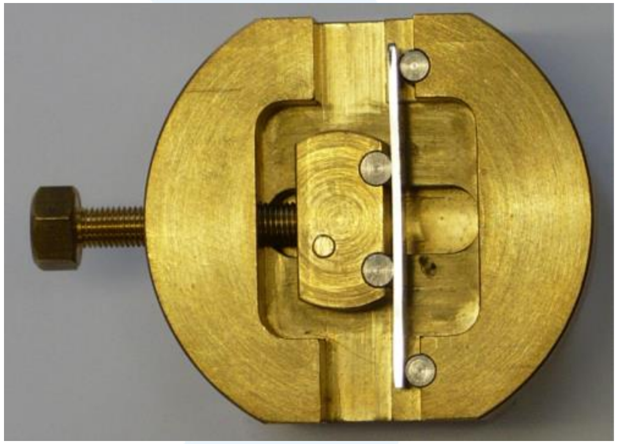
Plastic strain of the as-received microstructure or metallurgical aging plays a key role.

The SCC affects different materials (Ni, Al or Iron based alloys) used in various industries like nuclear, petrochemical, aeronautics, where the working environments widely differ (alogenuri pollution, hydrogen).

EDF R&D

HR-EBSD applications :
Bending test on a Tungsten monocrystal

Analytical solution of the stress profile through a beam.

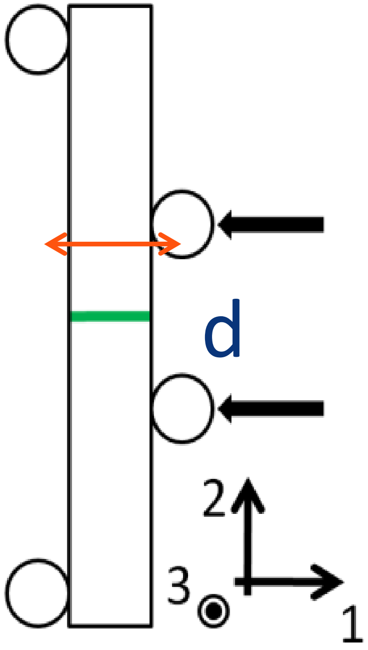


$$\sigma_{22} = -\frac{6}{5}\frac{E * u(d)}{d^2}x_1$$

$$\varepsilon_{22} = -\frac{6}{5}\frac{u(d)}{d^2}x_1$$

$$\varepsilon_{11} = \frac{6}{5}\frac{\nu * u(d)}{d^2}x_1$$

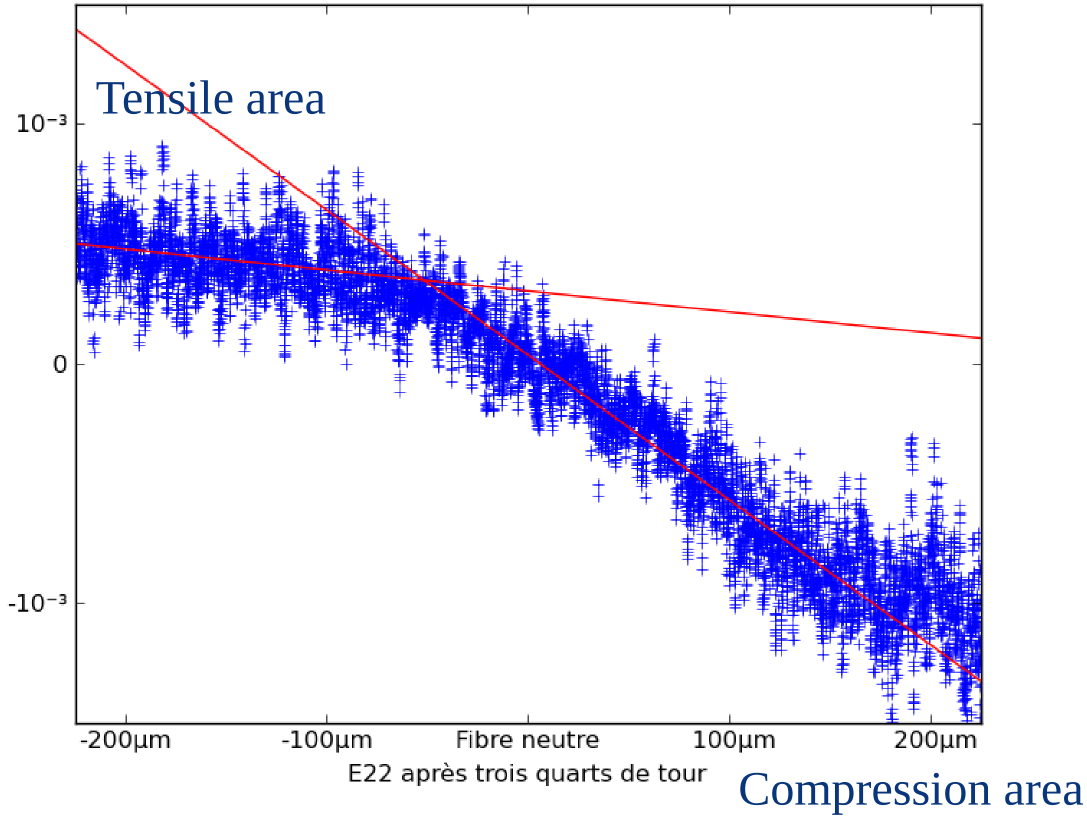
$$\varepsilon_{33} = \frac{6}{5}\frac{\nu * u(d)}{d^2}x_1$$



Bending test on a monocrystal sample of Tungsten

HR EBSD scan profile highlights a linear behavior (elastic) at the center of the beam. The red line corresponds to the elastic model.

The elastic limit is measured at 128 MPa corresponding to red lines intersection (to be compared to 150 MPa mentioned in the litterature).

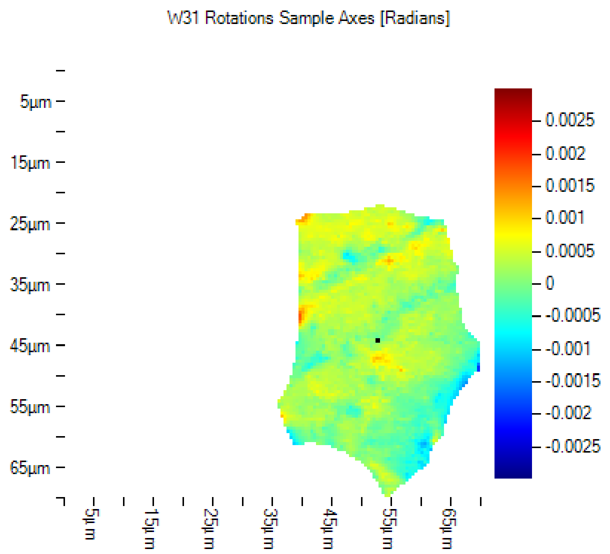


$$\sigma_{lim} = -\frac{6}{5}\frac{E * u(d)}{d^2}x_{1,lim}$$

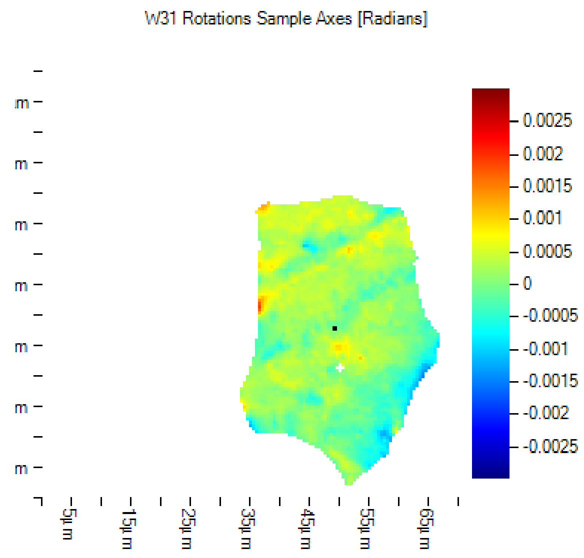
Bending test, up to rupture on a polycrystalline sample of Tungsten

The polycrystalline tungsten breaks in a brittle and intergranular manner at room temperature. This rupture occurs without any plasticity.

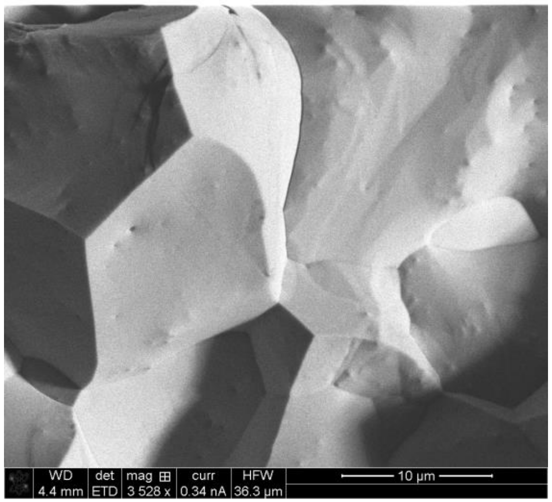
No crystalline rotation should appear during loading.



