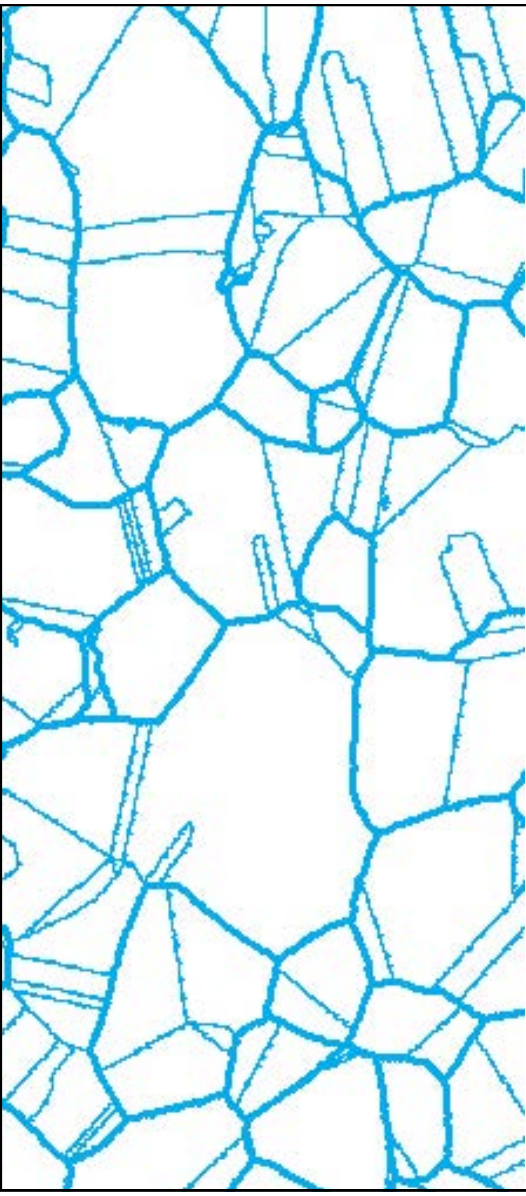


Spherical Indexing of EBSD Patterns

Stuart Wright, René de Kloe

GATAN + EDAX

Outline

- Hough Based Indexing
- Forward Model Based Indexing
  - Dictionary indexing
  - **Spherical indexing**
    - application results
  - Refinement
    - improved angular resolution
    - resolving pseudo-symmetry
  - Extension of Spherical Indexing

Will Lenthe

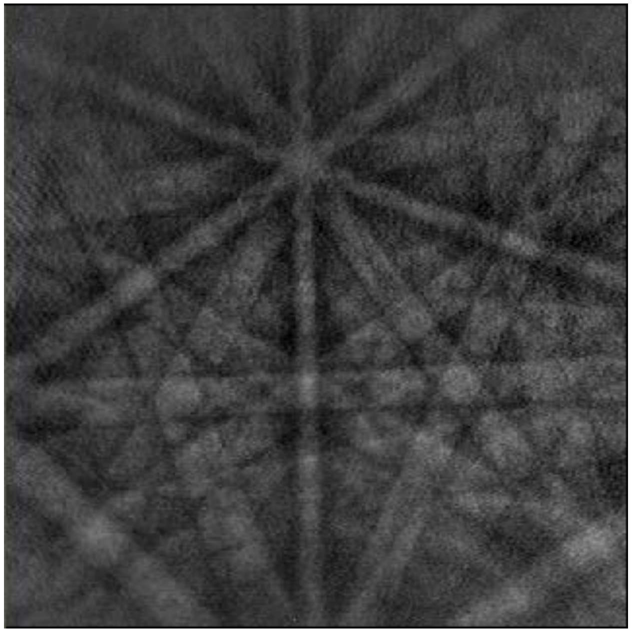
Stuart Wright

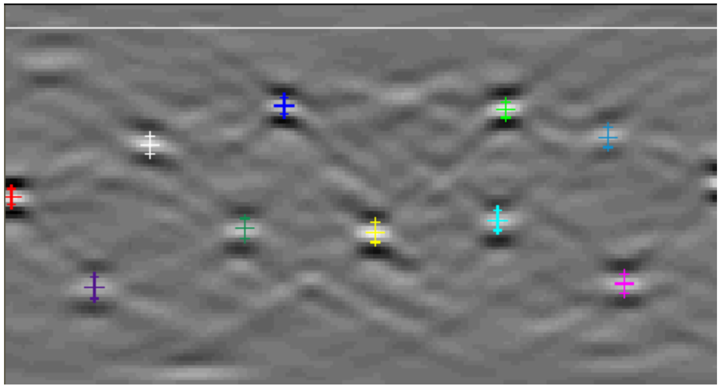
Matt Nowell

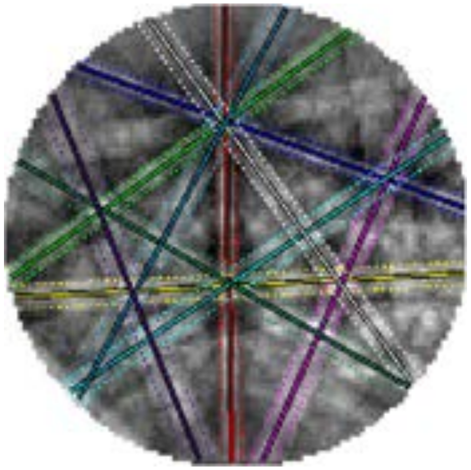
René de Kloe

Hough transform

N. C. Krieger-Lassen, K. Conradsen, and D. Juul-Jensen (1992) "Image Processing Procedures for Analysis of Electron Back Scattering Patterns", *Scanning Microscopy,* **6,** 115-121.







"Advances in Automatic EBSP Single Orientation Measurements" K. Kunze, S.I. Wright, B.L. Adams and D.J. Dingley. *Textures and Microstructures* **20**, 41-54 (1993).

Presented at *Microscale Texture of Materials Symposium, ASM/TMS Meeting,* Cincinnati, Ohio, October 1991.

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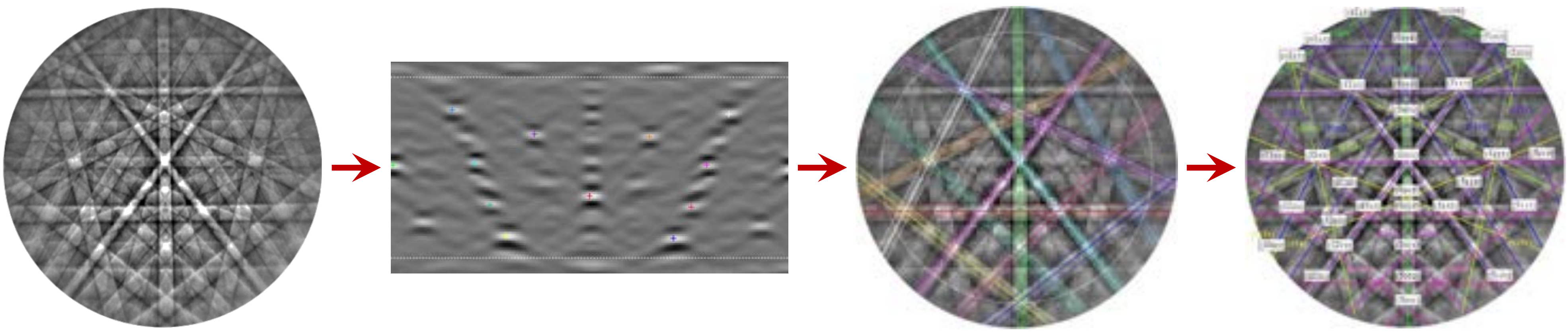
Hough transform and triplet indexing

- Hough-transform based indexing method
- Collect a pattern → detect the bands to "reconstruct" the pattern → match the interplanar angles against an (ideal) reference model → find a solution



- Works very well but requires:
  - Consistent band detection
  - Detecting enough bands
  - Accurate band direction determination
  - Minimal pattern shadowing

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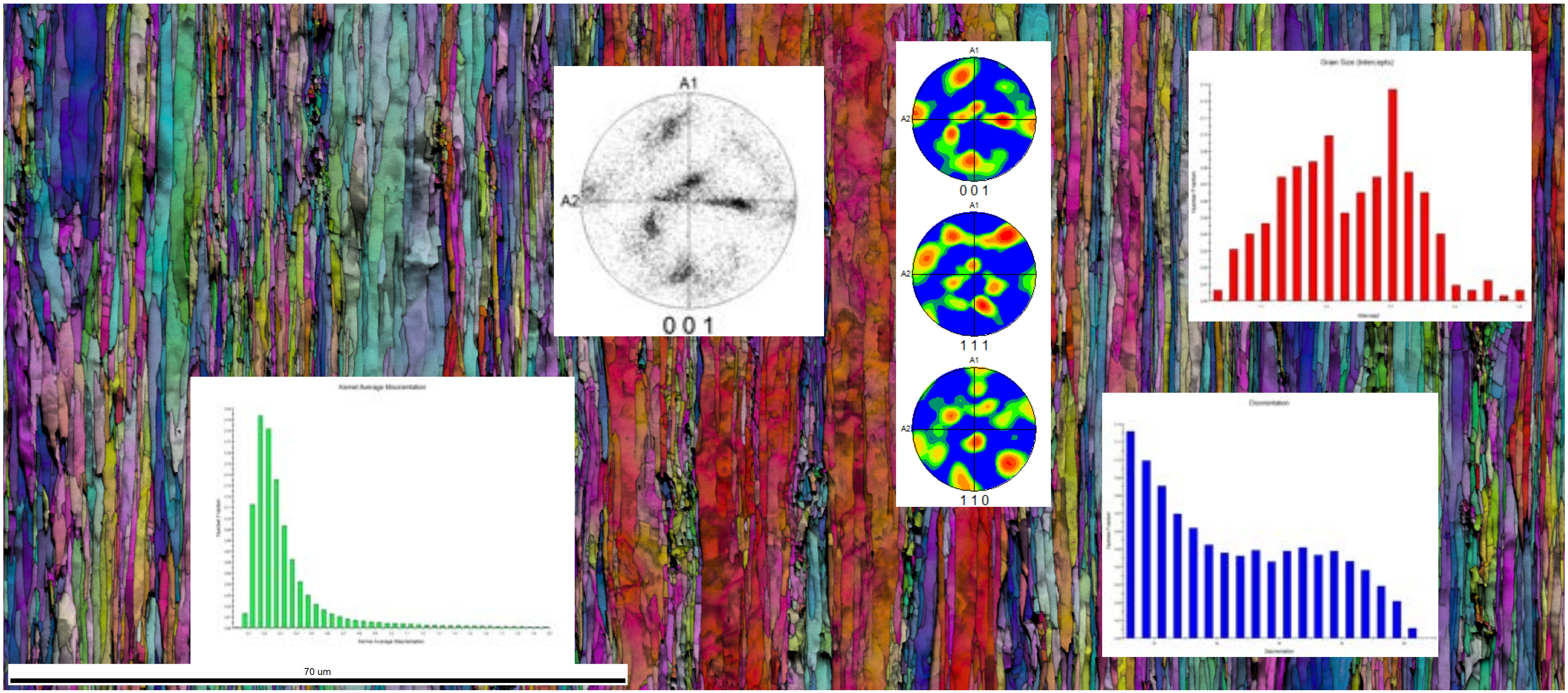
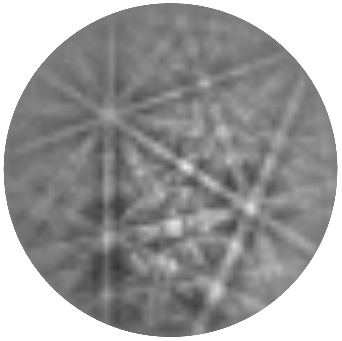
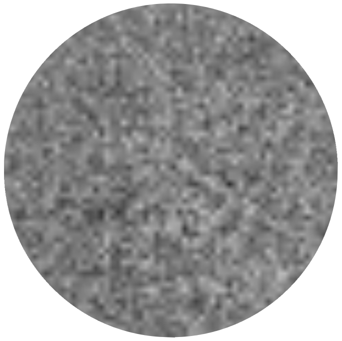
Example → heavily deformed Mo sample with triplet indexing



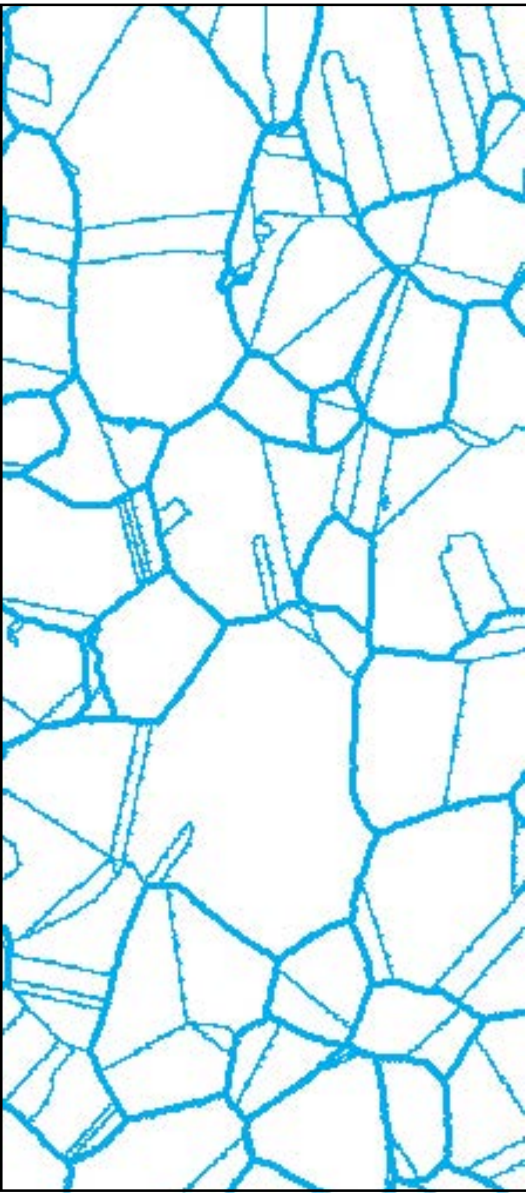
Hough transform and triplet indexing





- Pros:
  - Fast
  - Robust in many situations
    - Particularly with N(L)PAR

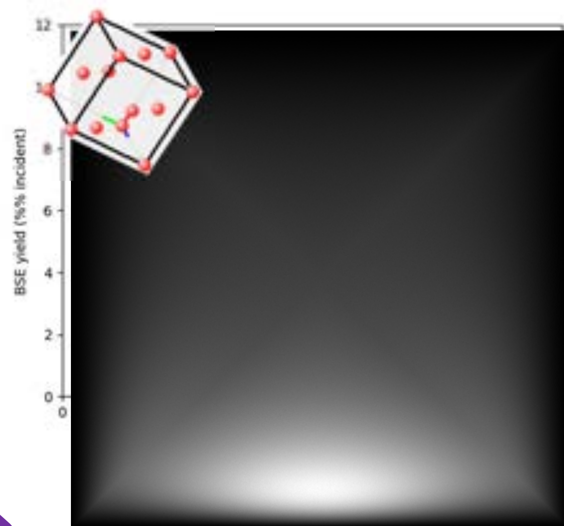
- Cons:
  - Struggles with very poor patterns
  - Determining the best reflectors can be a challenge for some crystals
  - Angular resolution is ~0.1° – 0.5° primarily dependent on pattern quality
  - Difficulty resolving pseudosymmetry
  - Unable to index non-centrosymmetric crystals (band intensity profiles vs band locations)