Before flexion



After loading

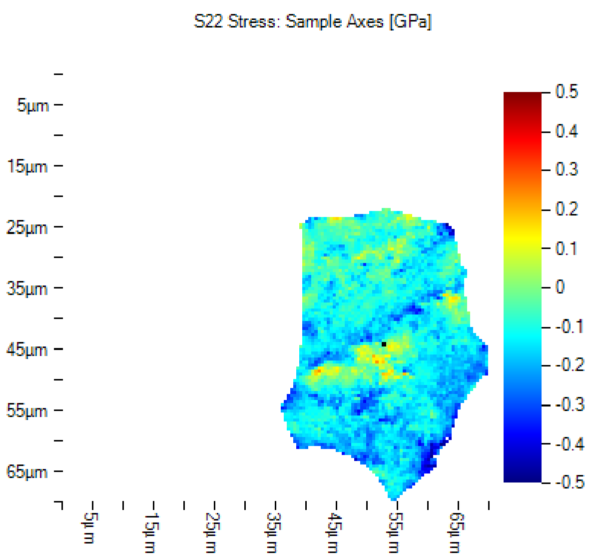


Intergranular rupture

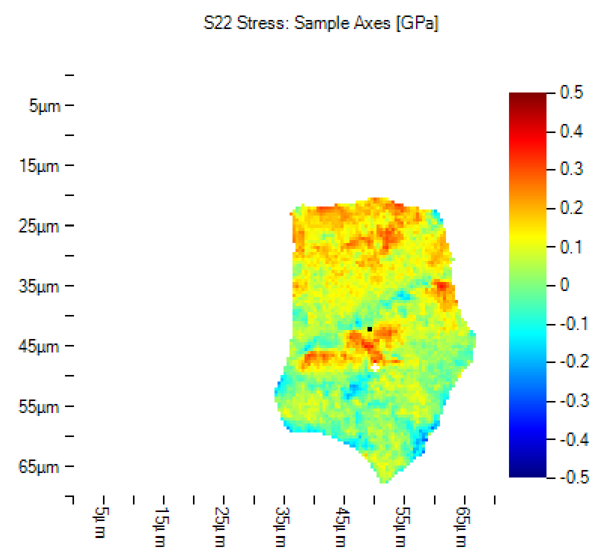
Bending test, up to rupture on a polycrystalline sample of Tungsten

The grain analyzed in the tensile zone shows a strong evolution of S22, but very weak gradients of misorientation.

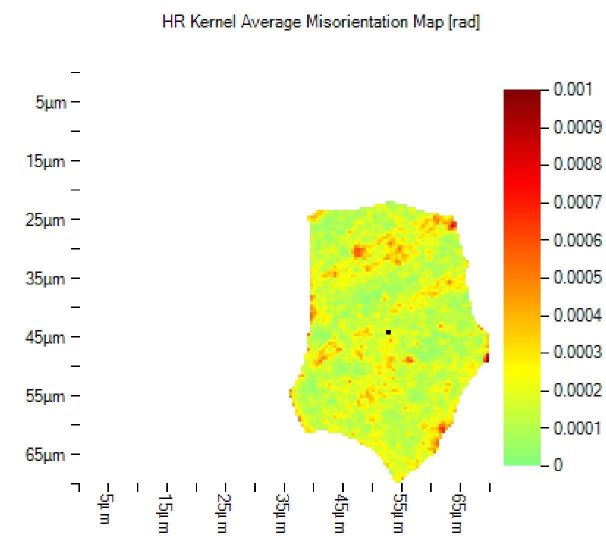
Very little intergranular plasticity just before rupture, all the stress is reported to the grain boundaries.



Traction component before flexion



Traction component after flexion



HR KAM just before rupture

Conclusions

HR EBSD is the dedicated technique allowing the study of the stress concentration at grain boundary.

Sample preparation and acquisition conditions must be optimal.

HR EBSD is a relative technique and does not provide absolute stress measurements.

Measurement of stress in the presence of plasticity requires a suitable post-treatment.

Wide field scan analysis is possible but subject to a good knowledge of the projection parameters (PC).

Thank you for your attention

And thanks to :

Lucie Saintoyant

Qiwei Shi

David Dingley

Graham meaden

Damian Dingley

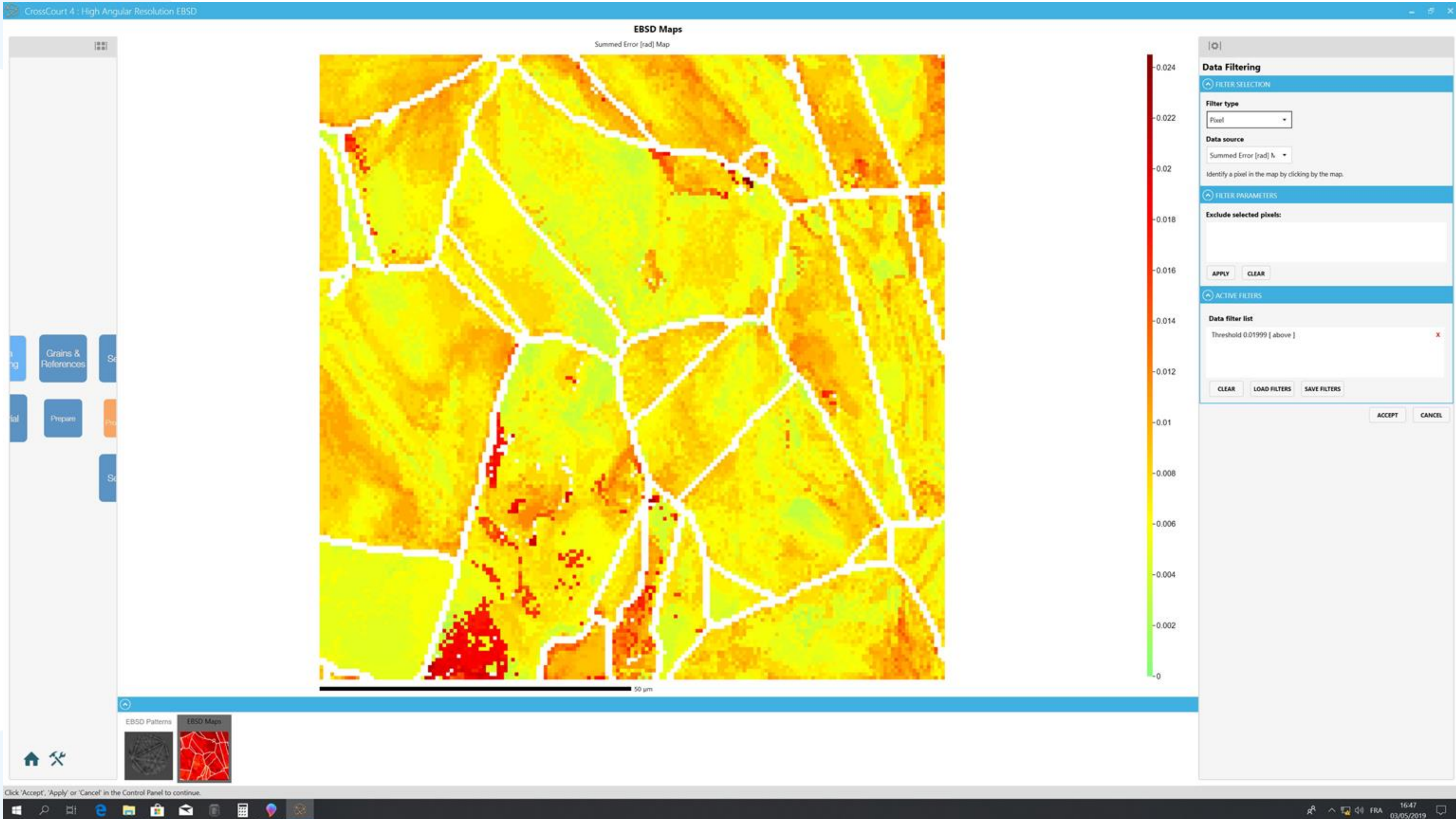
Claire Maurice

Emeric Plancher

François Brisset

How to process HR EBSD data using CC4 software. Good practice for accurate results.

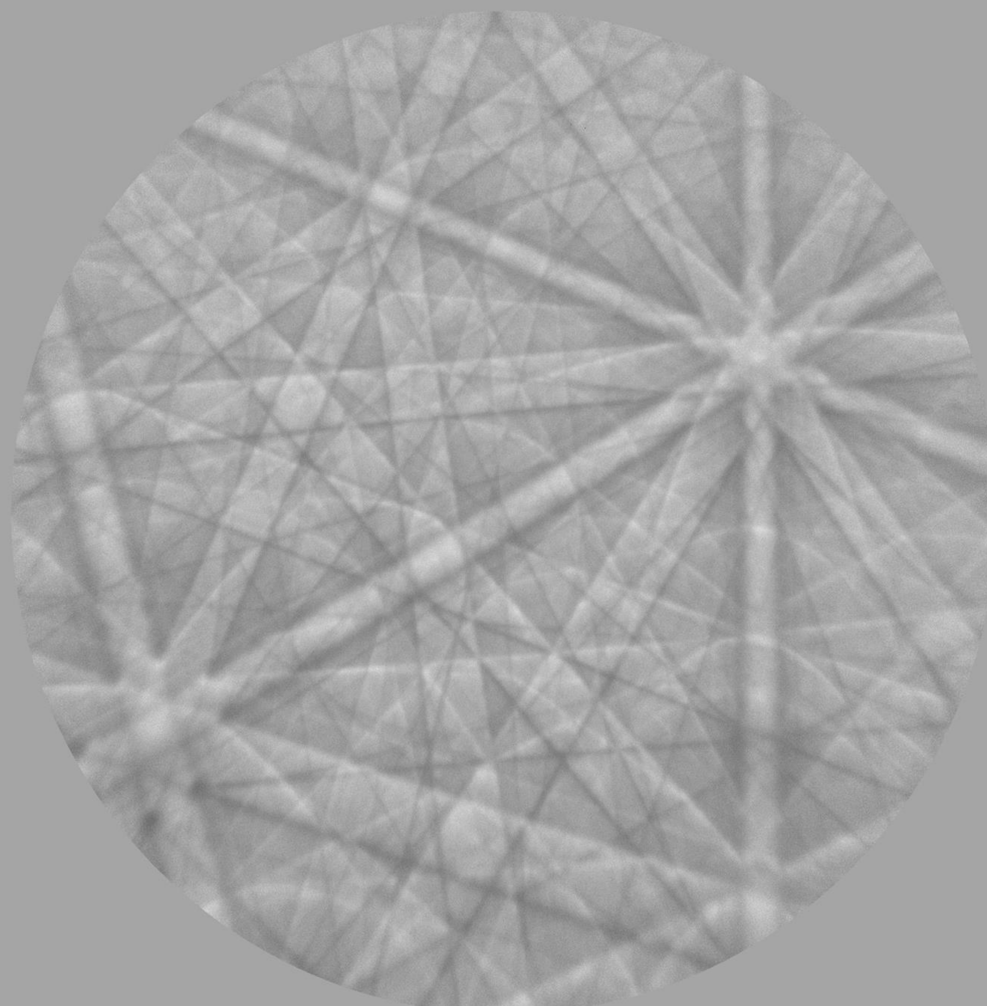
Filter poor quality individual pixels (summed error map)



Why use Kikuchi patterns of the highest quality ?