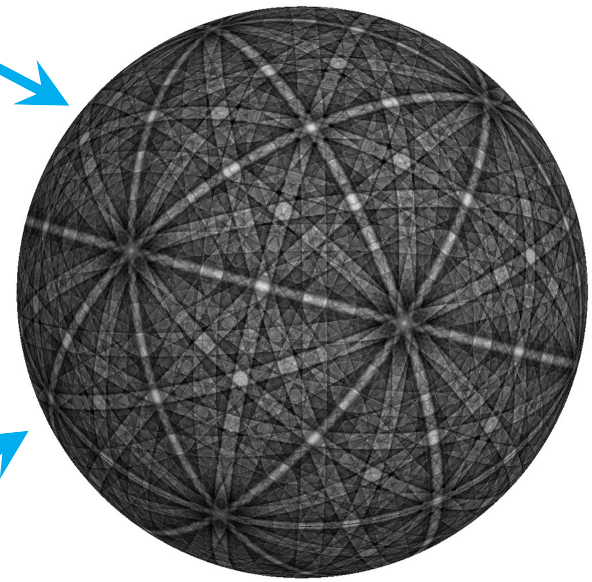


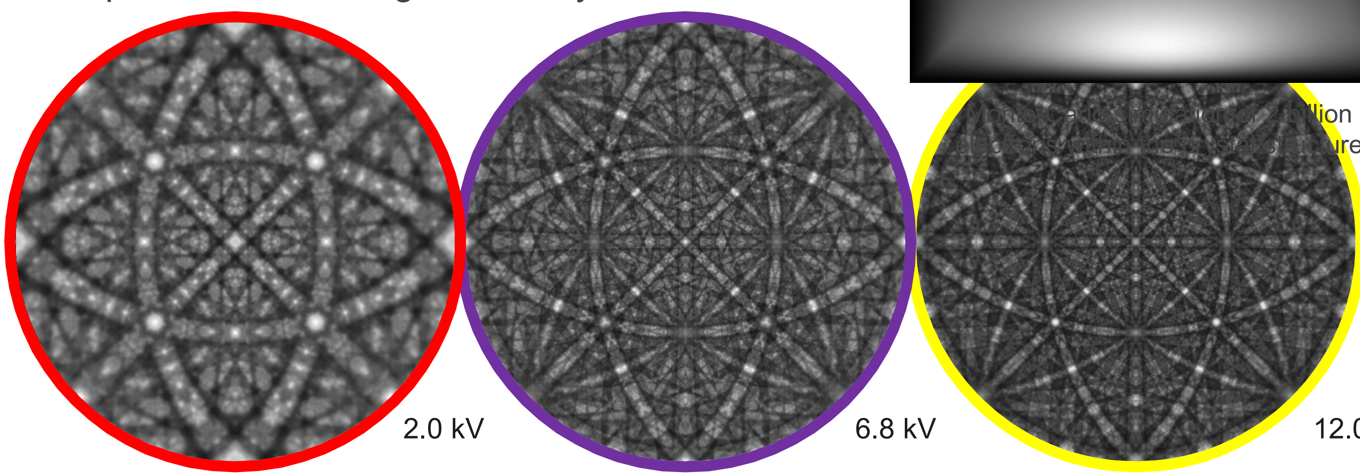
Can we improve on Hough transform-based indexing?

Alternative indexing method: using dynamic pattern simulations

- Model the diffraction path of 2 billion electrons in a crystal using a Monte Carlo simulation for BSE yield
- Dynamical diffraction simulation is performed for each energy
- BSE weighted sum from all energies is combined into a single Kikuchi sphere
- Requires full knowledge of the crystal structure





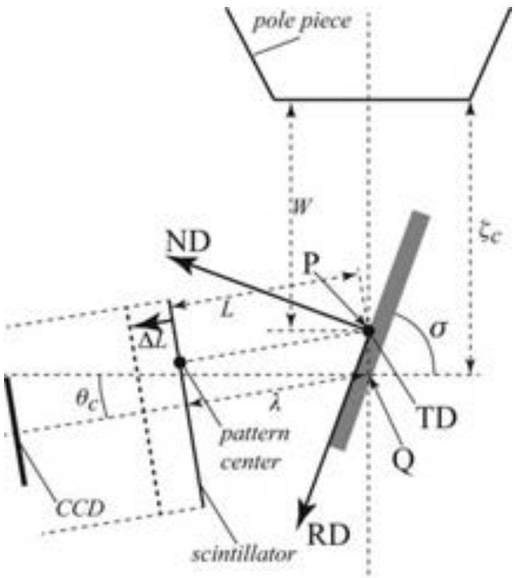


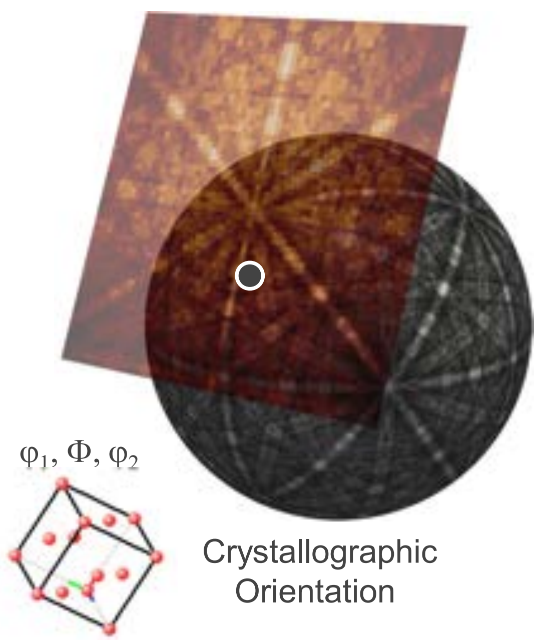
Callahan, P. G., & De Graef, M. (2013). Dynamical electron backscatter diffraction patterns. Part I: Pattern simulations. *Microscopy and Microanalysis*, *19*(5), 1255-1265.

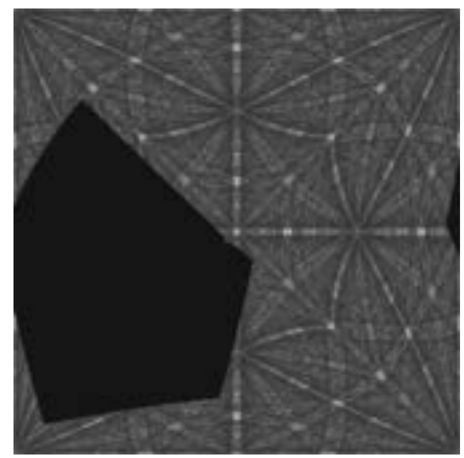
Dynamic pattern simulation

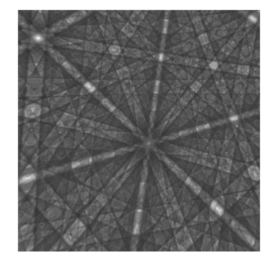
EBSD Pattern simulation

- Rotate Kikuchi sphere by orientation
- Project using the current calibration + crop to match the EBSD detector projection geometry





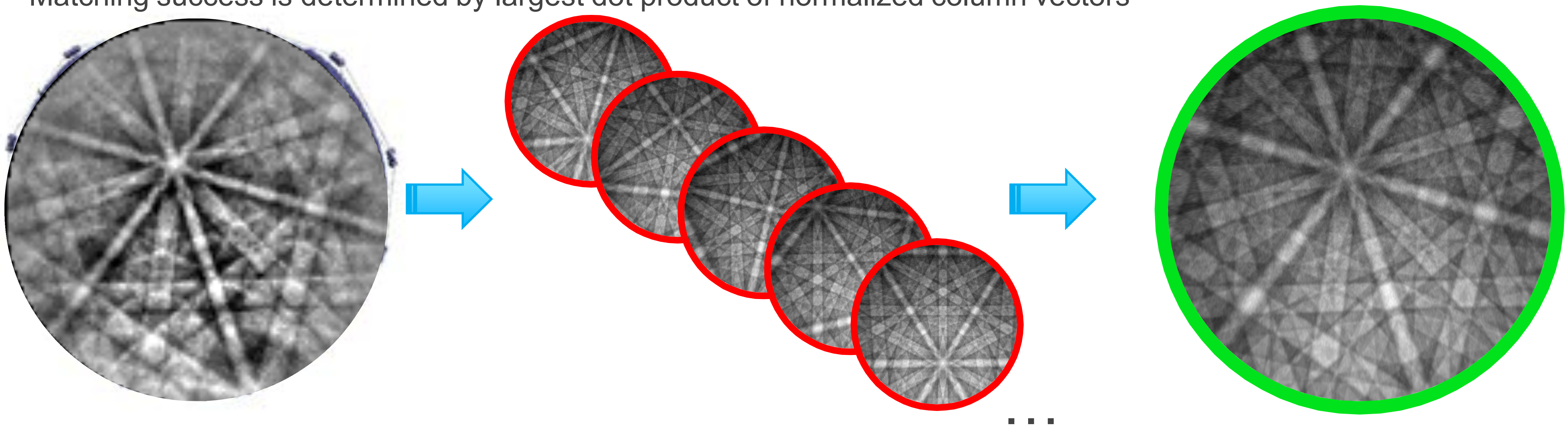




Callahan, P. G., & De Graef, M. (2013). Dynamical electron backscatter diffraction patterns. Part I: Pattern simulations. *Microscopy and Microanalysis*, *19*(5), 1255-1265.

EBSD mapping using Dictionary Indexing – procedure

- Create a library (dictionary) of all patterns of all possible orientations on a predefined grid (e.g. every 2º)
- Compare each pattern in a scan against all patterns in the dictionary and find the best match
- Patterns in dictionary are created to match the actual pattern projection geometry in the SEM
  - Need to create a new library for each map/WD
- Matching success is determined by largest dot product of normalized column vectors



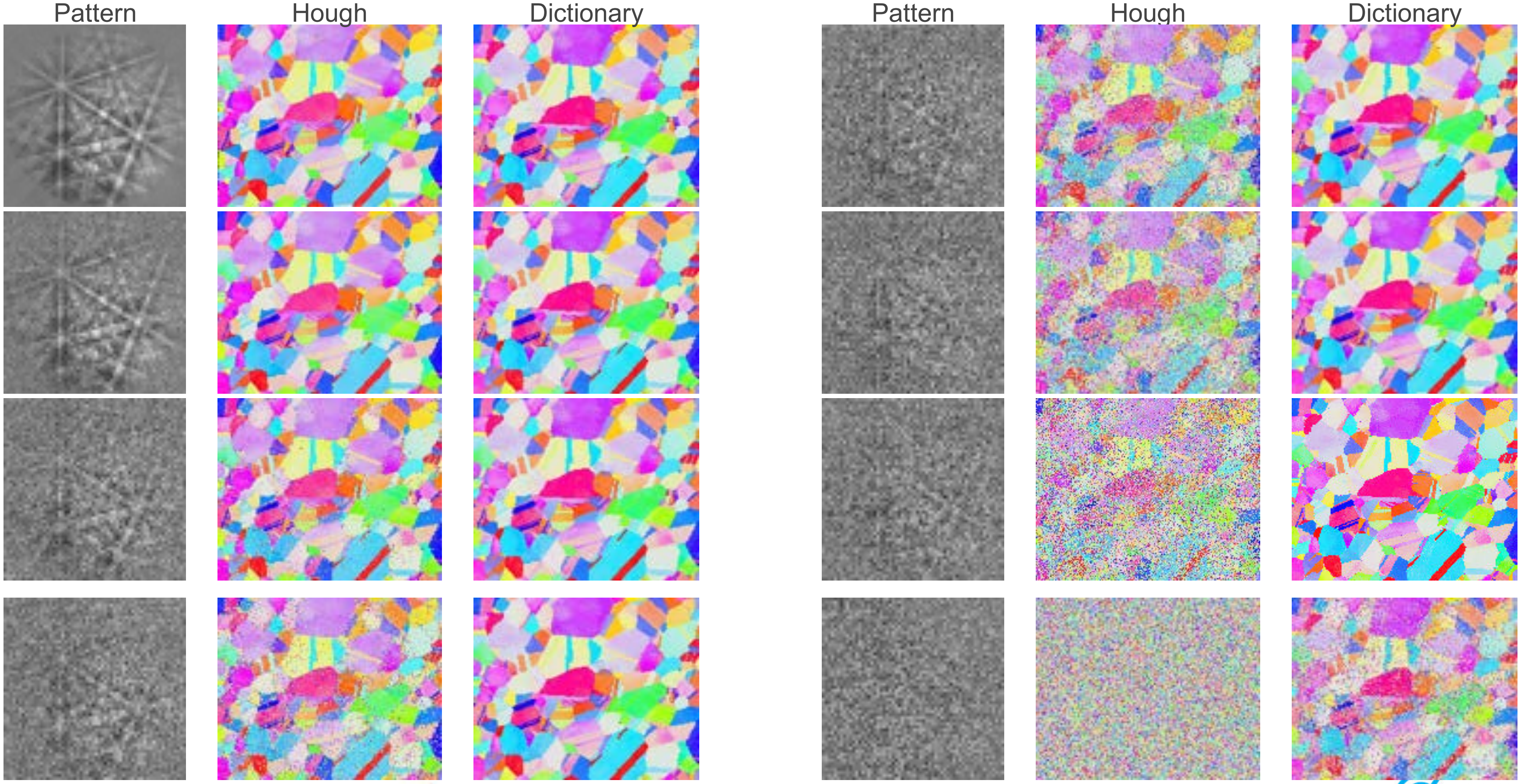
Experimental pattern

Comparison against all simulated patterns in dictionary

Best match

S. Singh and M. De Graef (2016). Orientation sampling for dictionary-based diffraction pattern indexing methods. *Modelling and Simulation in Materials Science and Engineering*, **24**: 085013.

Dictionary indexing robustness - Nickel with varying noise



Full pattern (dictionary) matching is extremely sensitive with noisy patterns

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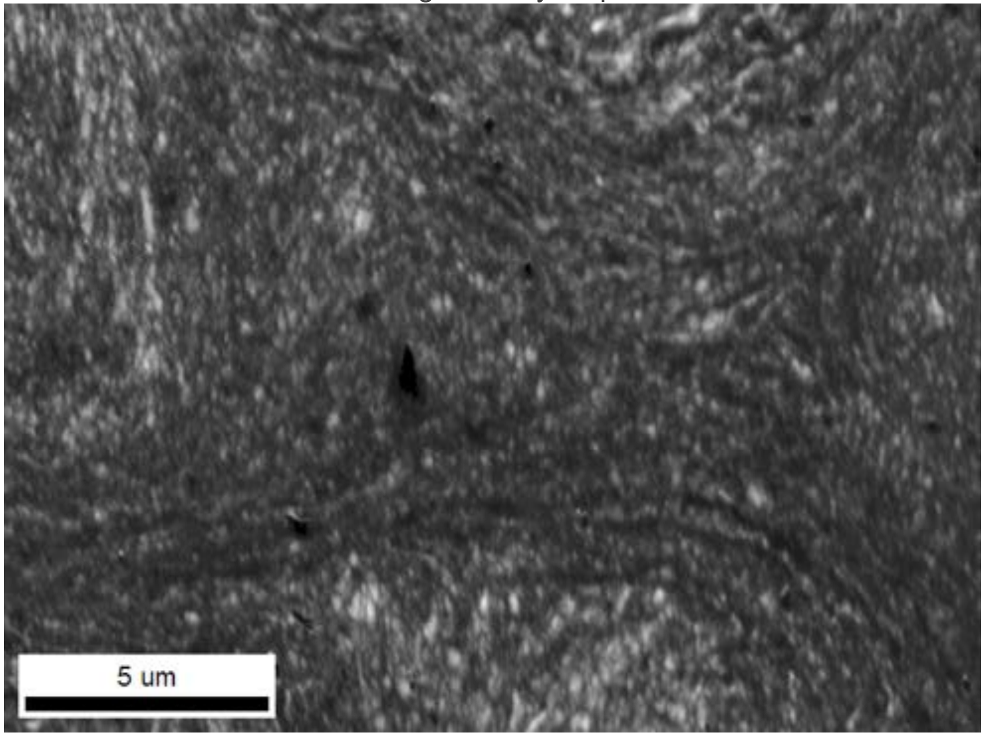
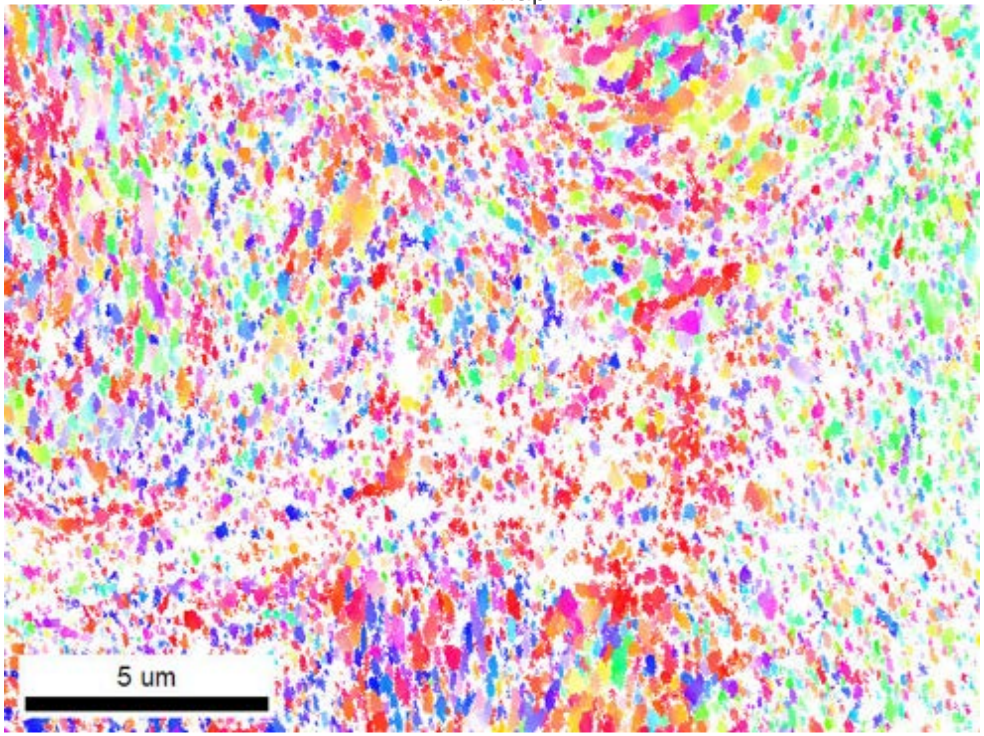
EBSD mapping using Dictionary Indexing – example

EBSD indexing improvements on highly deformed compressed ferrite powder sample

Image Quality map



IPF map



Hough indexing, ISR: 49.8%

Sample courtesy of Carsten Bonnekoh, KIT, Germany

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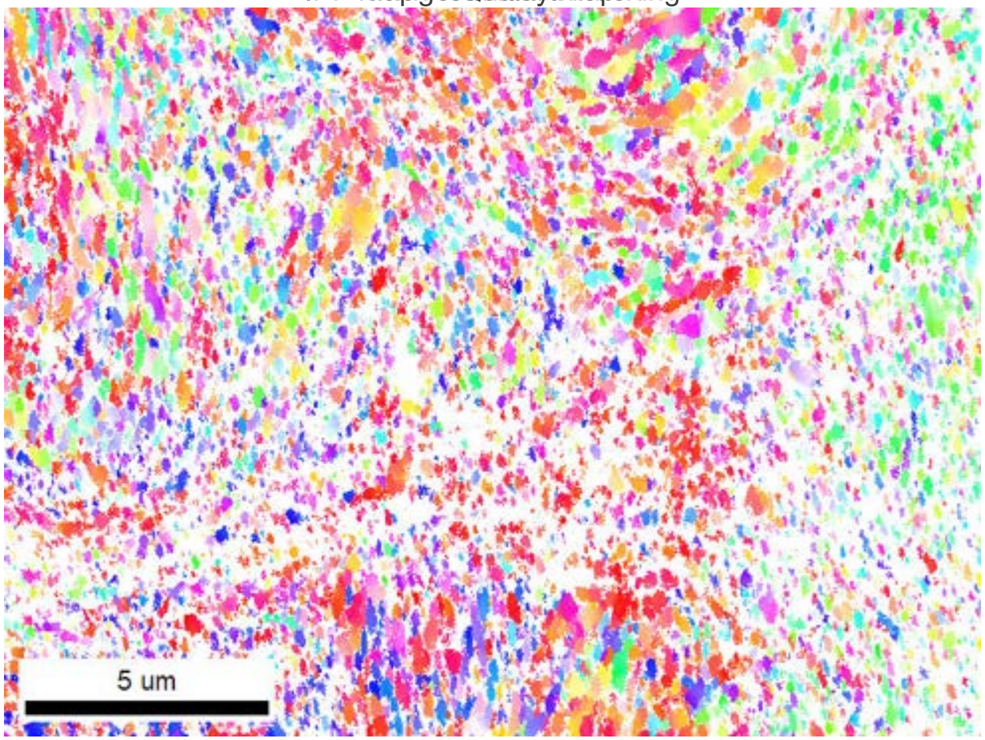
EBSD mapping using Dictionary Indexing – example

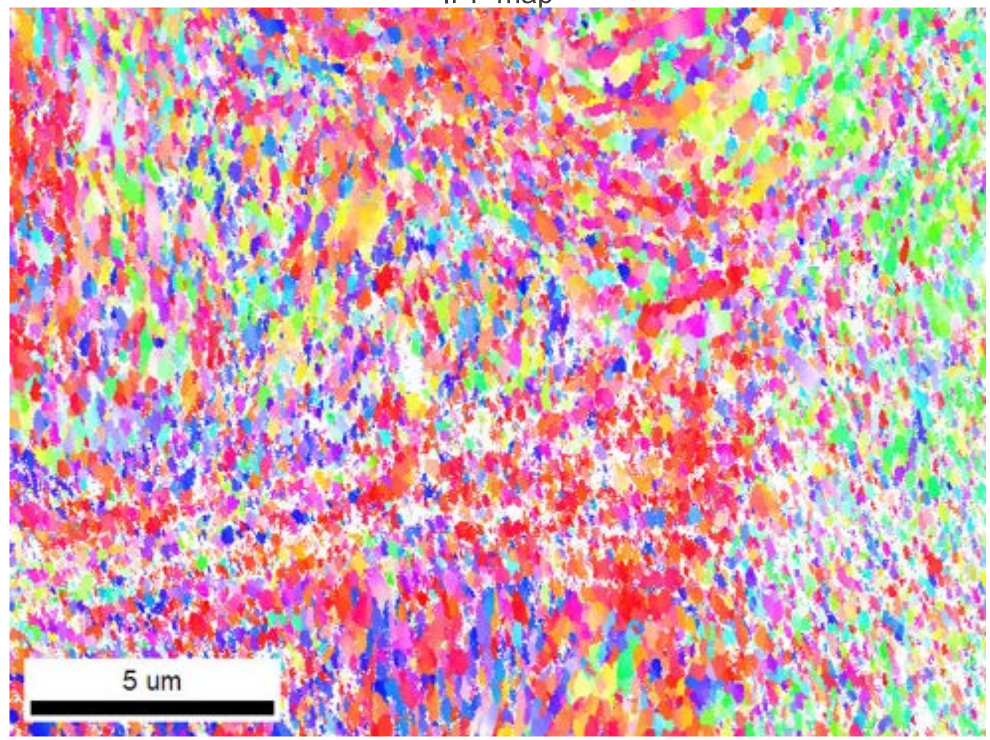
EBSD indexing improvements on highly deformed compressed ferrite powder sample

IPF map – standard indexing

Image Quality map



IPF map



Dictionary indexing, ISR: 82.3%

Sample courtesy of Carsten Bonnekoh, KIT, Germany

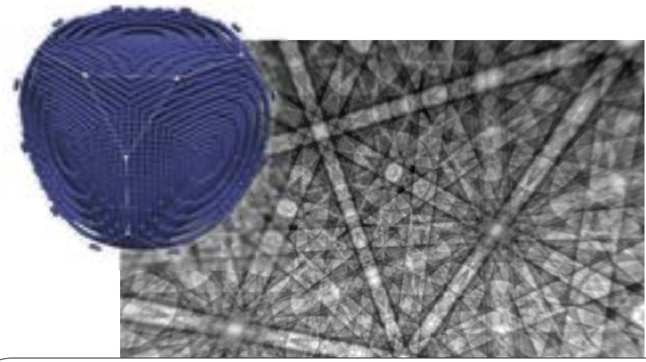
Dictionary indexing – Pros & cons

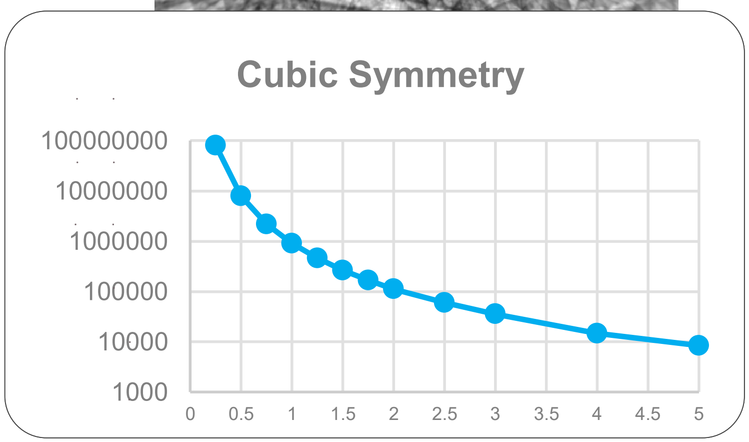
- More robust than Hough indexing
- Significantly slower than Hough indexing
  - Large dictionaries (particularly for low symmetries)
  - Angular resolution
- Steep learning curve
  - Master pattern calculation
  - Dictionary spacing
  - Refinement parameters

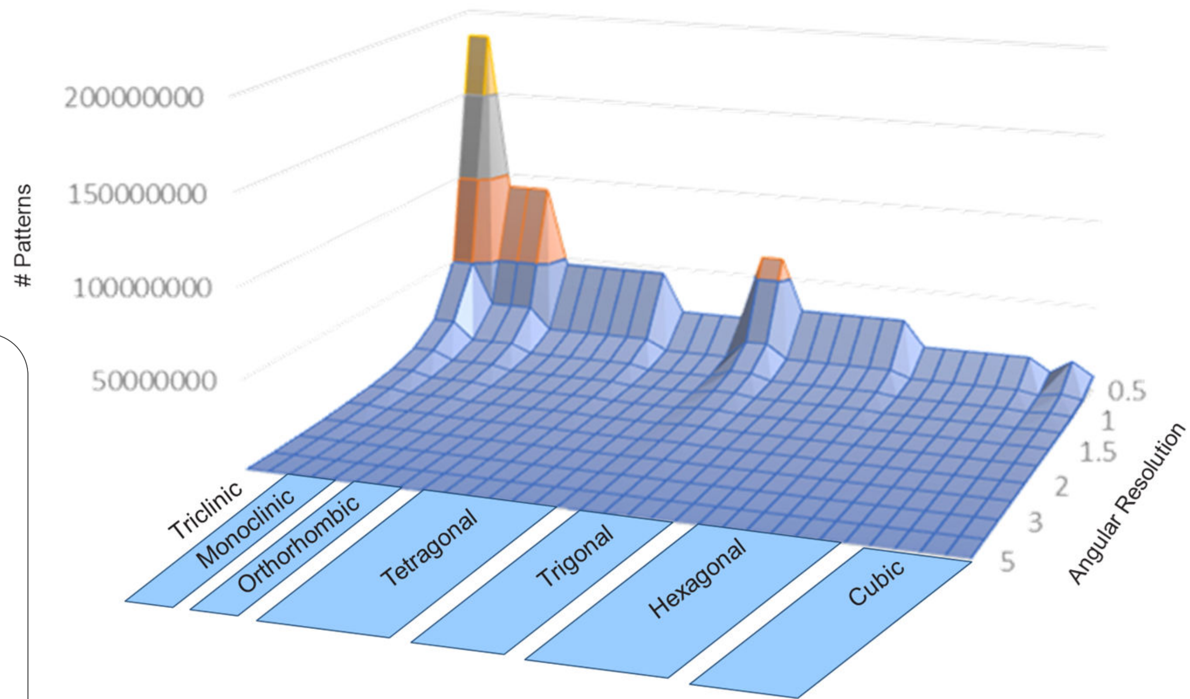
Singh, S., Ram, F., & De Graef, M. (2017). EMsoft: open-source software for electron diffraction/image simulations. *Microscopy and Microanalysis*, *23*(S1), 212-213.

Dictionary indexing limitation – dictionary size

- Dictionary size is related to crystal symmetry and initial grid resolution

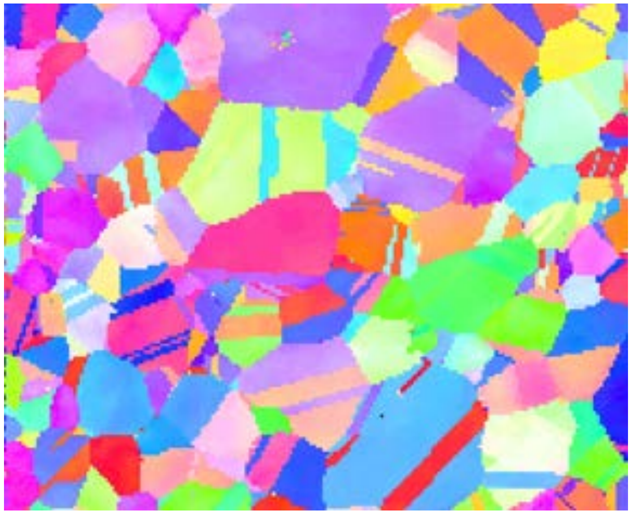




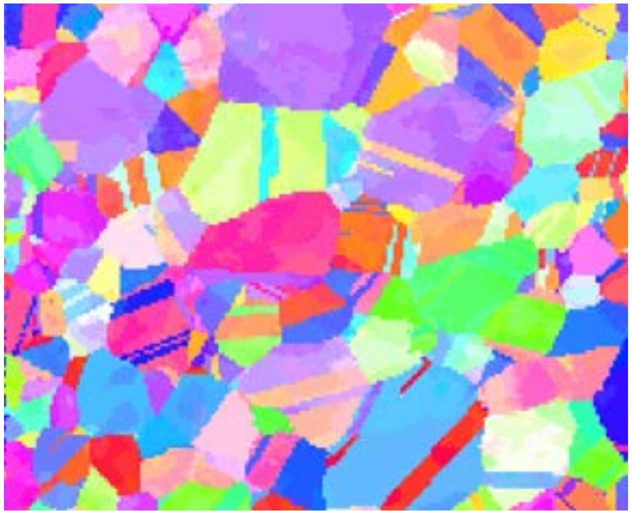


Minimum dictionary spacing

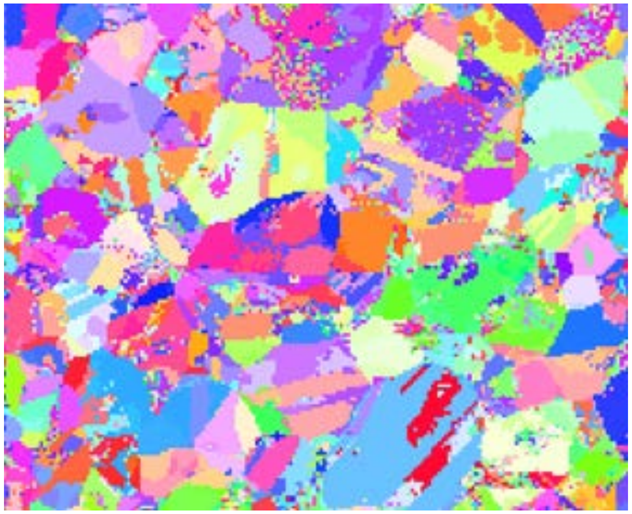
Hough-based



2.5° Dictionary



5° Dictionary



Minimum spacing is not just about angular precision but also indexing reliability