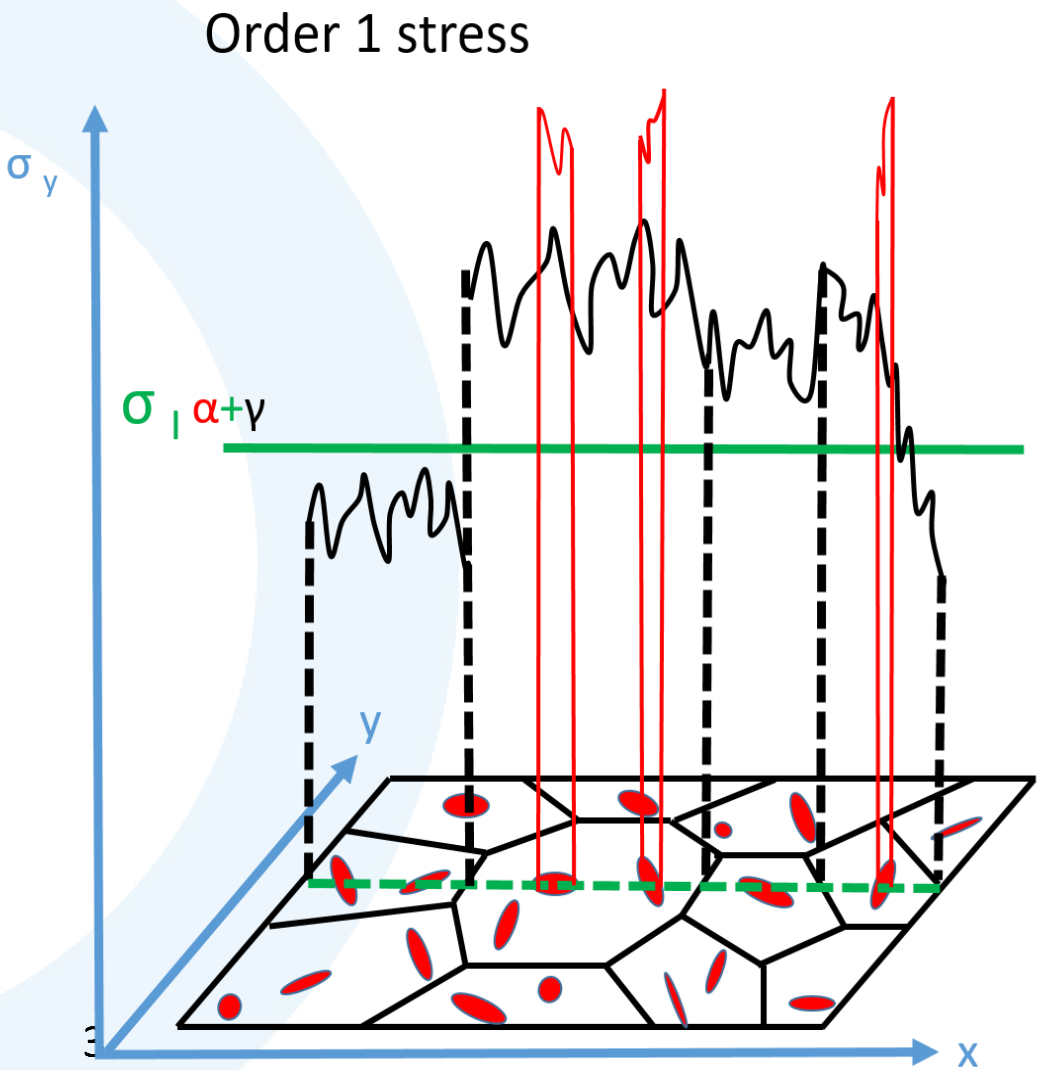
The HR EBSD technique is based on image processing measurements on all the pixels contained in the pattern and not only on the positions of a few bands.

The higher the quality of the patterns is, the more sensitive the technique will be.



Pattern of a nickel base alloy (image size 4K pixels).

Stress appears at different scales of space, which can be divided by 3 orders of stress.



Example of two-phases microstructure

The first order stress corresponds to the average stresses on all the metallurgical components of the microstructure.

This order is applied on a macroscopic scale (more than one square cm).

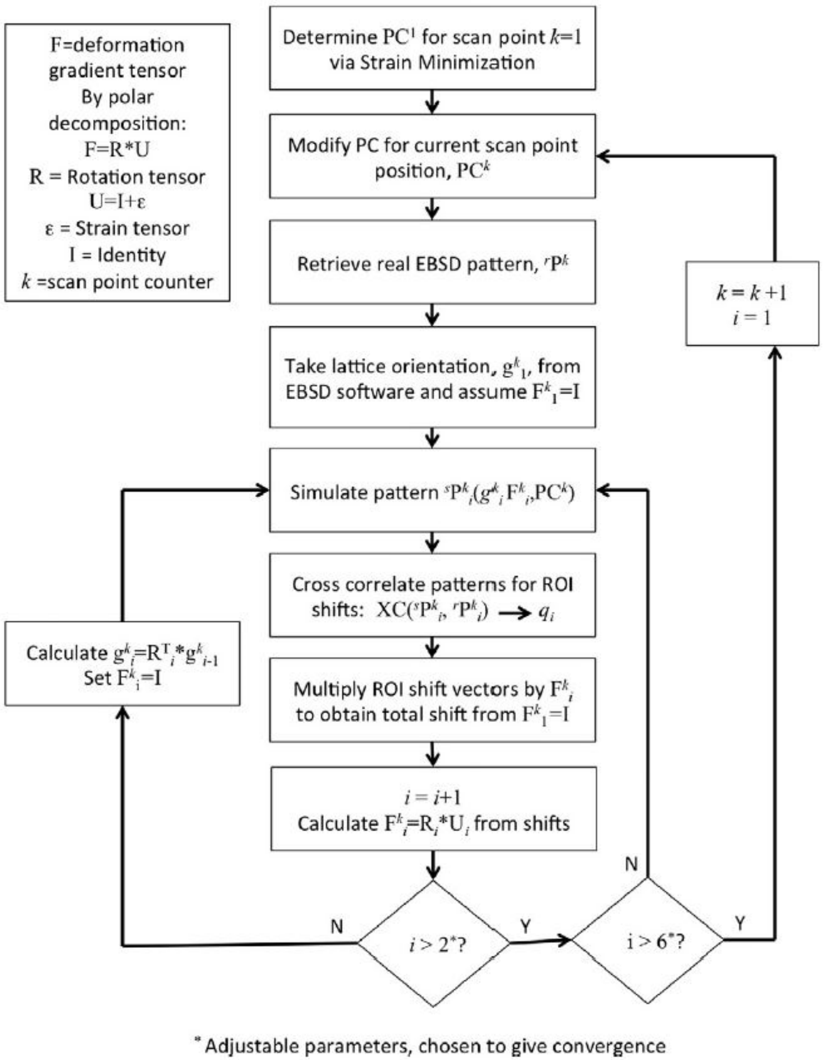
Is an absolute measure of stress possible?

Need an iterative improvement algorithm of projection parameters (PC).

Validation of kinematically simulated pattern HR-EBSD for measuring absolute strains and lattice tetragonality

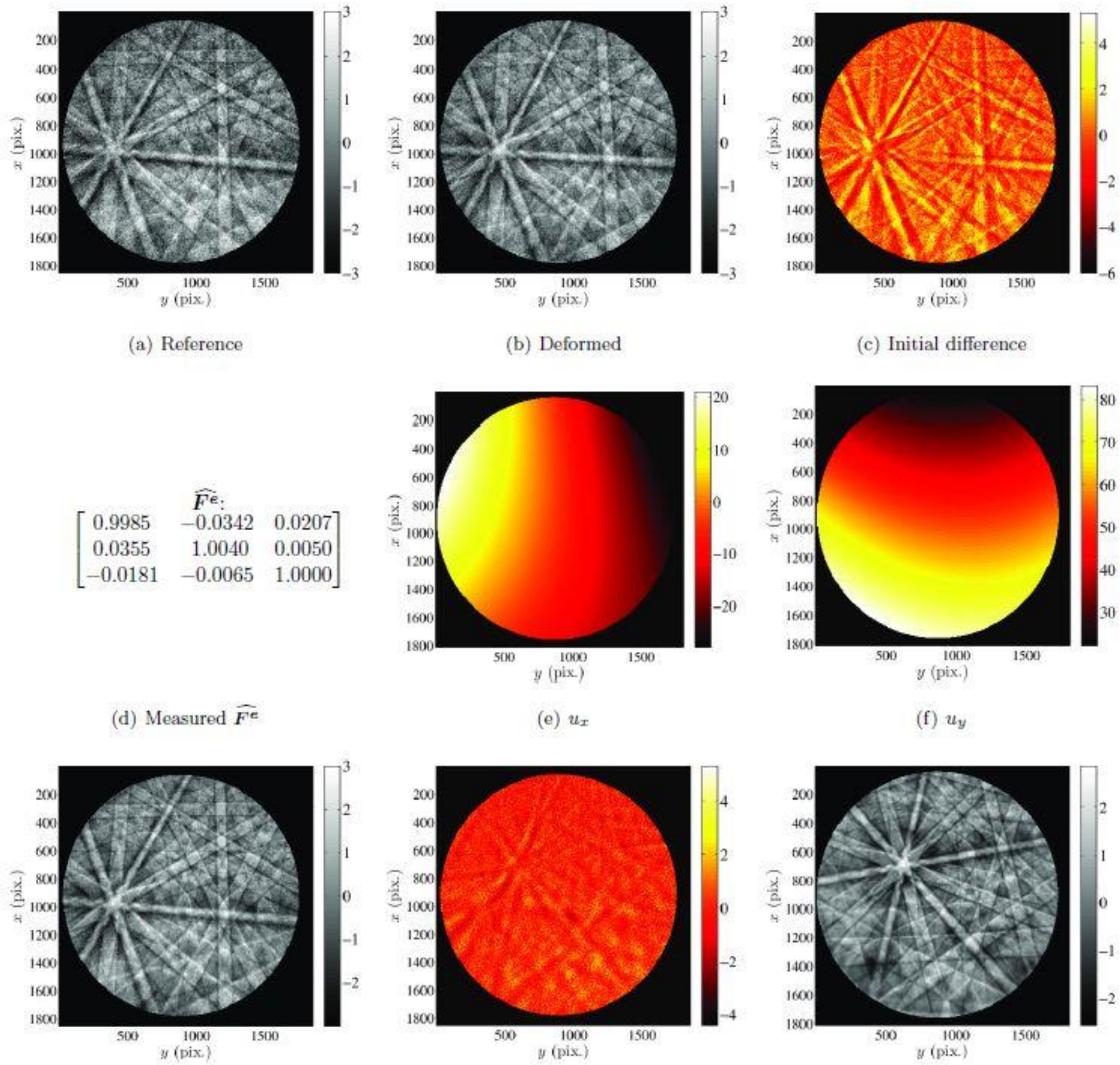
David Fullwood a, Mark Vaudinb, Craig Daniels a, Timothy Ruggles a, Stuart I.Wright c

Materials Characterization 107 (2015) 270–277



Absolute measure of stress.
Integrated Digital Image Correlation (IDIC)

IDIC is faster than the FFT cross correlation on synthetic and real pattern.