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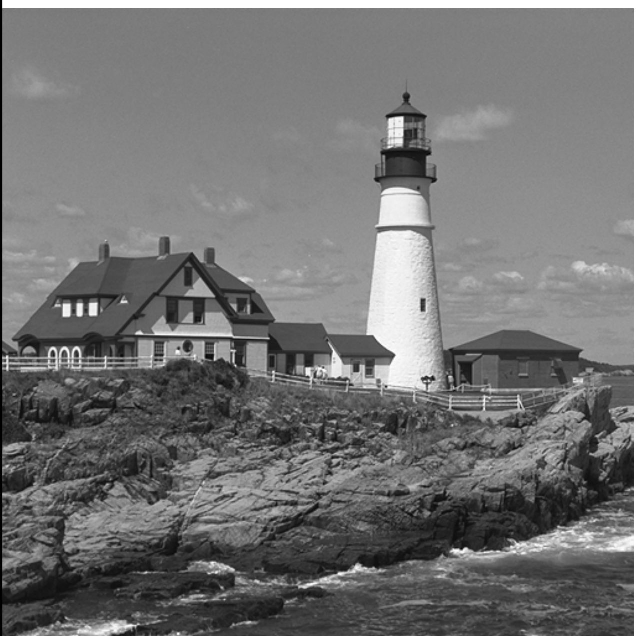
2D dictionary indexing

Known ‘full’ scene



Create a dictionary of patterns to match the detector size

Detected image (includes noise)

Find the best match against the dictionary to locate where the detected image falls within the full scene

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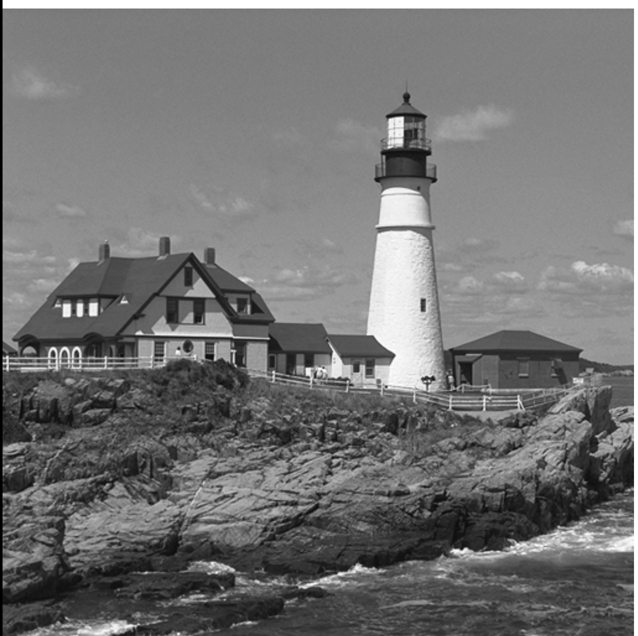
Alan Fink “Portland Head Light,” Kodak Image 21 (SP91052)

Alternative: 2D cross-correlation – Fourier transform

Known 'full' scene

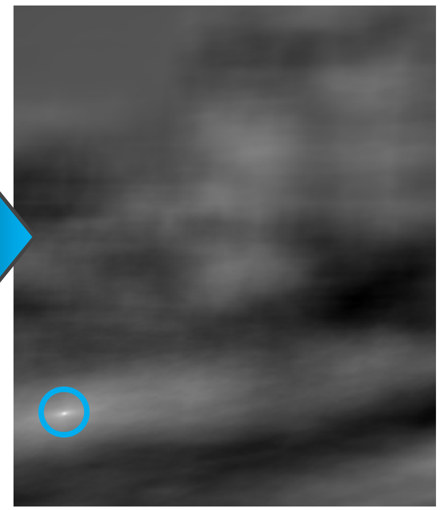


$f(x)$

Detected image (includes noise)

$g(x)$

Calculate cross-correlation



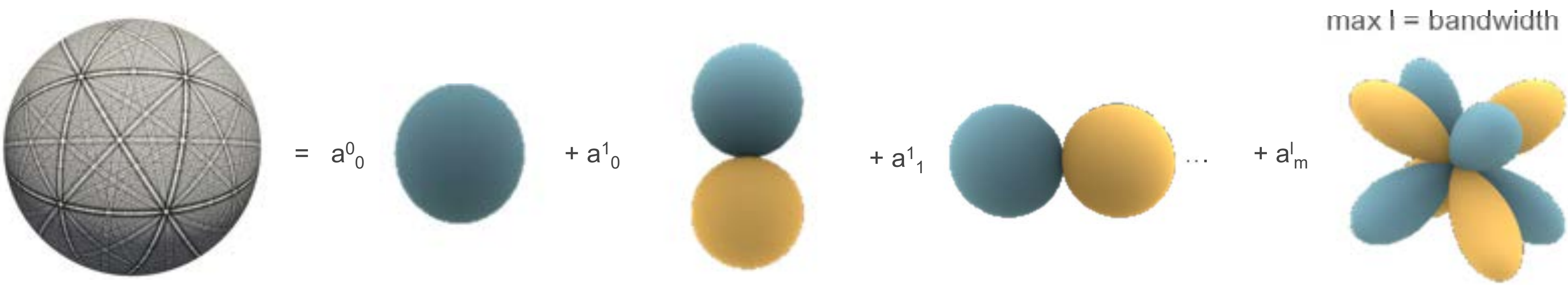
$$\mathscr{F}^{-1}\left\{\overline{\mathscr{F}\{f\}}\mathscr{F}\{g\}\right\}$$

Use Cross-Correlation peak to locate detected image within scene

$\mathscr{F}$ Fourier transform

Alan Fink "Portland Head Light," Kodak Image 21 (SP91052)

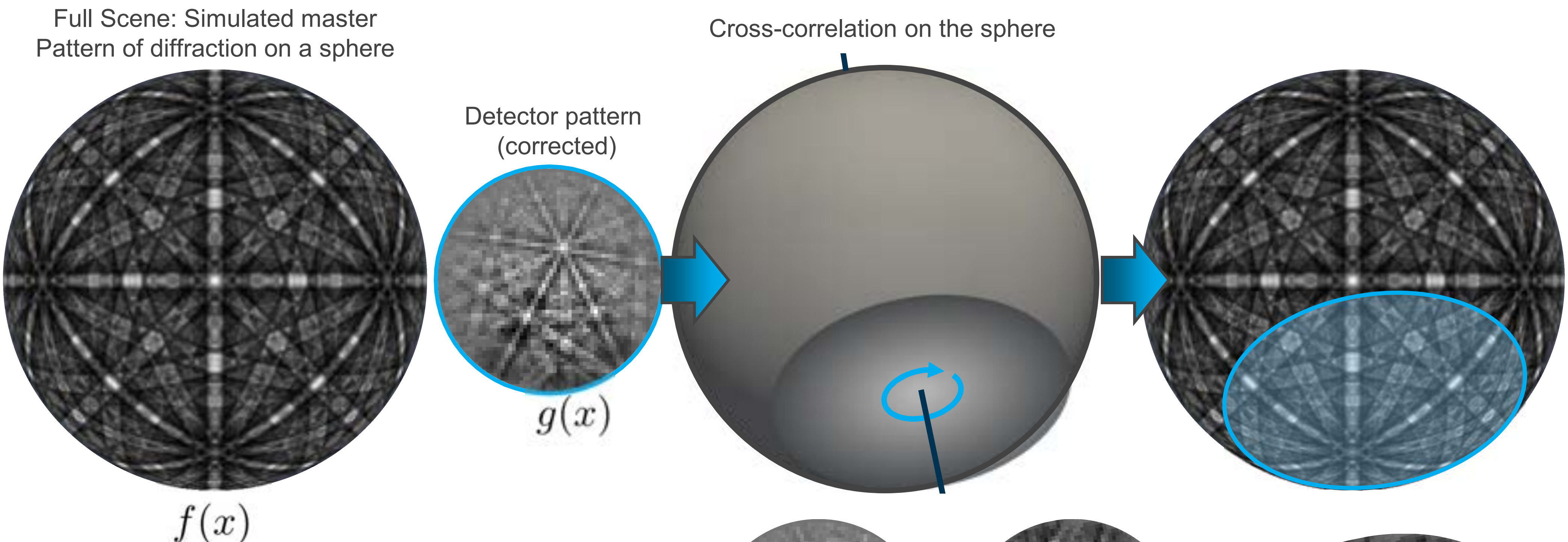
Spherical harmonic transform



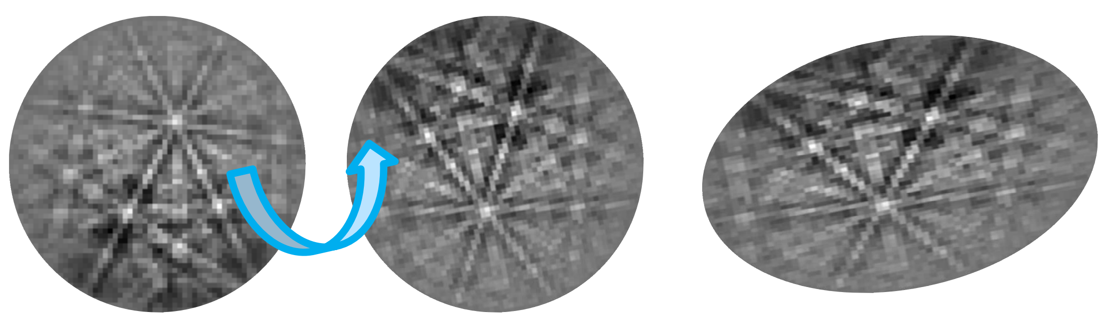
	Fourier transform	Spherical harmonic transform
Forward	$\mathscr{F}$	$\hat{f}^l_m \triangleq \mathscr{S}\{f(\theta,\phi)\} = \int_{\theta=0}^{\pi}\int_{\phi=0}^{2\pi} f(\theta,\phi)\overline{Y^l_m(\theta,\phi)}\sin\theta\, d\phi\, d\theta$
Inverse	$\mathscr{F}^{-1}$	$f(\theta,\phi) = \sum_{m=-l_{max}}^{l_{max}} \sum_{l=\lvert m\rvert}^{l_{max}} \hat{f}^l_m Y^l_m(\theta,\phi)$

Wikimedia commons

Can we do cross-correlation on a sphere?



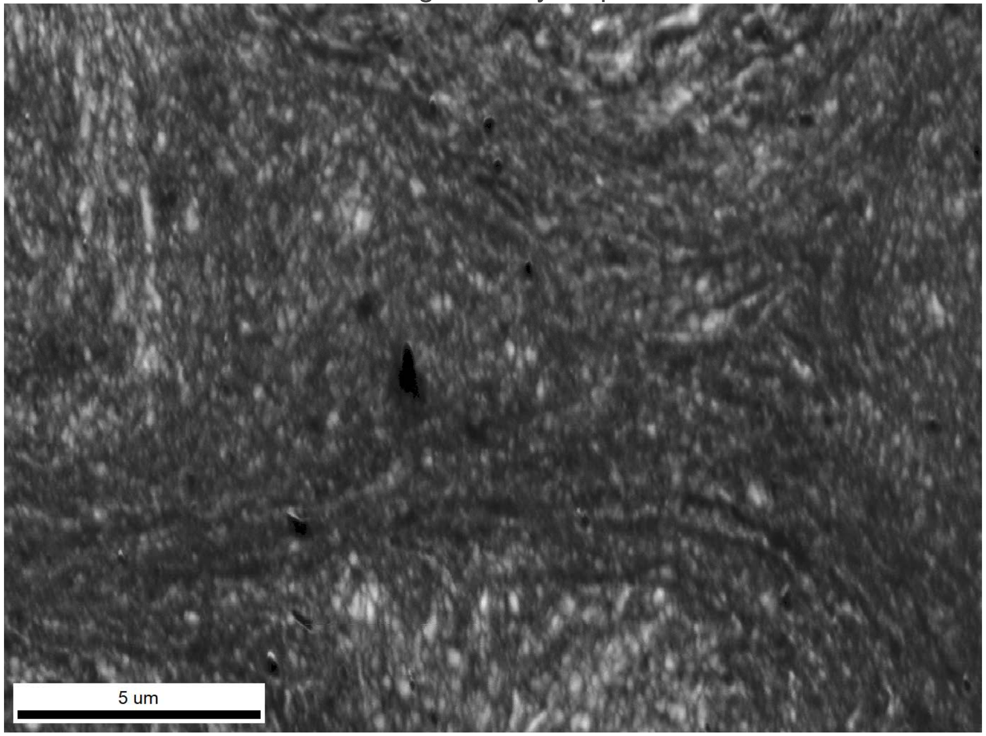
Note: this analog to the 2D lighthouse example is oversimplified. One difference of 3D spherical problem relative to the 2D planar example is the rotational component.



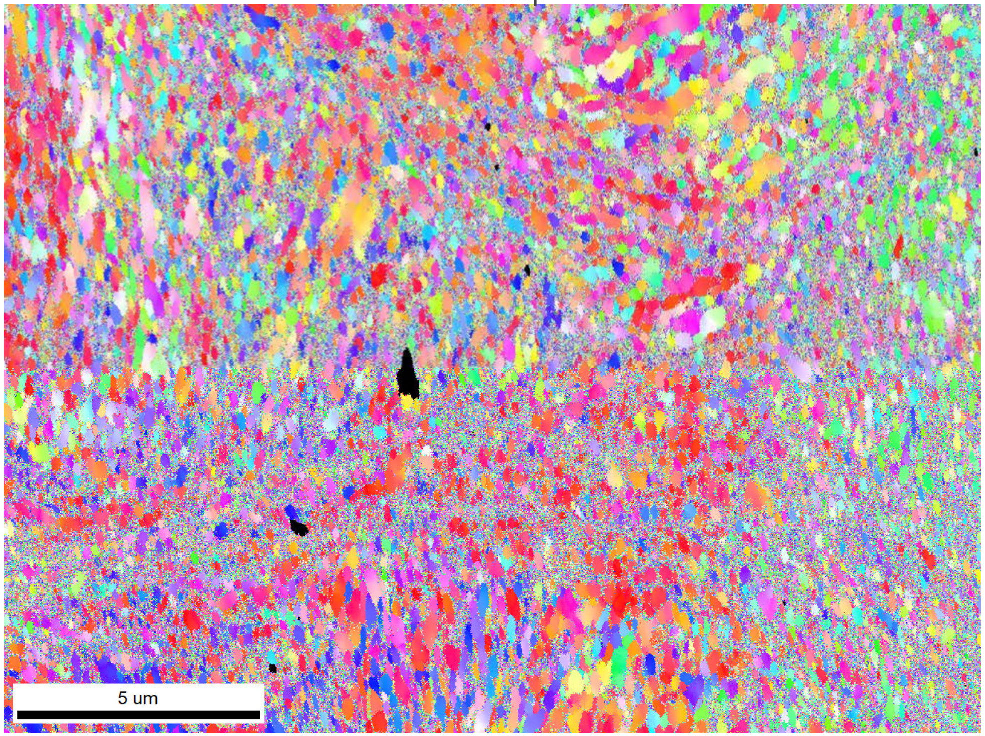
EBSD mapping using Hough-based indexing

EBSD indexing improvements on highly deformed compressed ferrite powder sample

Image Quality map



IPF map

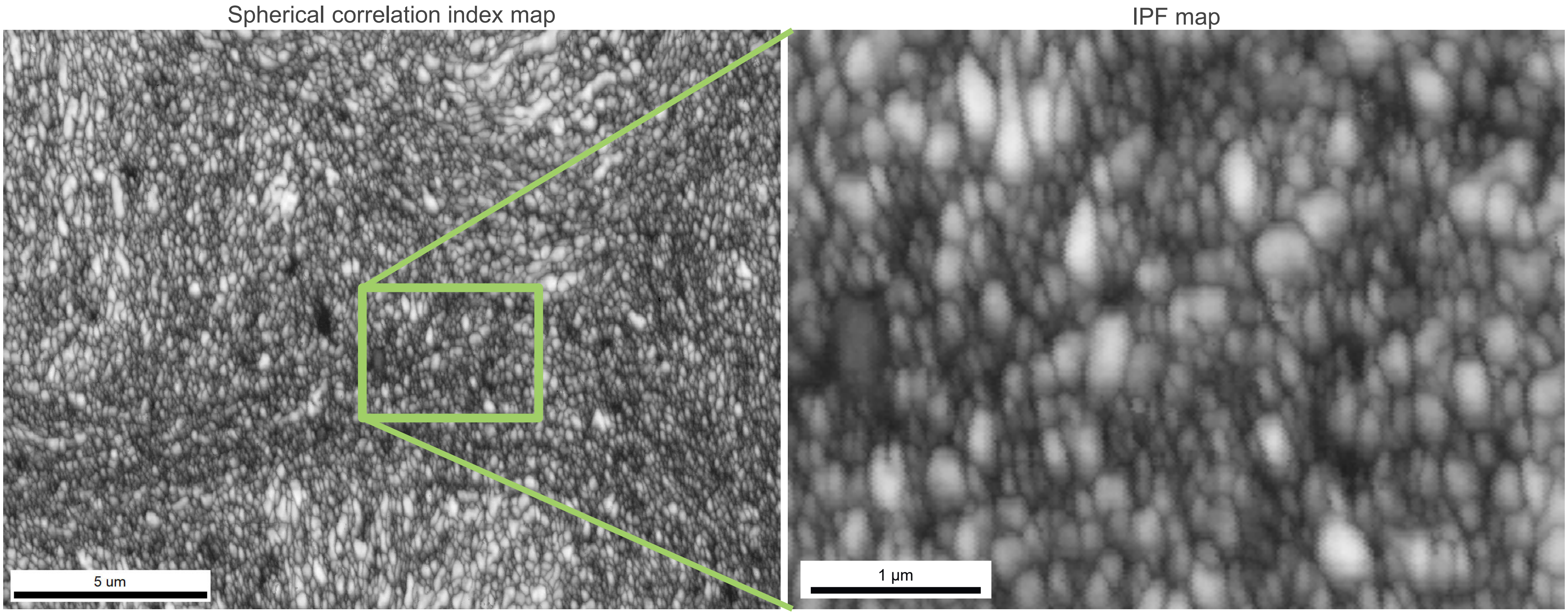


Hough indexing

Sample courtesy of Carsten Bonnekoh, KIT, Germany

EBSD mapping using Spherical indexing with NPAR

EBSD indexing improvements on highly deformed compressed ferrite powder sample

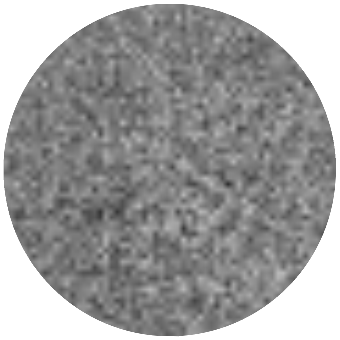


Hough indexing, ISR: 98.1%

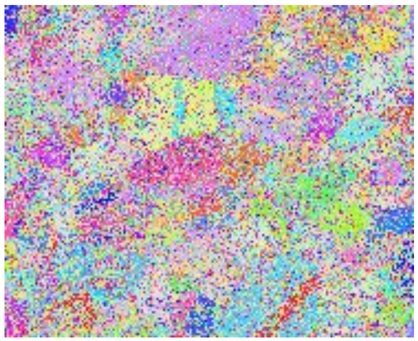
Sample courtesy of Carsten Bonnekoh, KIT, Germany

Spherical EBSD indexing robustness

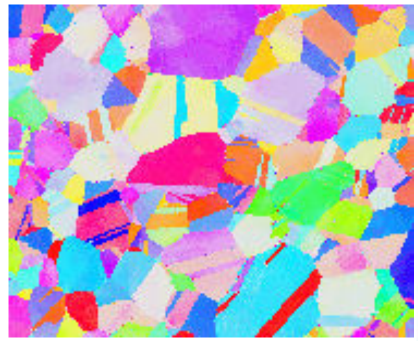
First Pattern

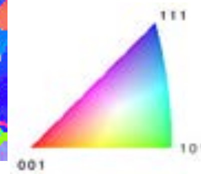


Hough Indexing (NPAR)

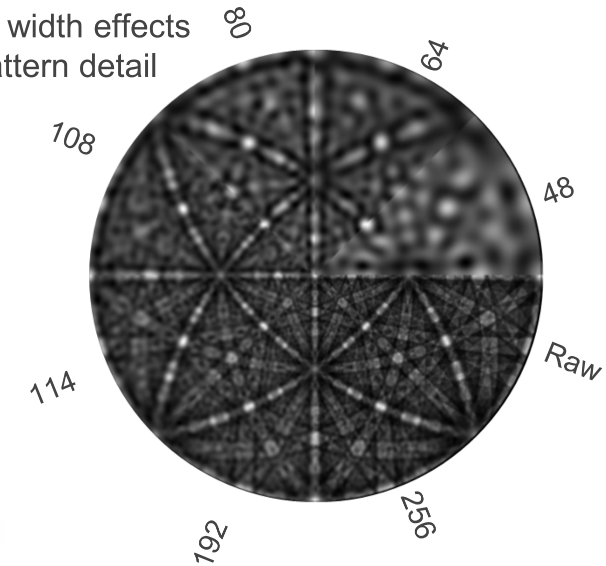


Dictionary Indexing (2.5°)



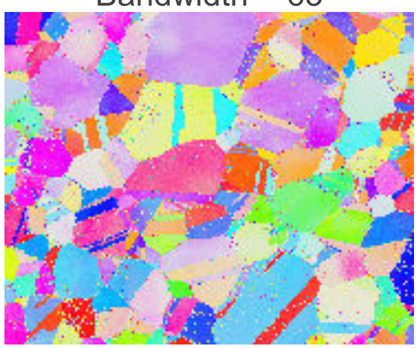


Band width effects on pattern detail



Bandwidth = 53

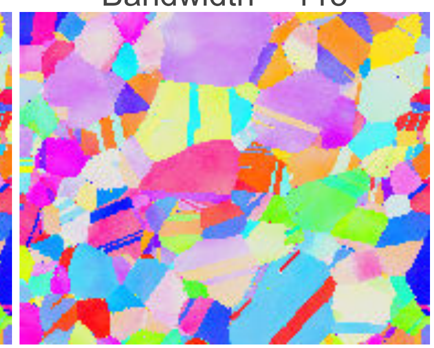
Bandwidth = 63



Bandwidth = 74

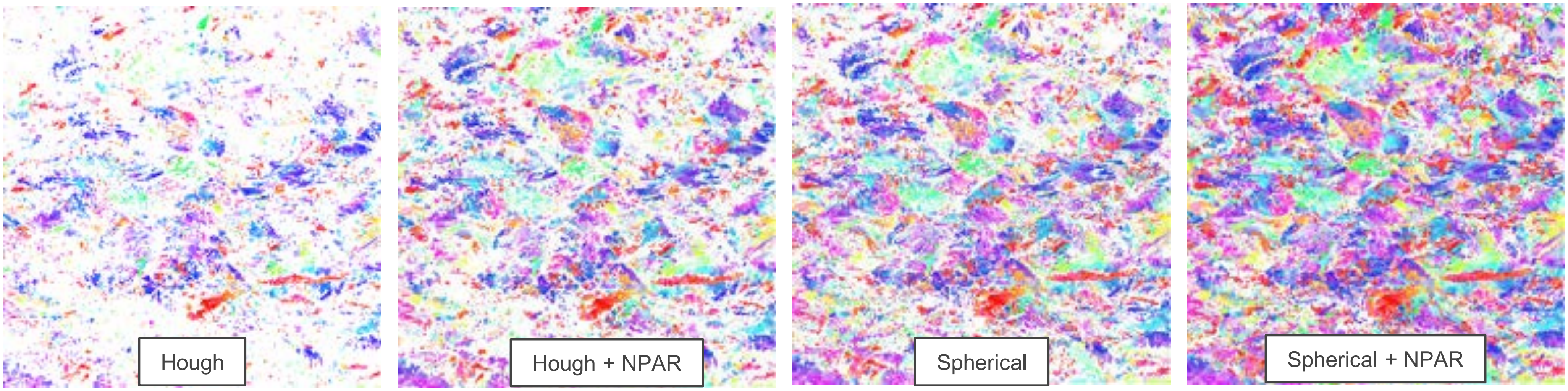
Bandwidth = 88

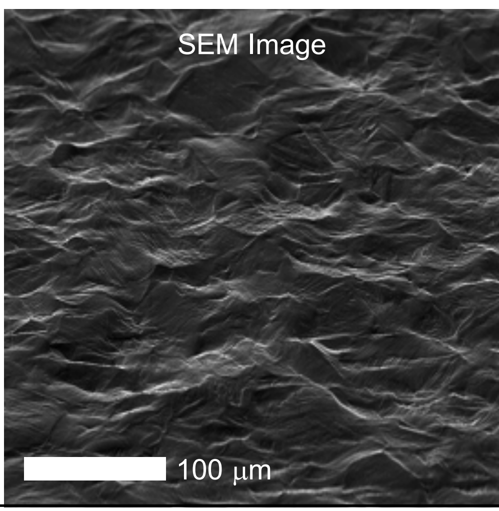
Bandwidth = 113

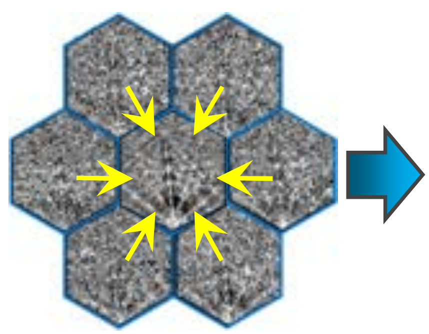


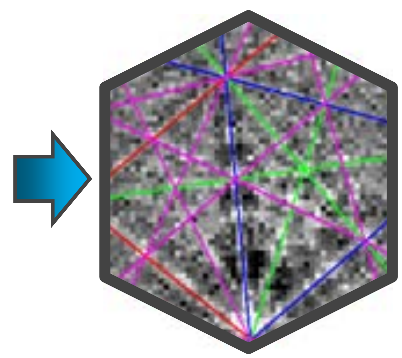
Lenthe, W. C., Singh, S., & De Graef, M. (2019). A spherical harmonic transform approach to the indexing of electron back-scattered diffraction patterns. *Ultramicroscopy*, *207*, 112841.

Tensile sample – rough surface







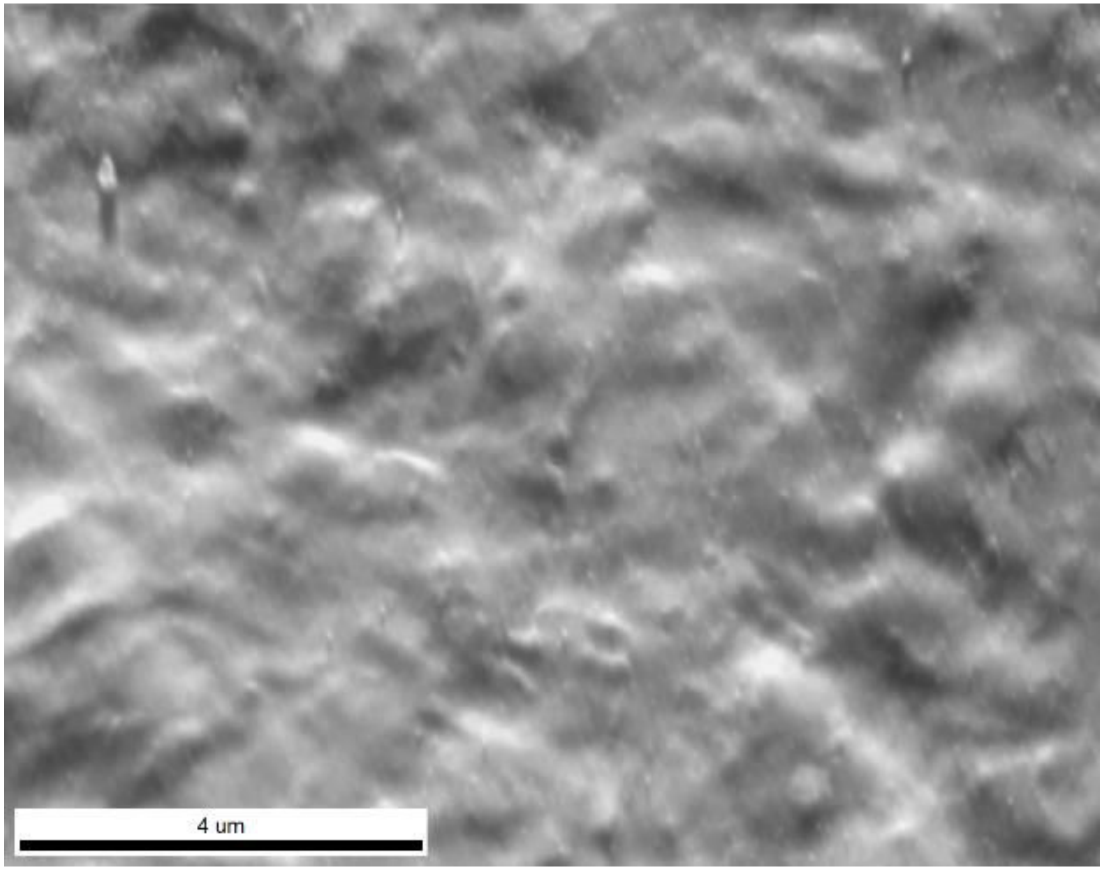


CI > 0.1
after CIS

Brewick, P.T., Wright, S.I. & Rowenhorst D.J. (2019). NLPAR: Non-local smoothing for enhanced EBSD pattern indexing. *Ultramicroscopy*, 200, 50-61.