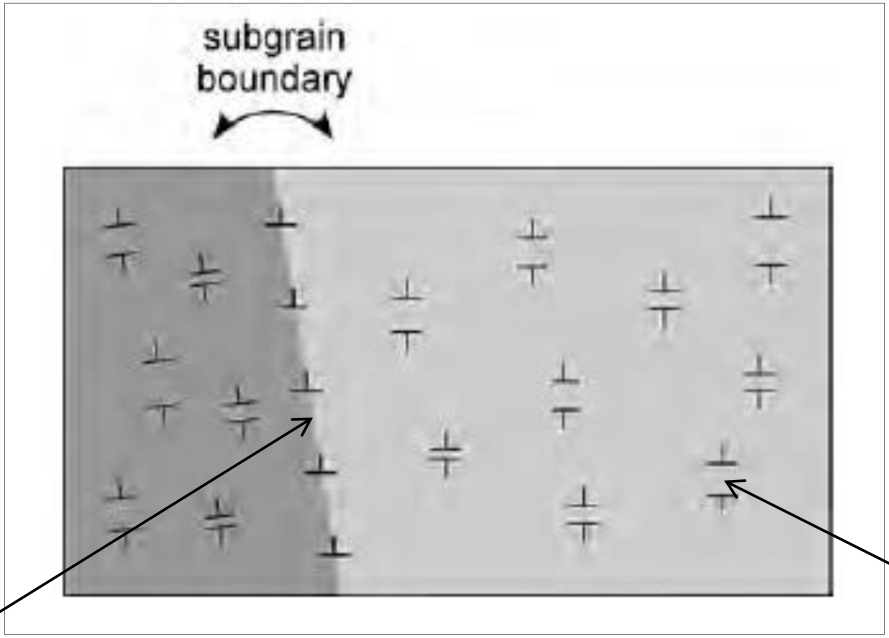
(a) Reference (b) Deformed (c) Initial difference

(d) Measured $\widehat{F^e}$ (e) u_x (f) u_y

(g) Corrected deformed image (h) Residual (i)

Estimation of plasticity : measurement of GND density

For the first time in 1953, Nye introduced the concept of GND.
Dislocations can be splited into two populations: GND and SSD



Geometrically necessary dislocations (GND) form a low misorientation within a crystal.

Burgers vectors vanish for statistically stored dislocations (SSDs)

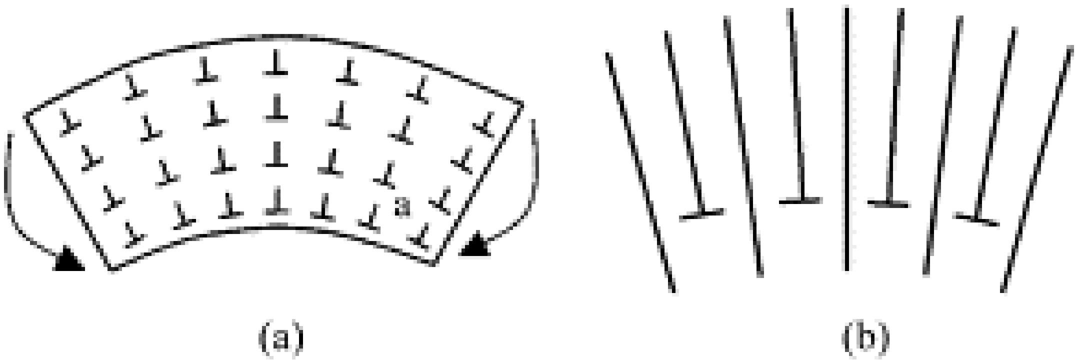
Principle of GND density measurements

Nye's work aims to relate the tensors of dislocations with the density of dislocations of different types.

The tensor of Nye is written: $\alpha_{ij} = \Sigma\ \rho k b k_i z k_j$

Or ρ is the scalar density of dislocations.

The couple $bk_i zk_j$ specifies a particular dislocation type where b is the Burgers vector and z is the dislocation line.



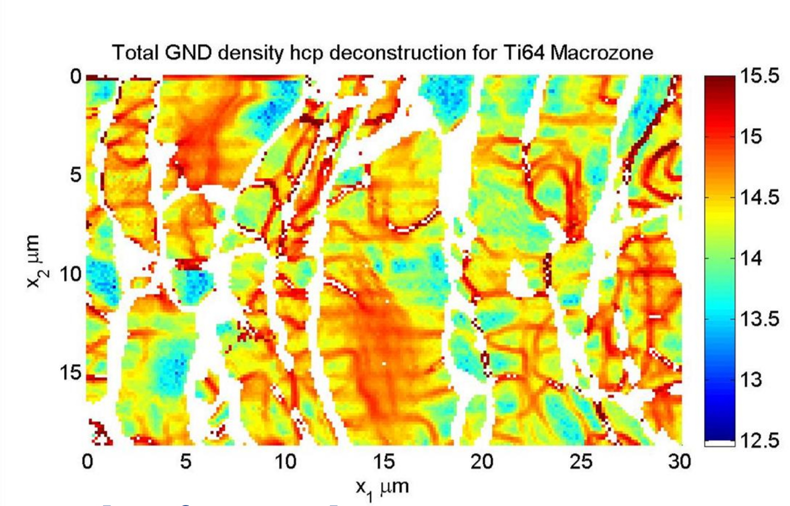
Sketch of GND induced by a bending type stress

Principle of GND density measurements

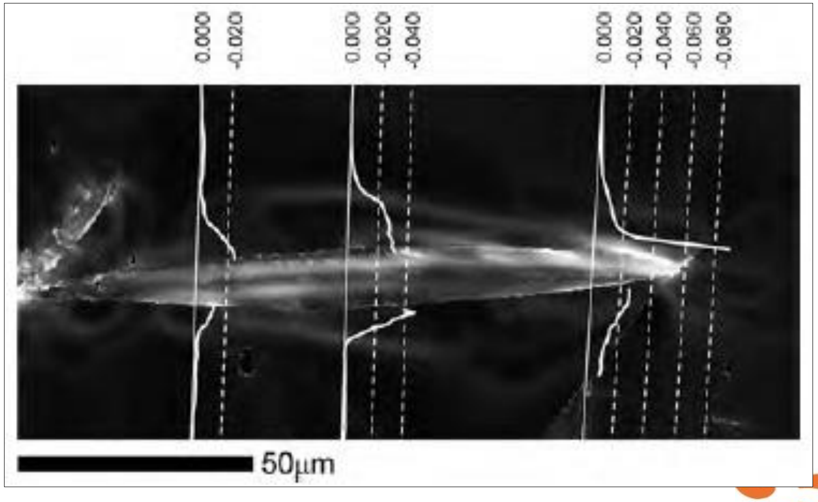
The EBSD allows the measurement of rotations between a couple of points : $\Delta\theta_\iota = \kappa_{ij}\delta x_j$

The Nye tensors are related to crystalline rotations by the following relation : $\kappa_{ij}=-\alpha_{jj} + {}^{1}/_{2}\delta_{ji}\alpha kk$

For a given combination of parameters b and z, the sum of the length of the dislocations in the field can be calculated, this allowing to obtain the dislocation density associated with a type of Burgers vector.



Example of GND density mapping, 10 x m-2



Profile of the dislocation tensor (w23) in the vicinity of a martensite lath.

Principle of GND density measurements

The first measurements of GND were made by El-Dasher in 2003. These measurements are highly dependent on the mathematical formalism used (taking into account or not the elastic deformation measurements). Moreover they suffer from the limitation of the spatial resolution and the surface character of the EBSD.

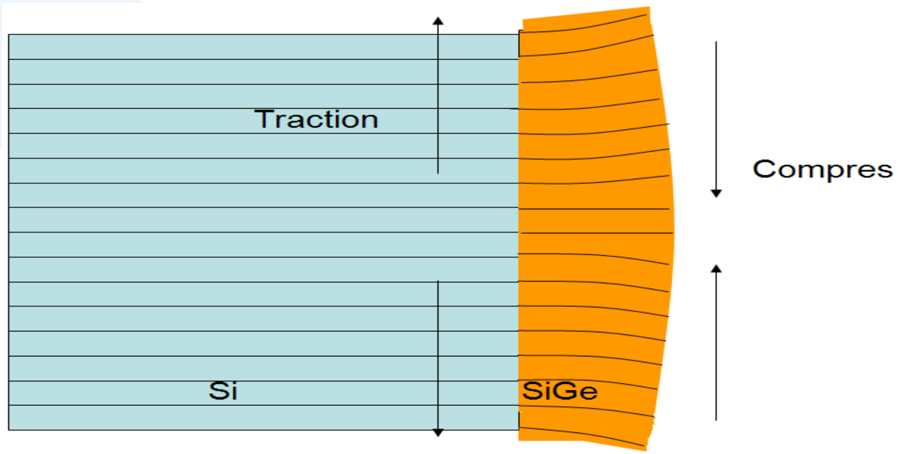
Z the dislocation line is a three-dimensional datum (inaccessible), most models make an approximation of this magnitude. However, EBSD can be extended to the third dimension by successive ion beam cross sections [Zaafarani 2006].