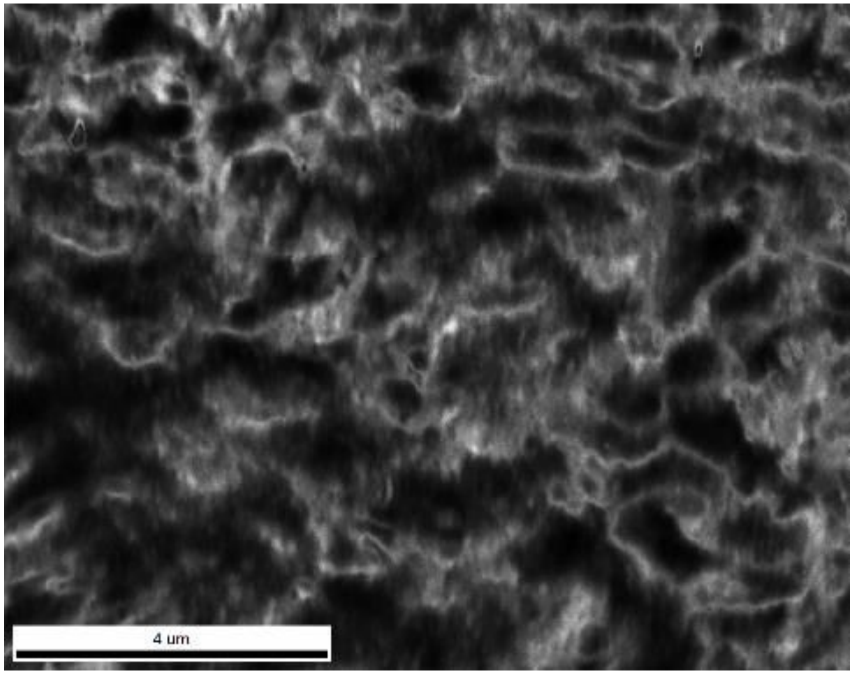
GATAN AMETEK EDAX

Titanium nitride coating with sub-micron grain size



SEM image of analysis area

- TiN coating on coarse grained polycrystalline substrate – rough surface
- No additional surface preparation for EBSD



IQ map as measured

480,217 points, 10.89 x 8,57 µm, 15 nm steps

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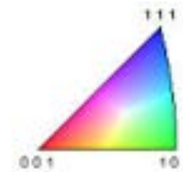
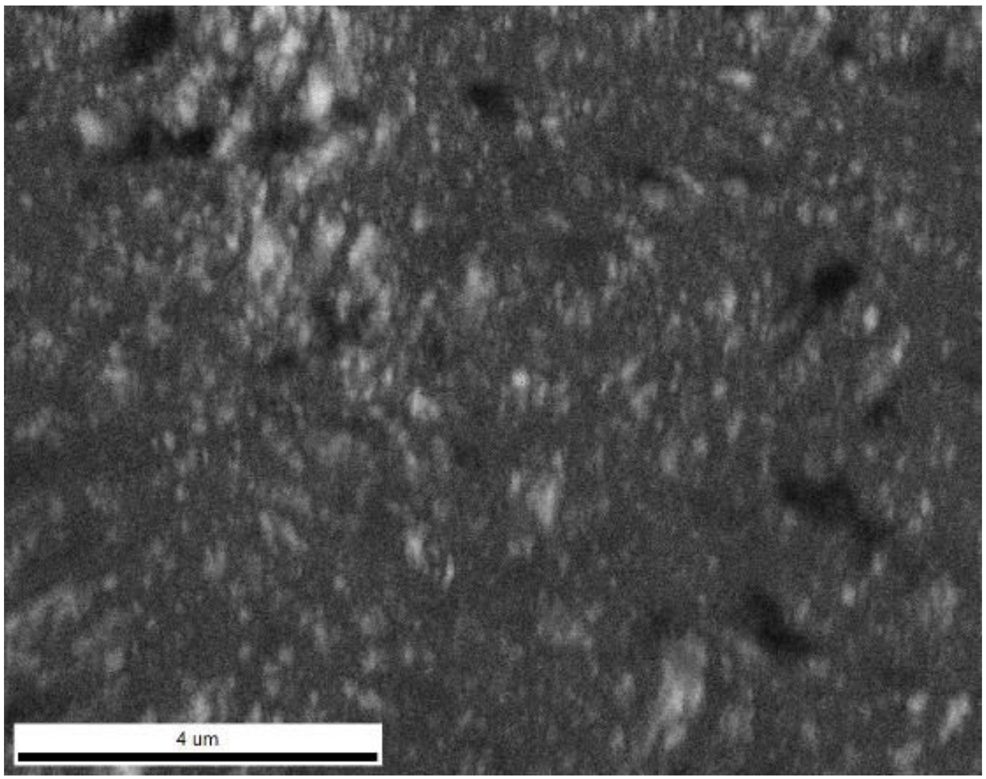
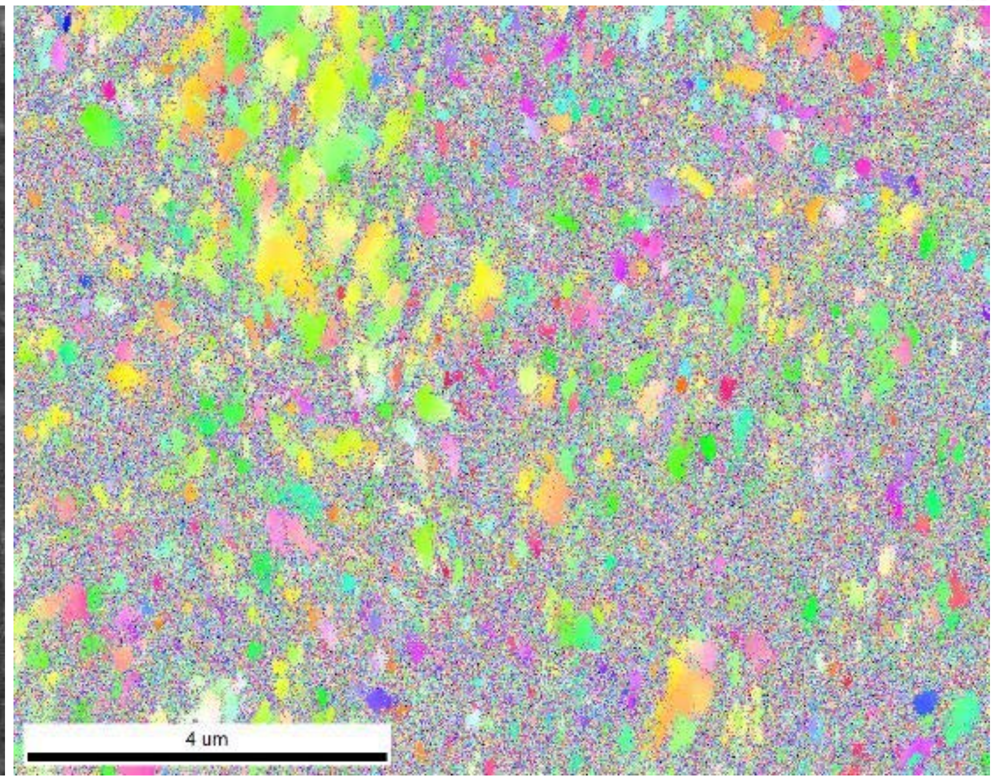
Titanium nitride coating – Hough results



- Weak patterns combined with strong overlap at boundaries prevents successful indexing



IQ map after pattern intensity homogenization



IPF map: Indexing success: 23.2%

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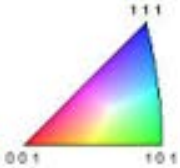
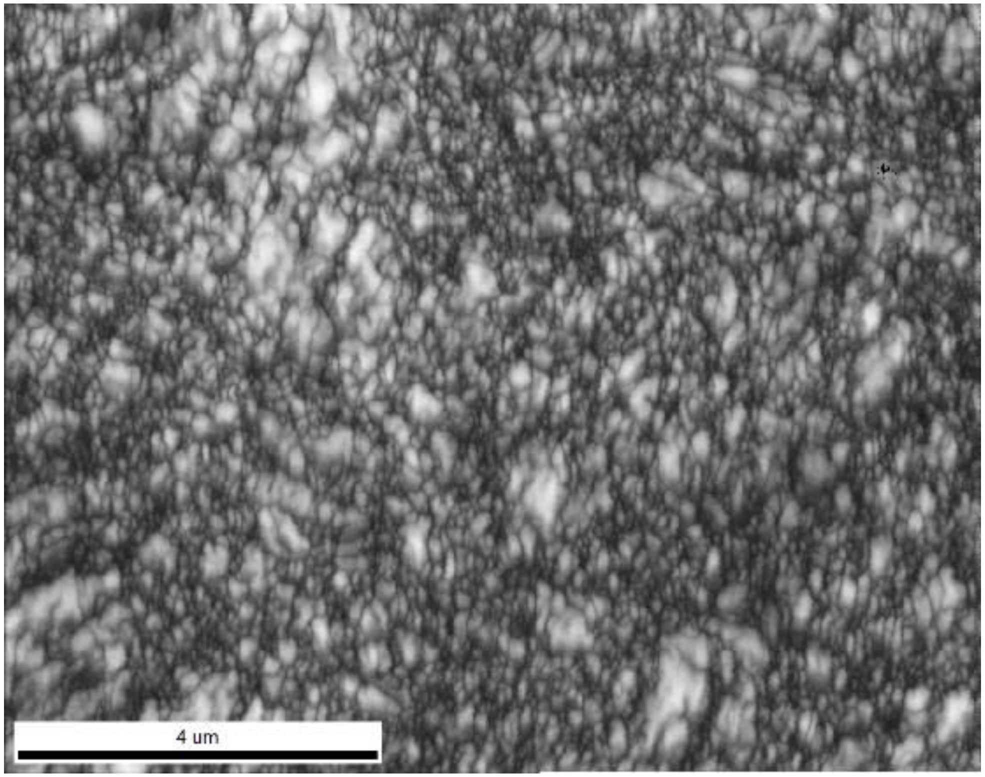
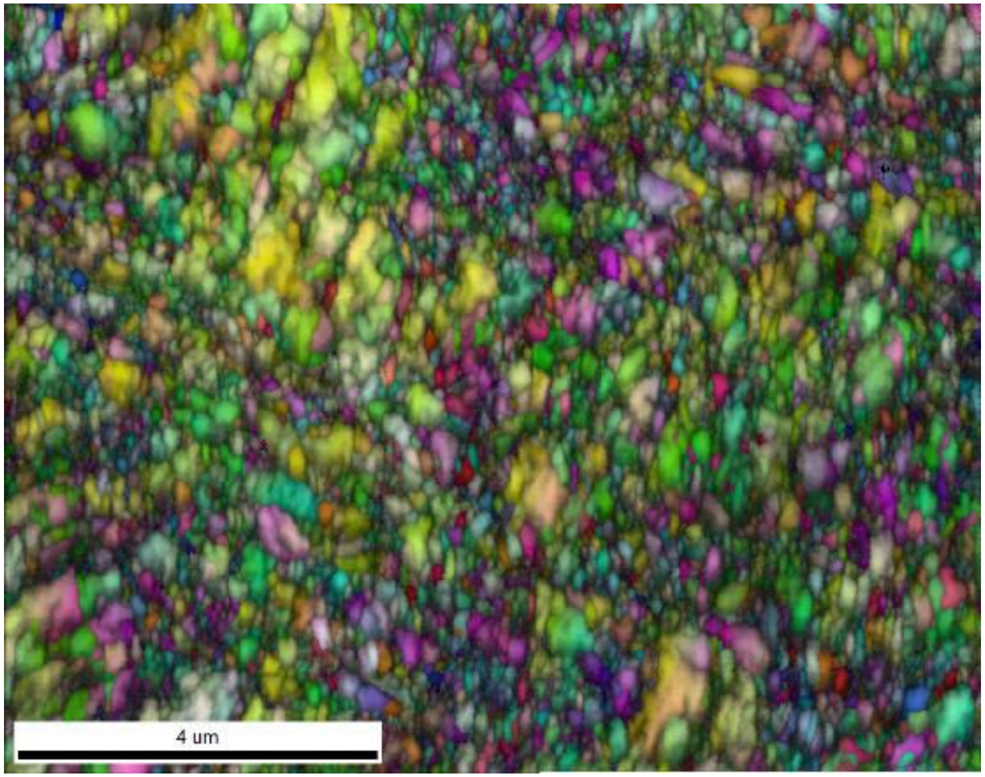
Titanium nitride coating – Spherical indexing results

- Indexing results with spherical indexing





Spherical correlation map



IPF map – indexing success: 96%

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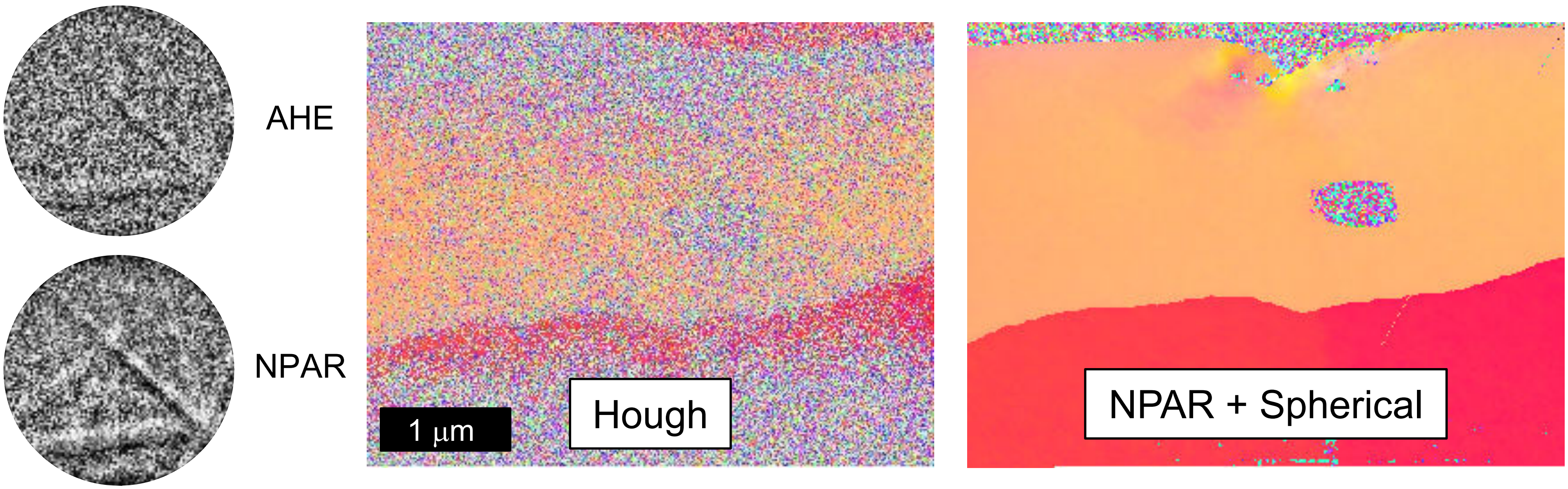
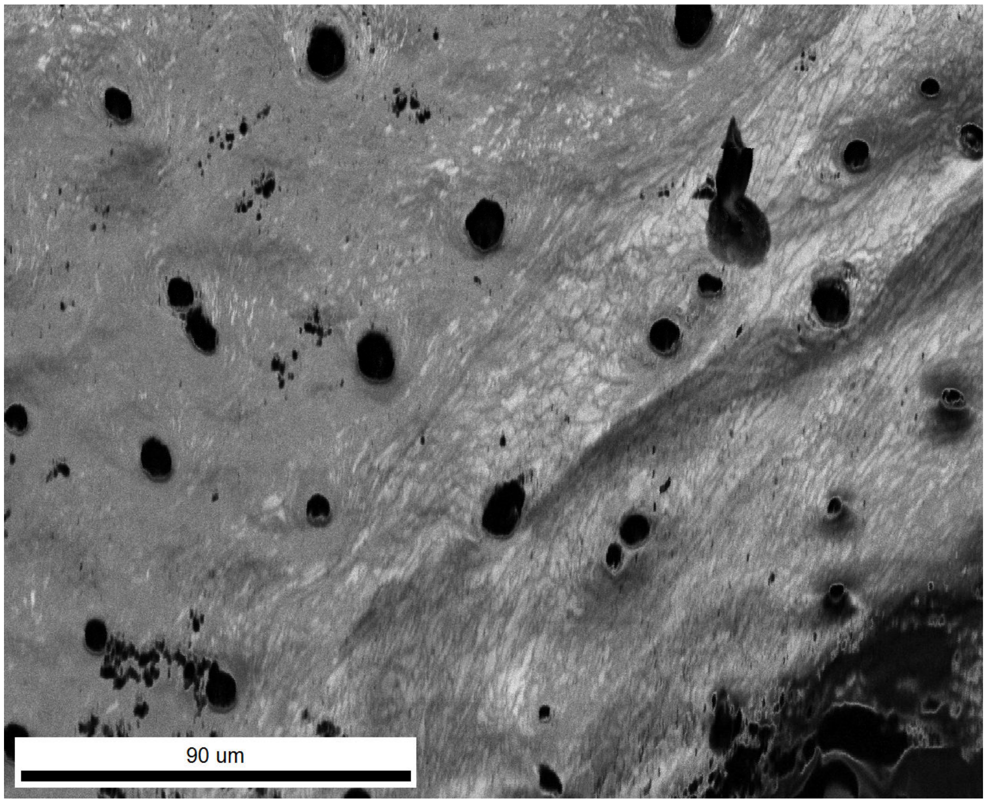
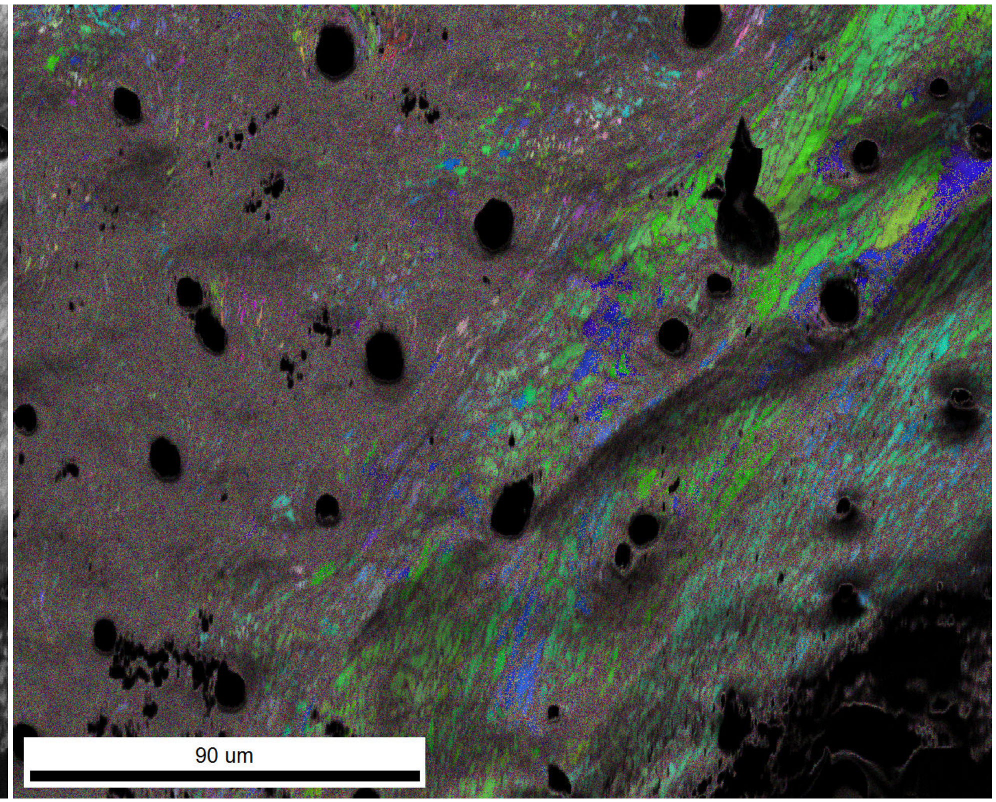
TKD - ferrite