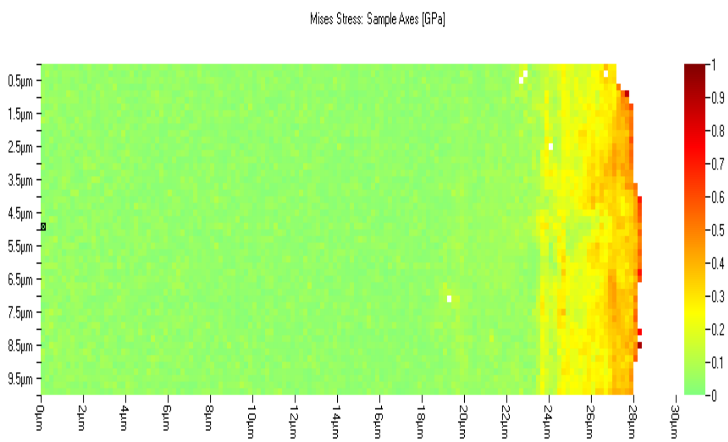
Finally, the relaxation effect related to the free surface is important, but it is possible to estimate the relaxed depth using digital simulation by dislocation dynamics [D.P.Field 2009 Phil Mag]

The angular resolution of the HR-EBSD greatly improves the sensitivity of geometrically necessary dislocation density measurements compared to the measurements made by the standard EBSD.

eDF R&D

Standard sample for HR EBSD : Silicon-Germanium epitaxial layers





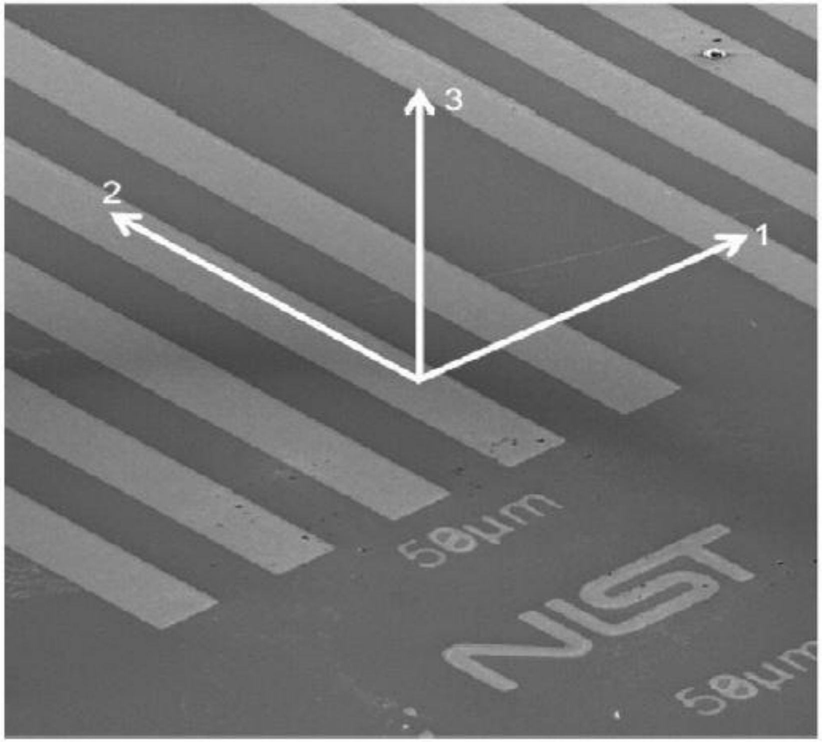


Fig. 2. SEM image of the end of five 50 µm wide 50 nm thick $Si_{1-x}Ge_x$ stripes on a Si substrate. The coordinate system for strain measurement is indicated.

Von Mises map of Si

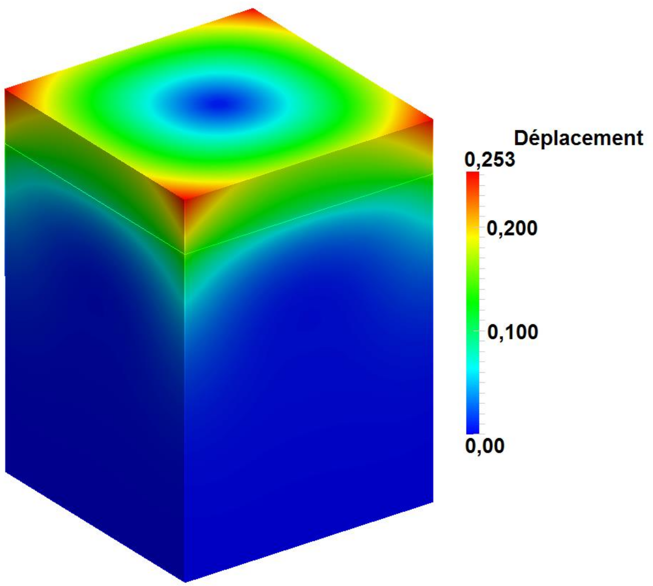
Standard sample for HR EBSD : Silicon-Germanium epitaxial layers

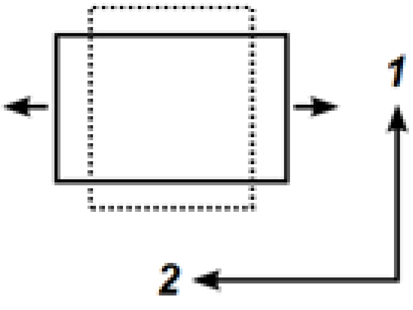
Check the coherence of the experimental results with a finit element simulation :

The strain e22 is representative of the lattice mismatch between Si and SiGe

Silicon is in traction near the interface

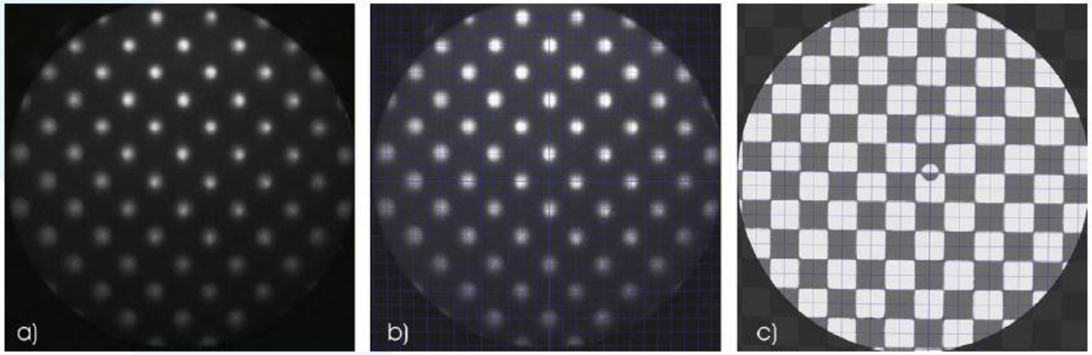
The strain e11 has a behavior opposite to that of e22 because of the « Poisson effect »

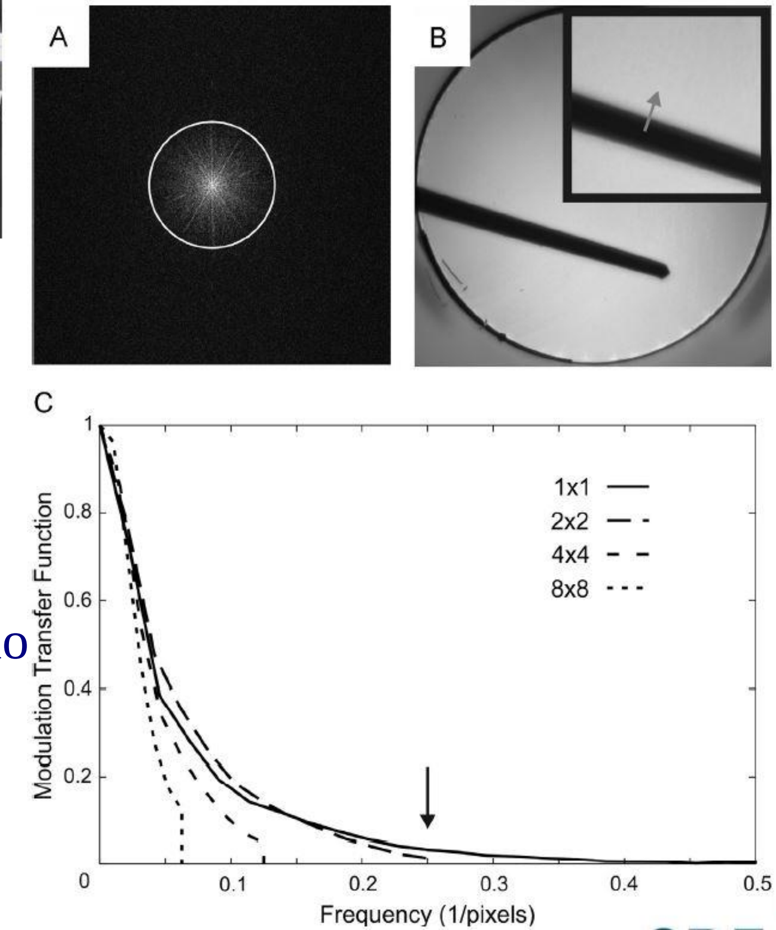




Performances of the camera

To use pattern matching, it is necessary to know the characteristics of the camera.





Mingard, Day, Maurice, Quested,

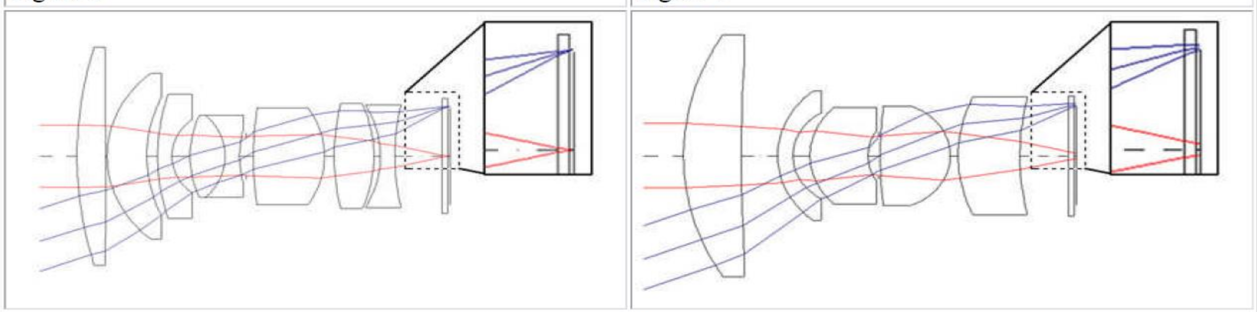
Ultramicroscopie 2011

Need to characterize the aberrations and the modulatio transfer function (MTF) of the optical system.