- TKD samples often show significant gradients in pattern quality and intensity due to thickness variations
  - Image processing using AHE (adaptive histogram equalization) allows consistent matching with the master patterns
- Band width may change strongly within patterns due to the projection geometry
- Spherical indexing can help overcome these challenges

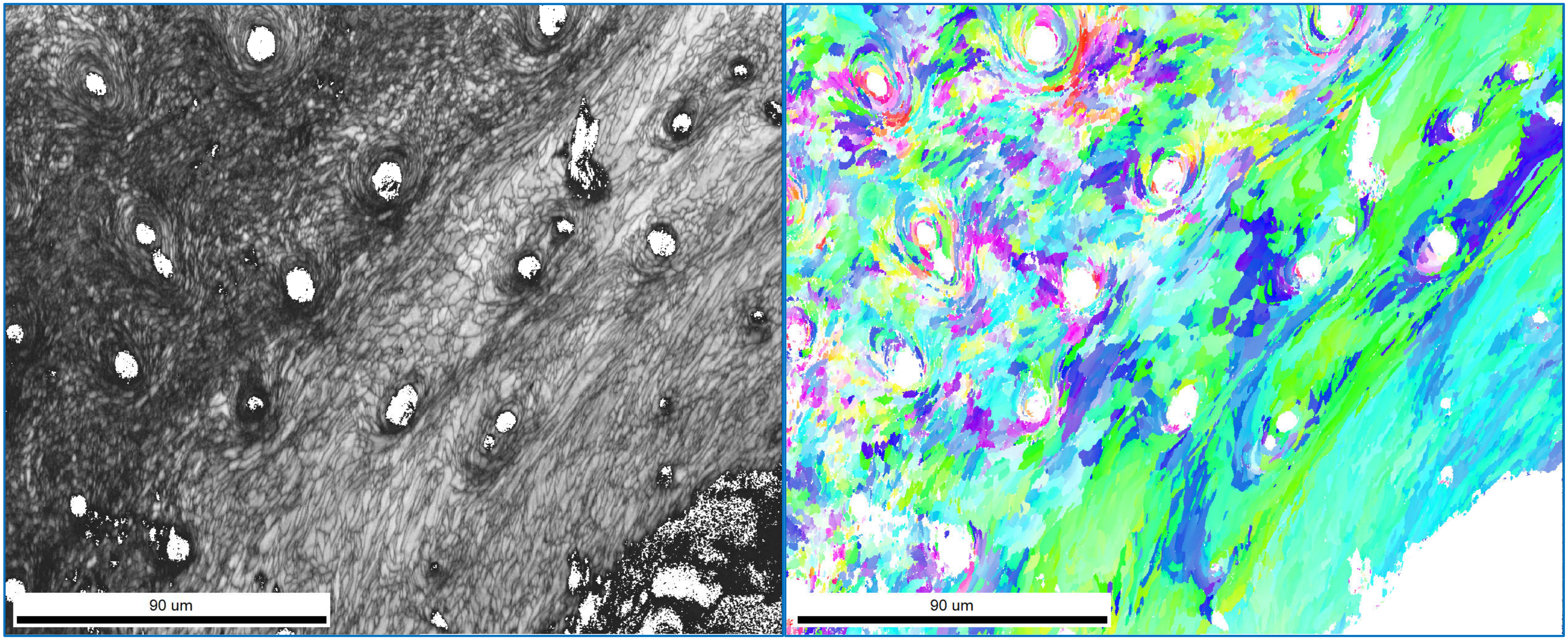
Beam sensitive bio-mineral example: brachiopod cross-section





Specimen courtesy of E. Griesshaber, Geo- und Umweltwisschaften, Ludwig-Maximilians-Universität München

Brachiopod shell – spherical indexing



Specimen courtesy of E. Griesshaber, Geo- und Umweltwisschaften, Ludwig-Maximilians-Universität München

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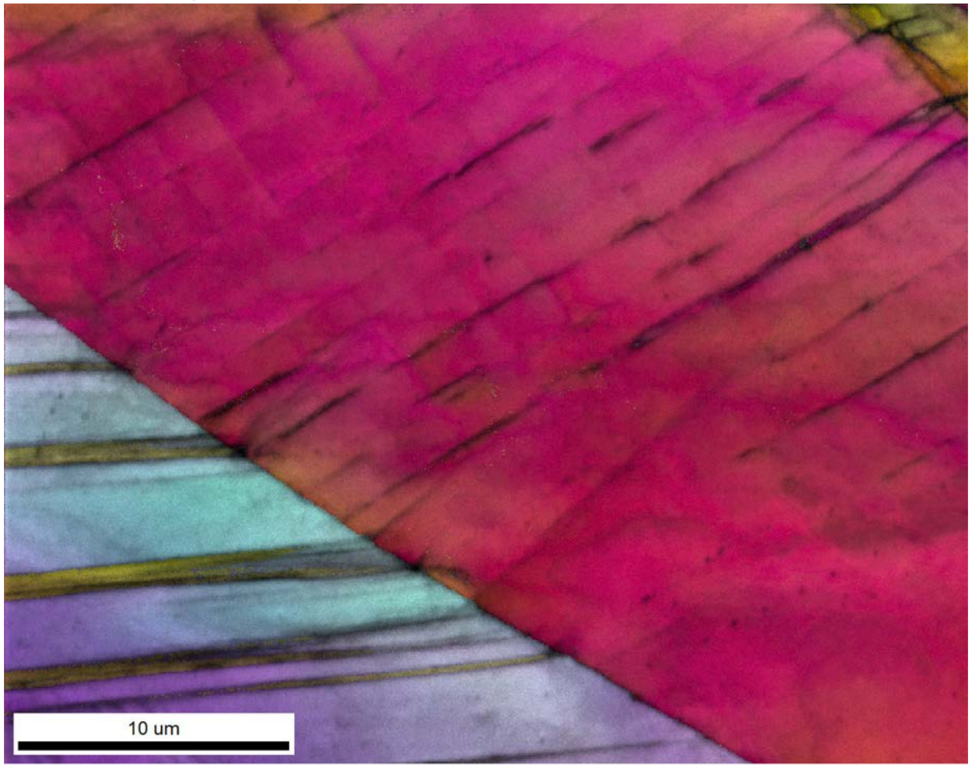
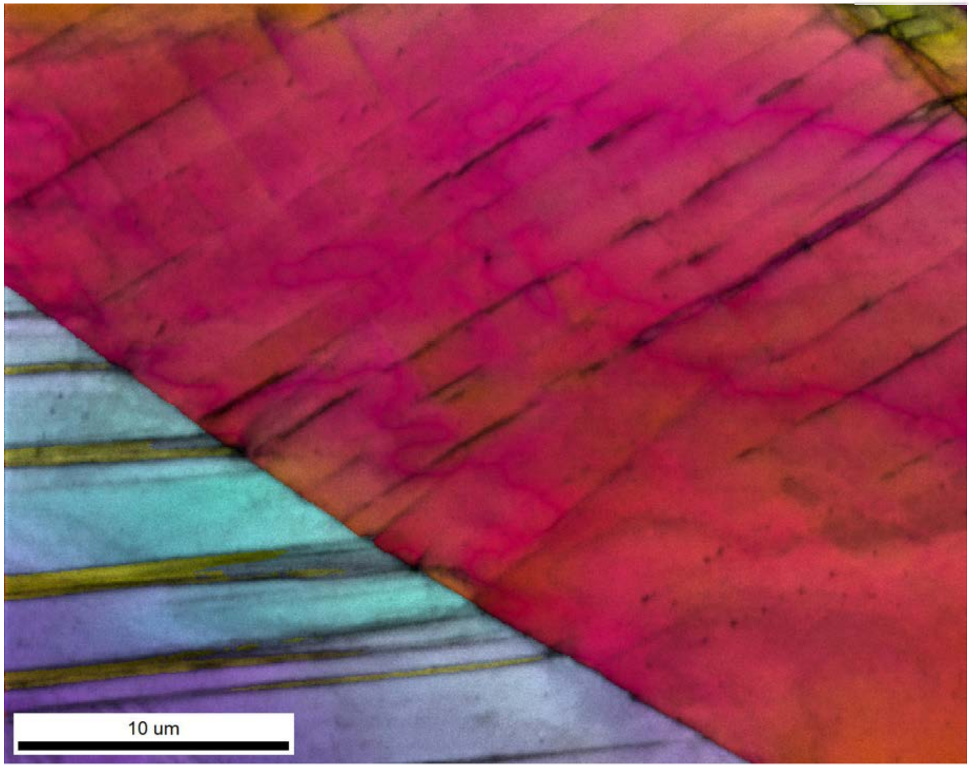
Spherical indexing – orientation precision improvements

- Because spherical indexing uses the entire pattern, you can get greatly improved orientation precision when working with good patterns



Triplet voting indexing



Spherical indexing

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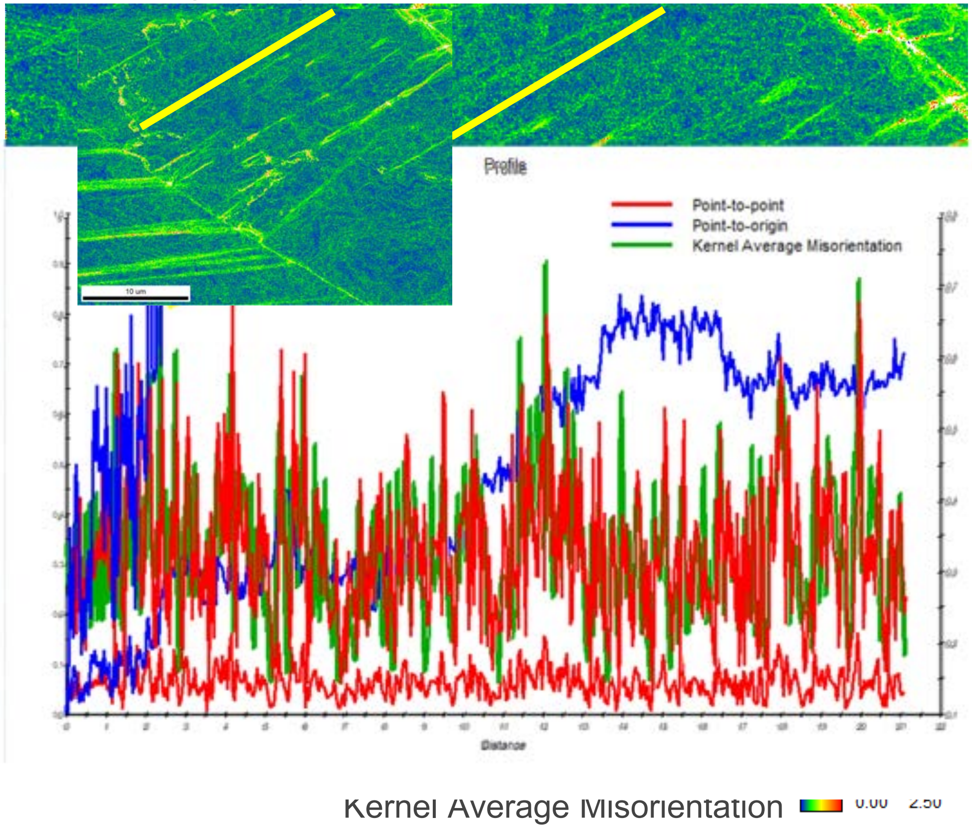
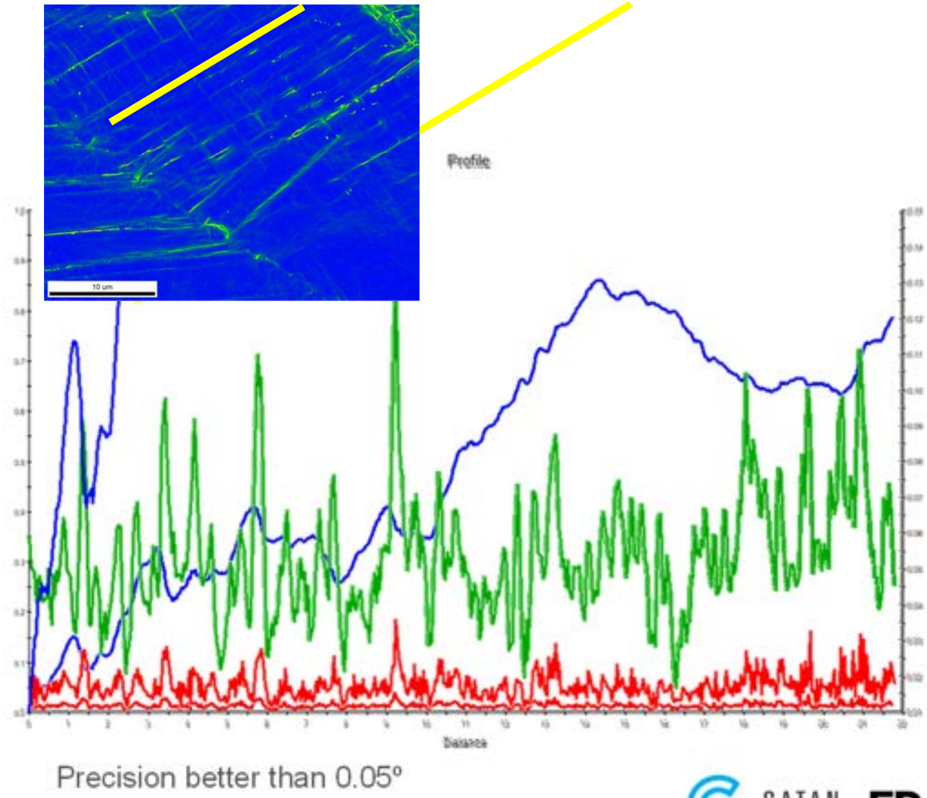