Performances of the HR4M camera

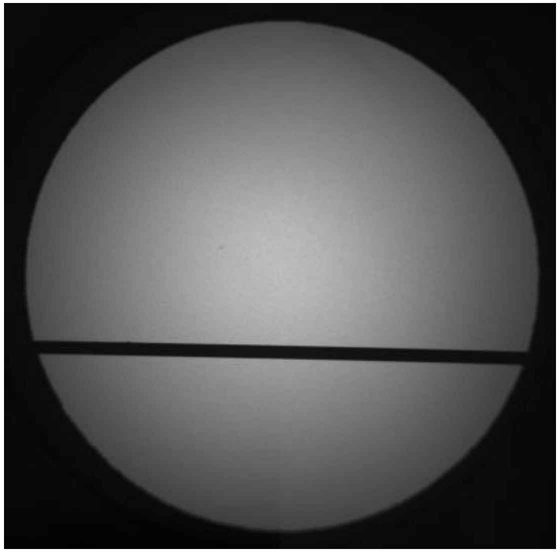
The HR4M camera has megapixel optics

Figure 1 | Figure 2

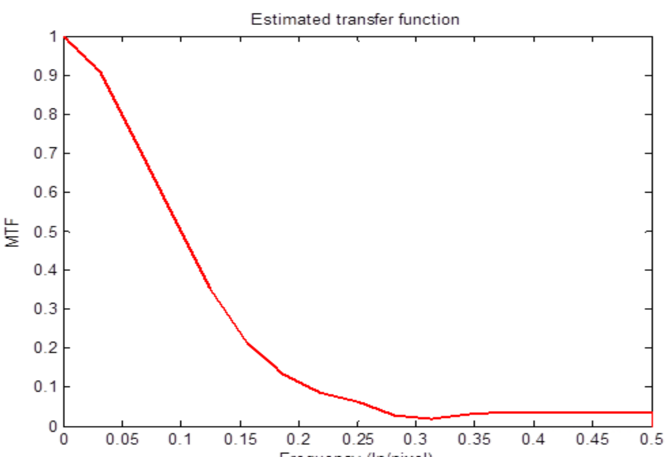


Spot size of a megapixel lens is much smaller, required for good focus on megapixel sensors.

Spot size of a standard lens won't allow sharp focus on a megapixel sensor.



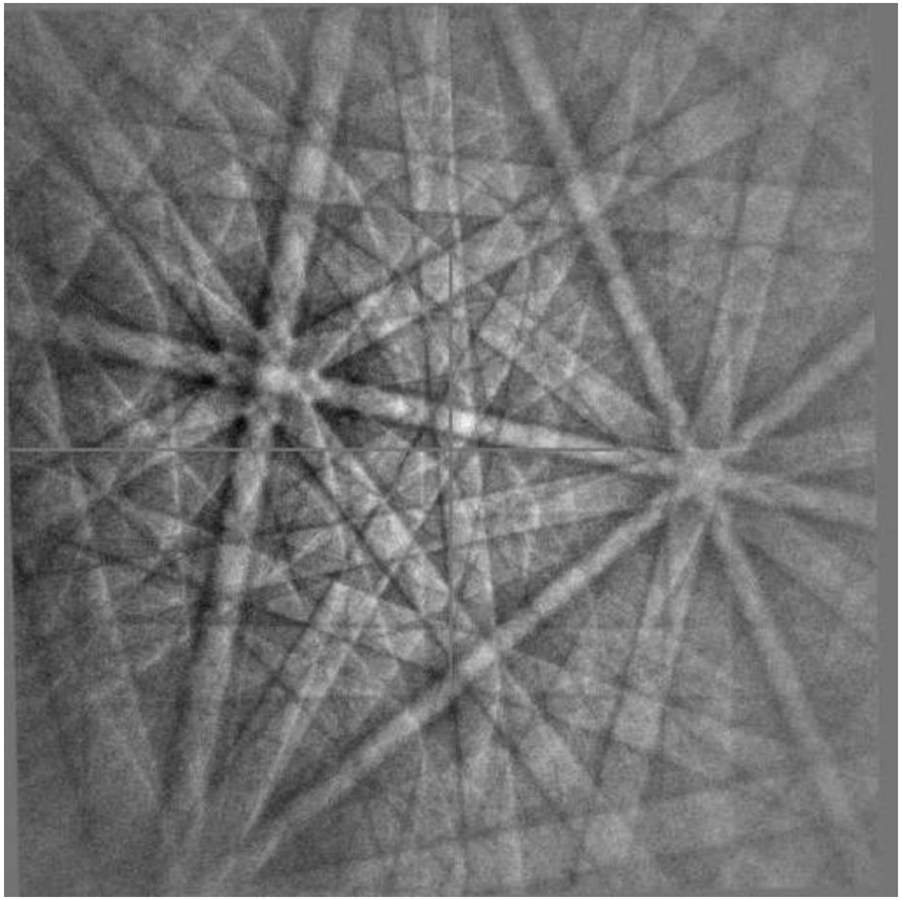
HR4M improved MTF but complex aberrations still need to be corrected.



What about new generations of cameras ?

CMOS cameras with optical fiber screen coupling must be tested.

Direct detection has no aberration, but consists of four non-perfectly joined quadrants.

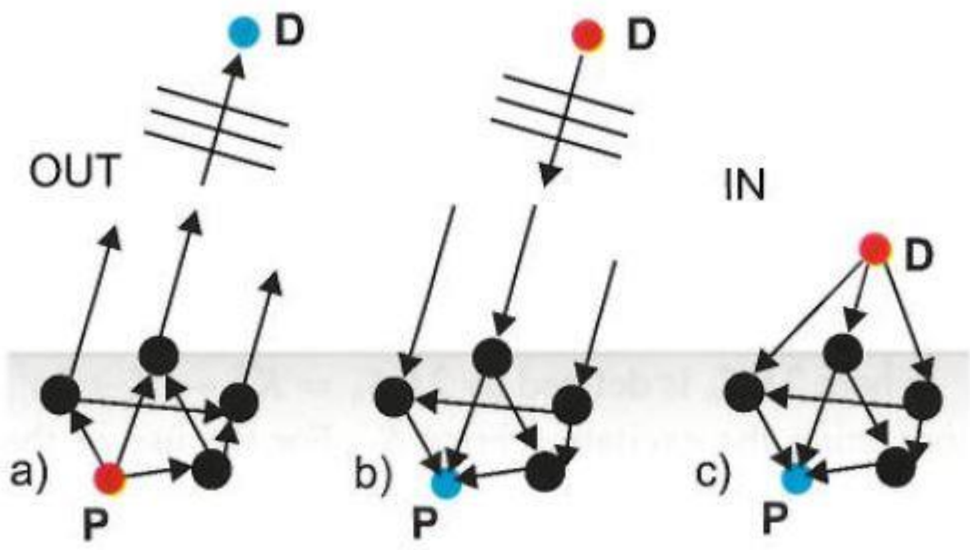


HR EBSD Kikuchi patterns : the physics

The EBSD patterns are produced mainly by the incoherent scattering of an electron beam interacting in a crystalline volume. This source of primary electrons comes from elastic and inelastic scattering.

Less than ten percent of the total backscattered electrons are channeled into high and low reflections to form a Kikuchi pattern. We know that backscattered electrons that have diffracted come mainly from elastic scattering.

The principle of reciprocity allows us to understand the similarities between the EBSD method and the electron-channelling pattern (ECP).



HR EBSD Kikuchi patterns :

influence of the beam divergence

The divergence of the beam influences the signal contrast of the lines inside the Kikuchi bands.

The more the beam is divergent, the more the lines contrast contained in the bands will be clear.

This goes against the idea of focusing on the surface of the sample so that it appears clear.

For HR EBSD, it is necessary to have a beam as divergent as possible.

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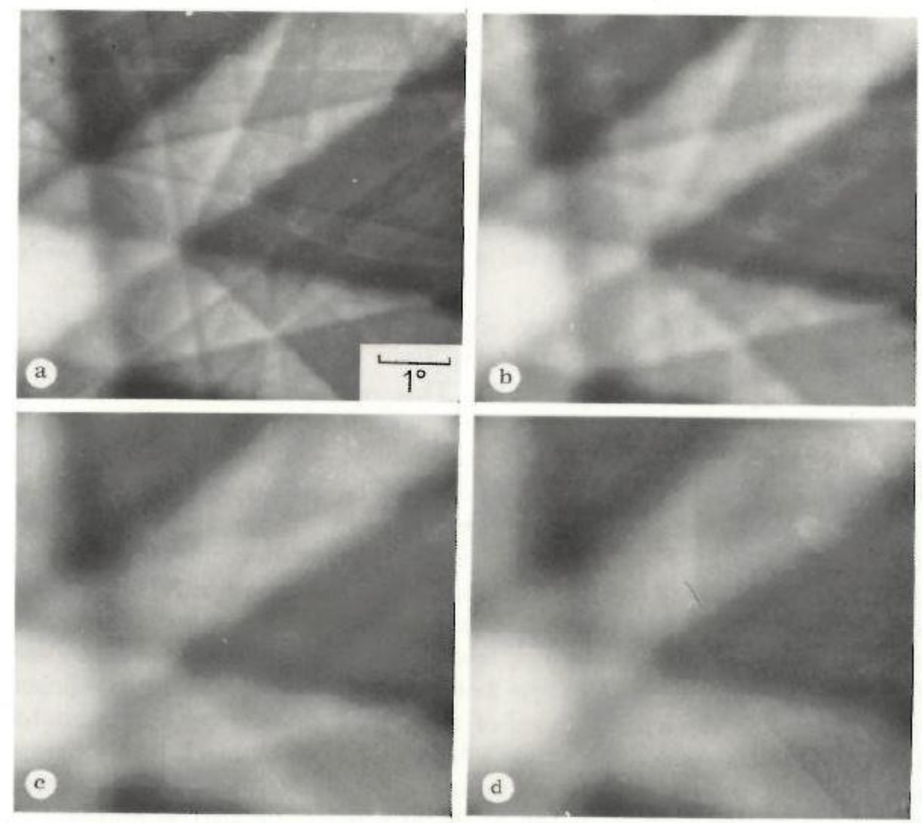


FIGURE 8. Effect of increasing beam divergence (decollimation) on electron channeling pattern quality. The divergence changes from (estimated) 2×10^{-3} rad in (a) to 10^{-2} rad in (d).

HR EBSD Kikuchi patterns :

recommendations of the scanning mode

Generally, the divergence of the beam causes a loss of spatial resolution.

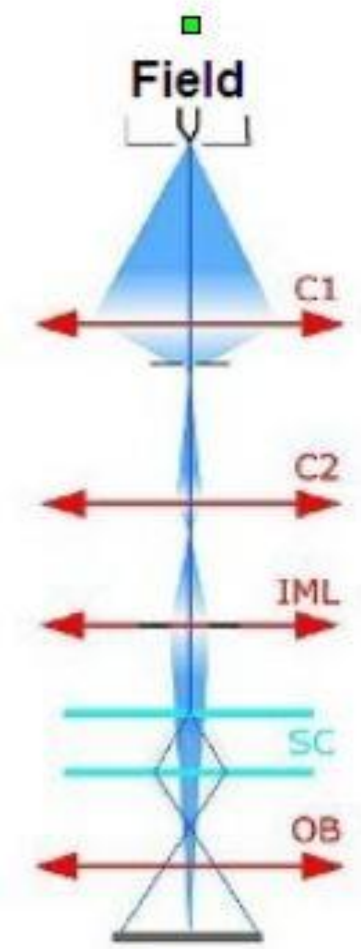
The Field mode of the TESCAN microscope produces an almost parallel beam that does not degrade a lot the spatial resolution.

This mode has a such depth of field that the surface of the sample is sharp and the patterns contain details. It corresponds to the least divergent beam and presents the best compromise :

High probe current / small probe size / lowest convergence

On other microscopes, you should find the best mode (most of the time this configuration is performed using a small condenser aperture and a long working distance).

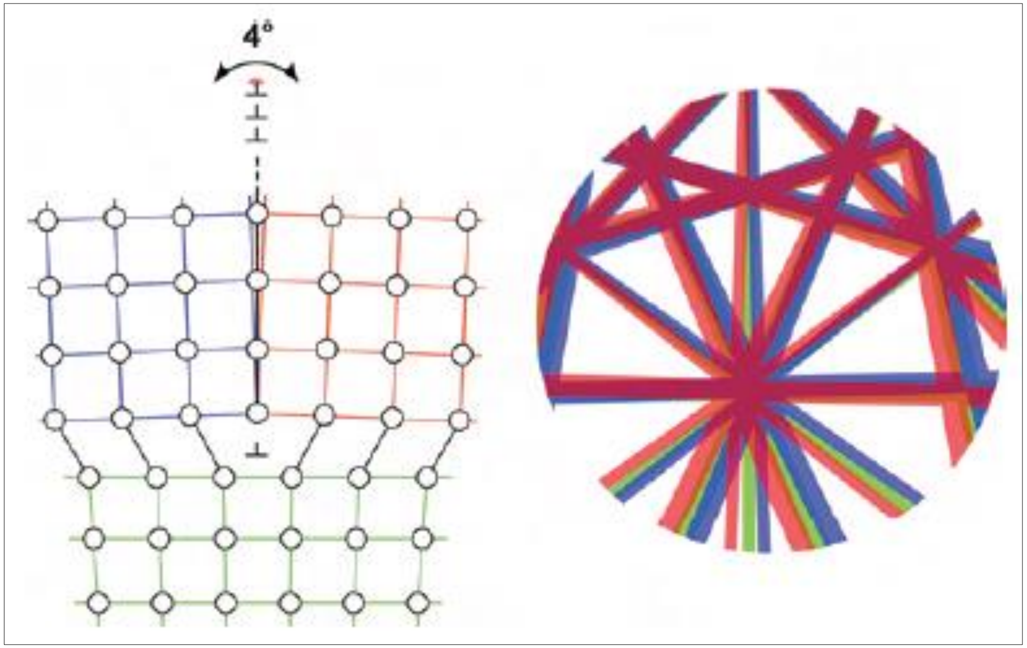
Take care that patterns should be sharp but not necessary the sample surface !



What is the effect of the plastic strain on the crystal ?

A crystal under load in the plastic domain, produces lattice rotations within grains. This effect decreases in the quality of the pattern (IQ, BS) related to the presence of dislocations.

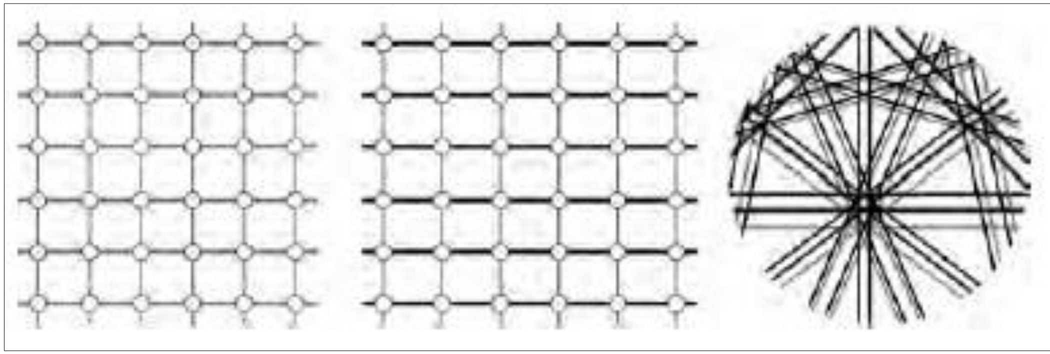
The rotation of a crystalline domain with respect to the other domains, is measured by the local misorientation, which allows the calculation of the density of GND.



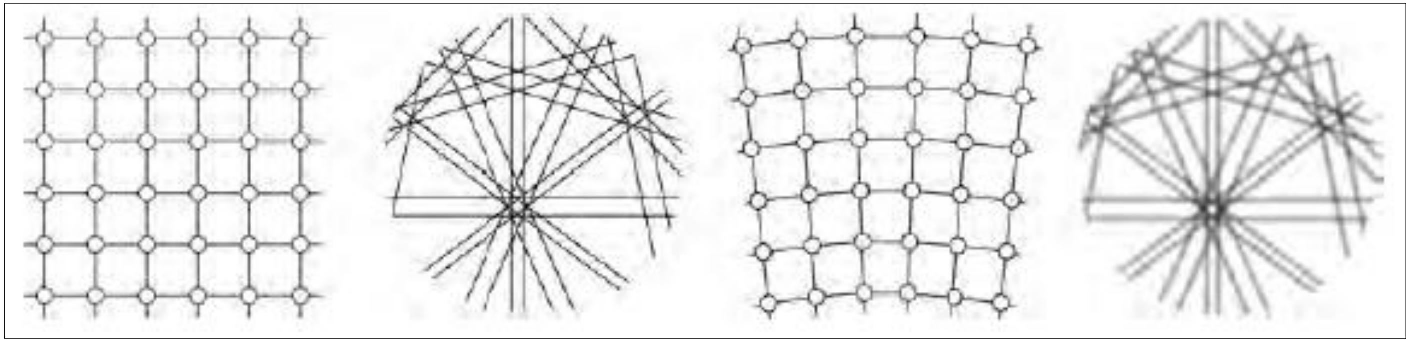
What is the effect of stress on the distortion of the crystal unit cell ?

There is a change in bandwidth and blurring of Kikuchi bands, as well as the position of the zone axes relative to each other changes.

Tensile direction ↔

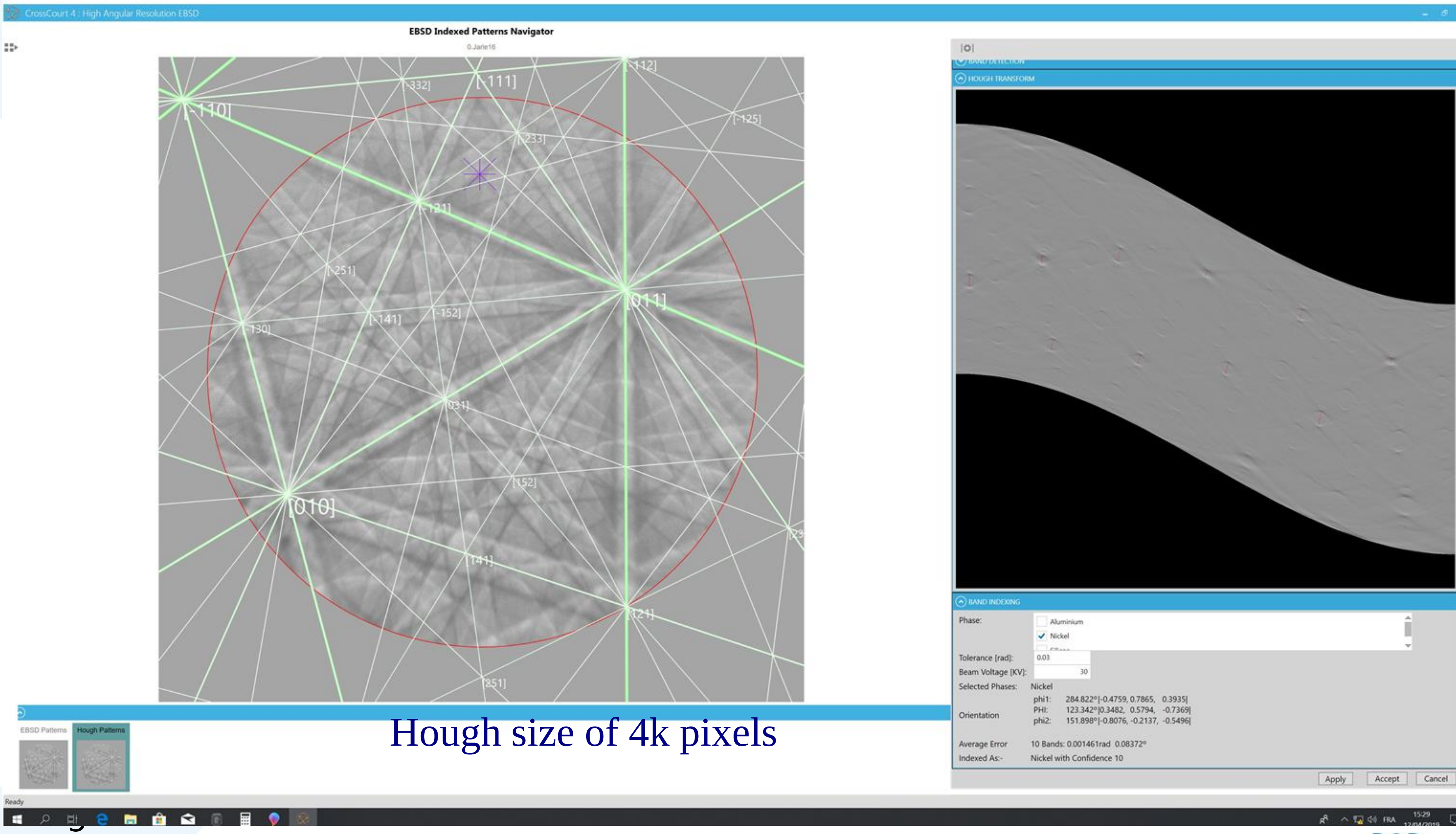


Flexion



The change of some lattice parameters results from a greater distribution of the angles, for which the crystallographic plans diffract. According to Bloch's wave theory, this distribution limits wave propagation, which reduces the contrast at the boundary of the Kikuchi bands.

Use high resolution patterns to improve absolute orientation accuracy.



Hough size of 4k pixels

R&D

Use pattern matching between real patterns and synthetic patterns simulated by the dynamical theory.

Gert Nolze a,n, AimoWinkelmann b, AlanP.Boyle
Ultramicroscopy160(2016)146–154

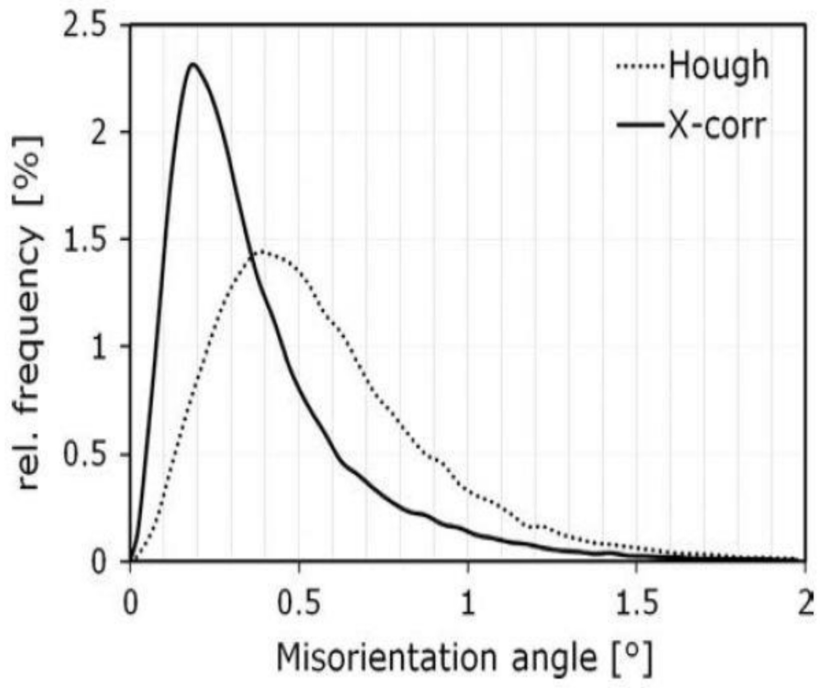
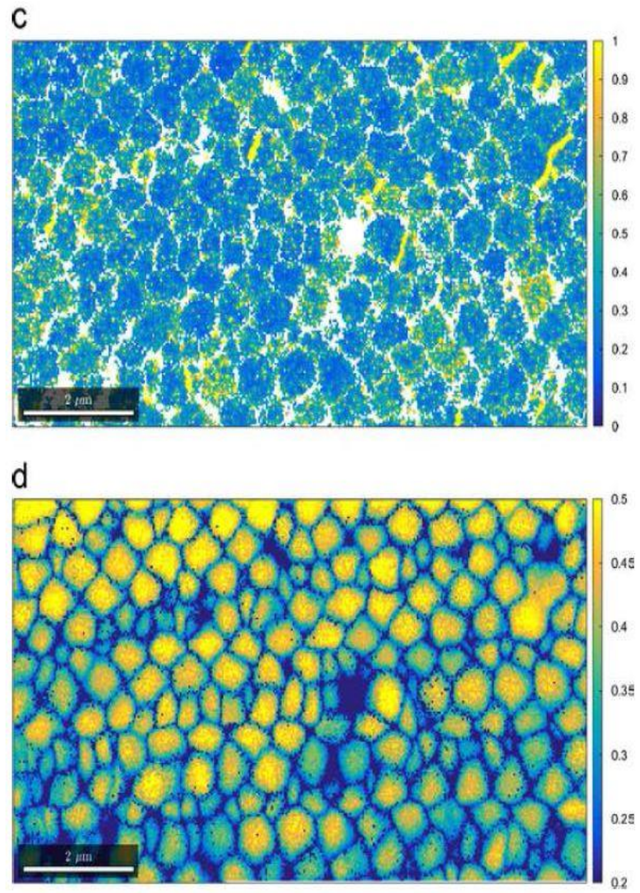
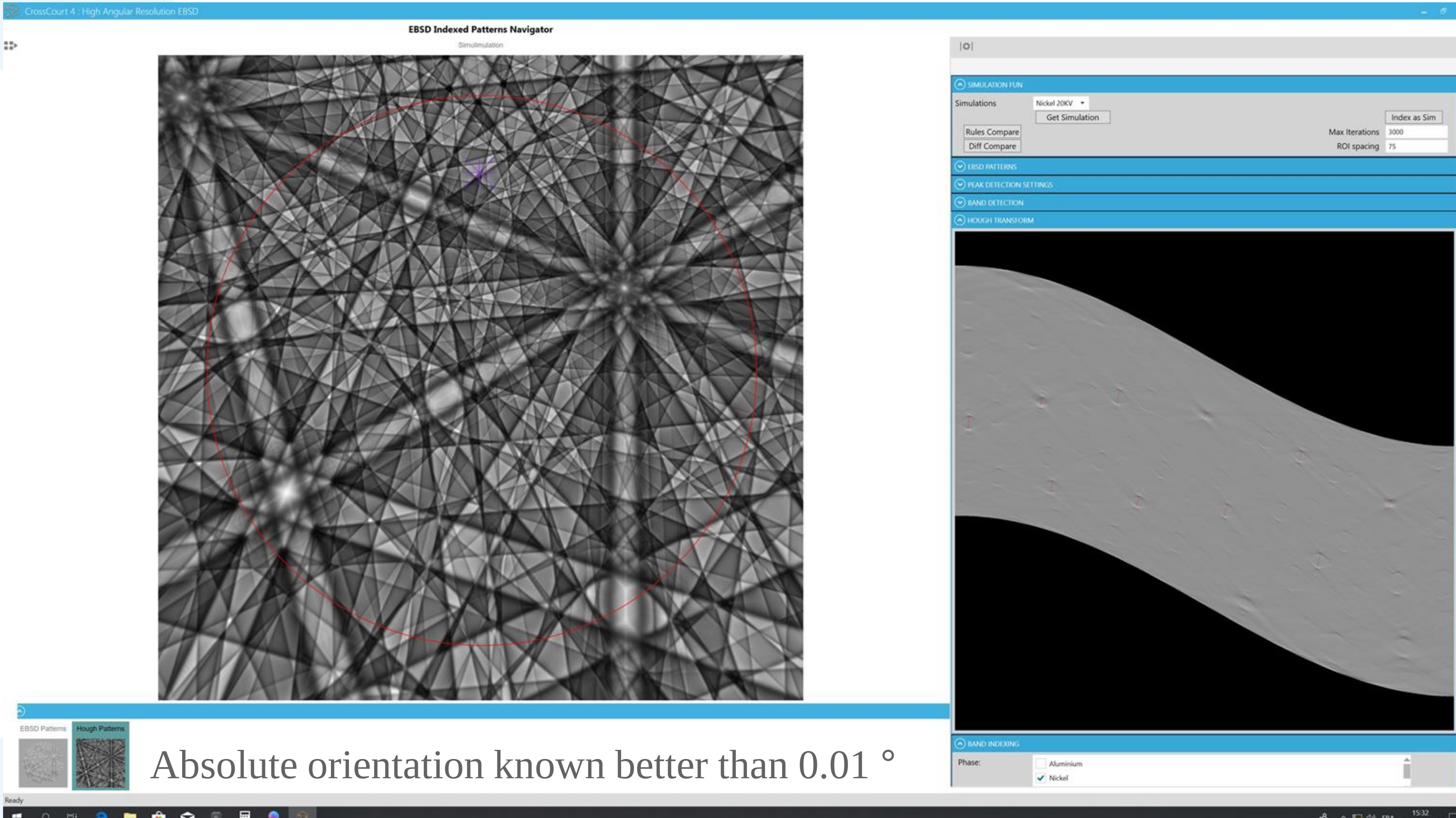


Fig. 8. The correlated misorientation distribution (Channel5, Oxford Instr.) in indicates a twice higher precision for the pattern matching – X-corr, Fig. 7(b) – in comparison to the Hough-related standard orientation determination (a). The class width applied was 0.05°. (a) Misorientation distribution.



Refine absolute orientation using a cross-correlation between real (4k pattern) and synthetic patterns.



Absolute orientation known better than 0.01 °