Triplet voting indexing



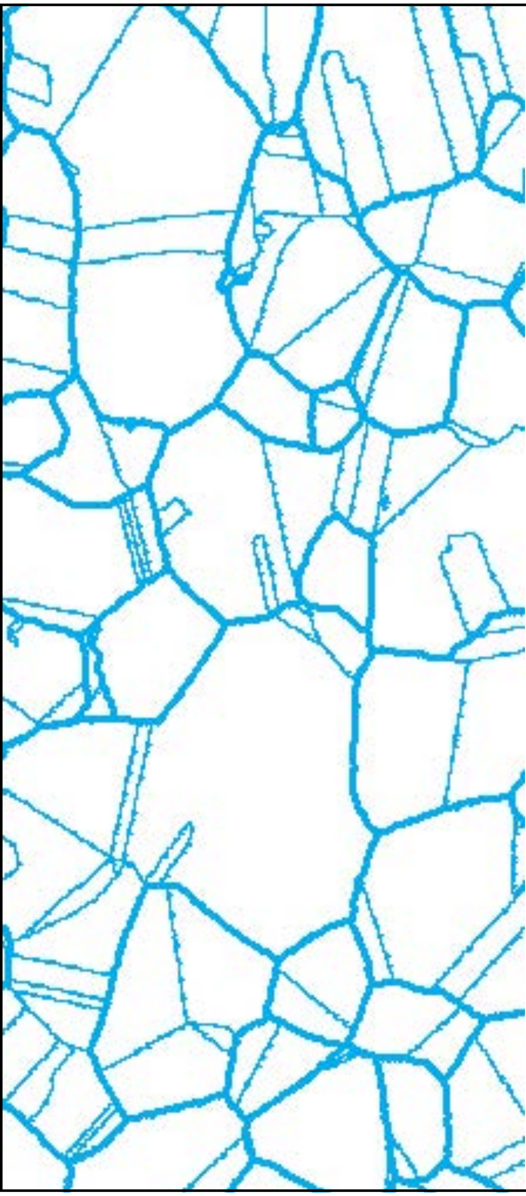
Spherical indexing

Summary

Spherical Indexing

- + As robust as Dictionary Indexing but faster
- + Higher angular precision than Hough
- + No pre-calculation of a dictionary required
- − Slower than Hough
- − Master Pattern required
- Image Processing: Adaptive Histogram Equalization

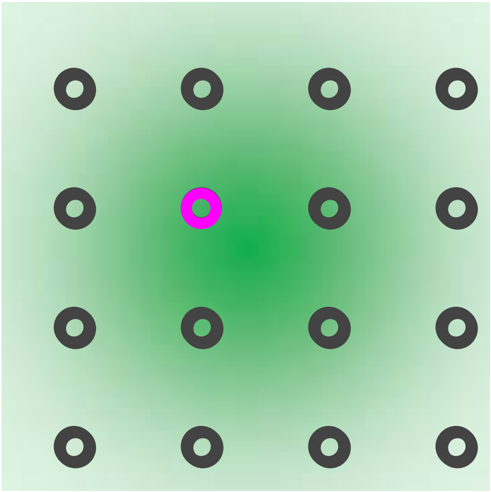
The robustness of spherical indexing doesn’t mean you no longer have to prepare your samples well!!!



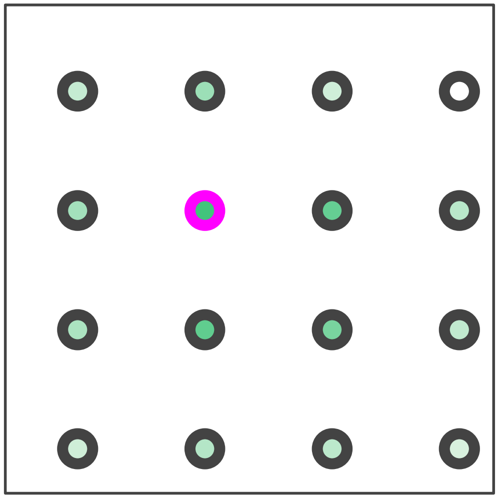
Orientation refinement

Orientation refinement: 2D example

Darker green
↓
Better model fit



Continuous Orientation Space



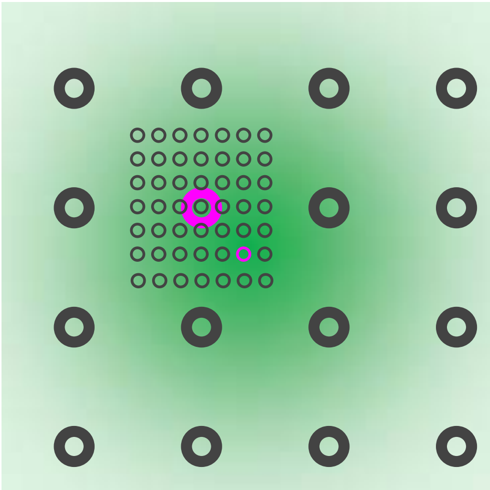
Discrete dictionary grid points

Refinement for increased angular precision

- No good match is found for too large of step size
- Too small of orientation step sizes are intractable
- Problem becomes intractable before desired precision

Orientation refinement: 2D example

Darker green
↓
Better model fit



Realspace refinement

- No good match is found for too large of step size
- Too small of orientation step sizes are intractable
- Problem becomes intractable before desired precision
- Re-index using a high precision localized dictionary (in 3D)

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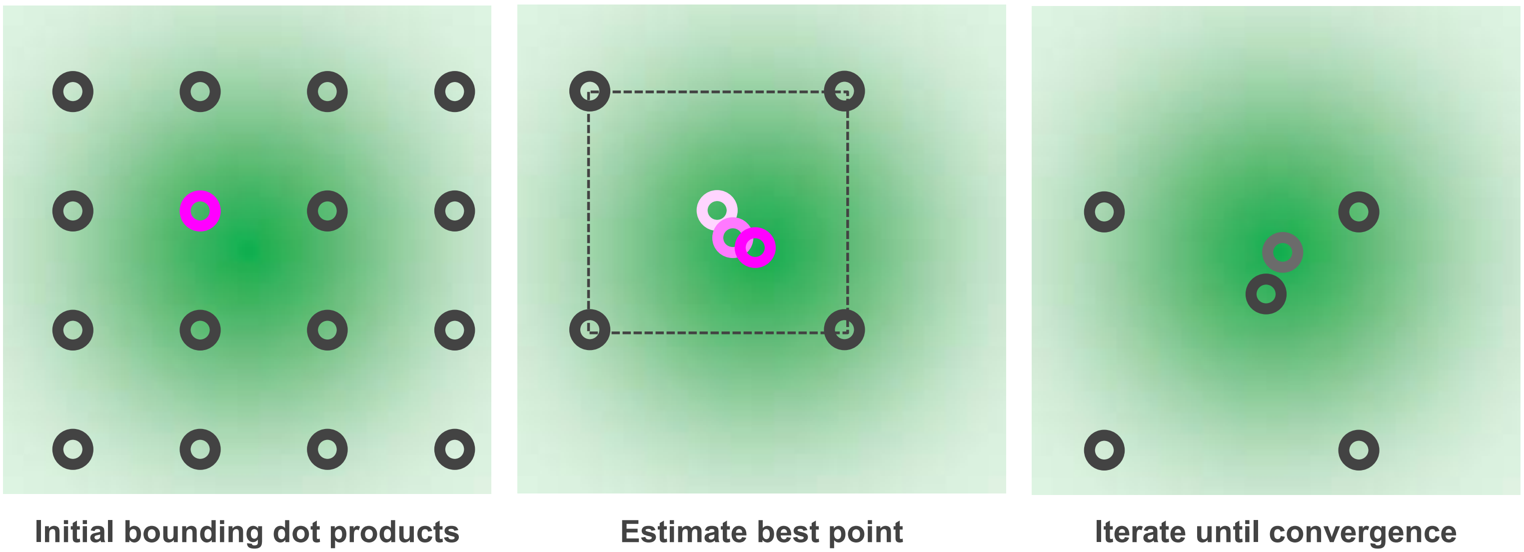
Orientation refinement: non-linear optimization example

Bound **O**ptimization **By** **Q**uadratic **A**pproximation

- **B**ounds are based on indexing grid
- **O**ptimize to maximize normalized dot product
  - At each iteration choose a new iteration point by:
  - Fitting a **Q**uadratic **A**pproximation to the current points
  - Selecting the maxima of the quadratic constrained by the bounds

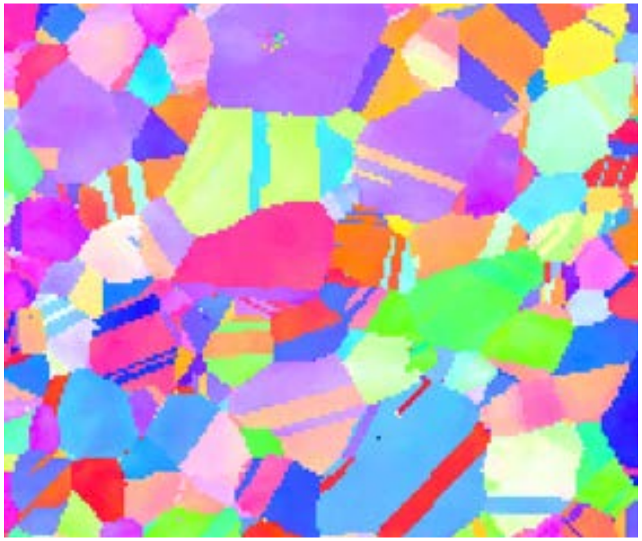


- Non-linear iterative optimization to refine orientation
- Bounded by specified dictionary parameters
- We are using a variant on the BOBYQA approach more suitable to EBSD

EL Pang, PM Larsen and CA Schuh (2020) "Global optimization for accurate determination of EBSD pattern centers“ *Ultramicroscopy* **209**, 112876.
MJ Powell (2009) The BOBYQA algorithm for bound constrained optimization without derivatives. *Cambridge NA Report NA2009/06, University of Cambridge, Cambridge*, 26-46.

Realspace refinement

Hough Based



Continuous orientations

2.5° Dictionary

Discretized indexable orientations
before refinement

Refined

Continuous orientations after refinement

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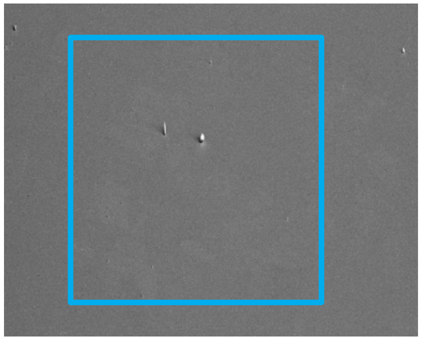
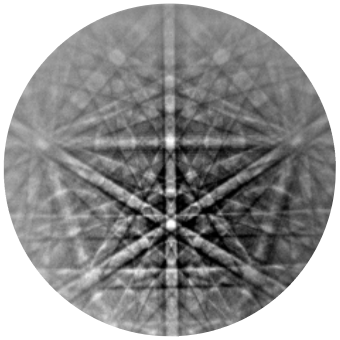
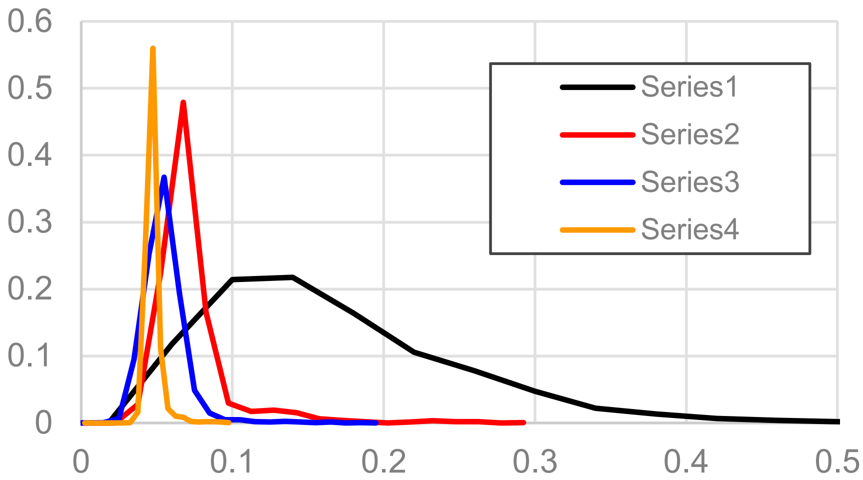
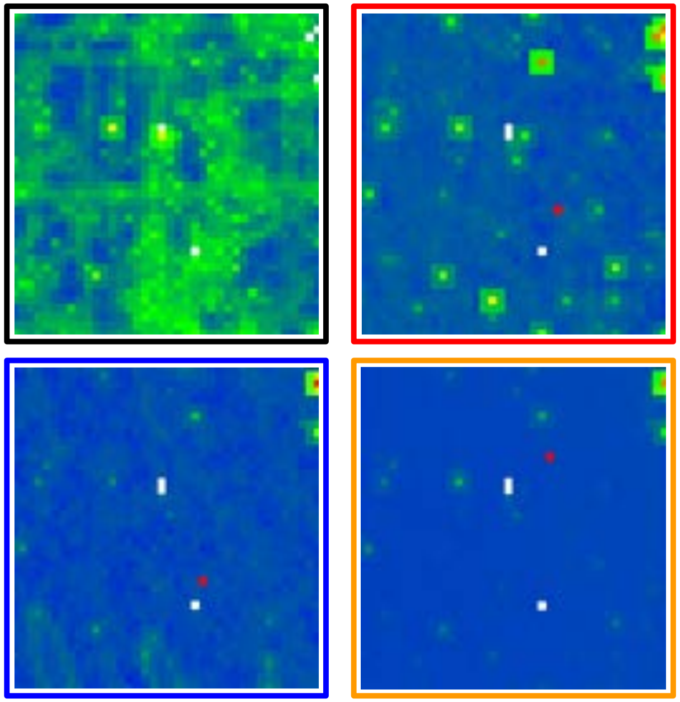
Preliminary precision tests

- Single crystal silicon
- 1 mm x 1 mm scan
- 480 x 480-pixel patterns
- 25-micron step size

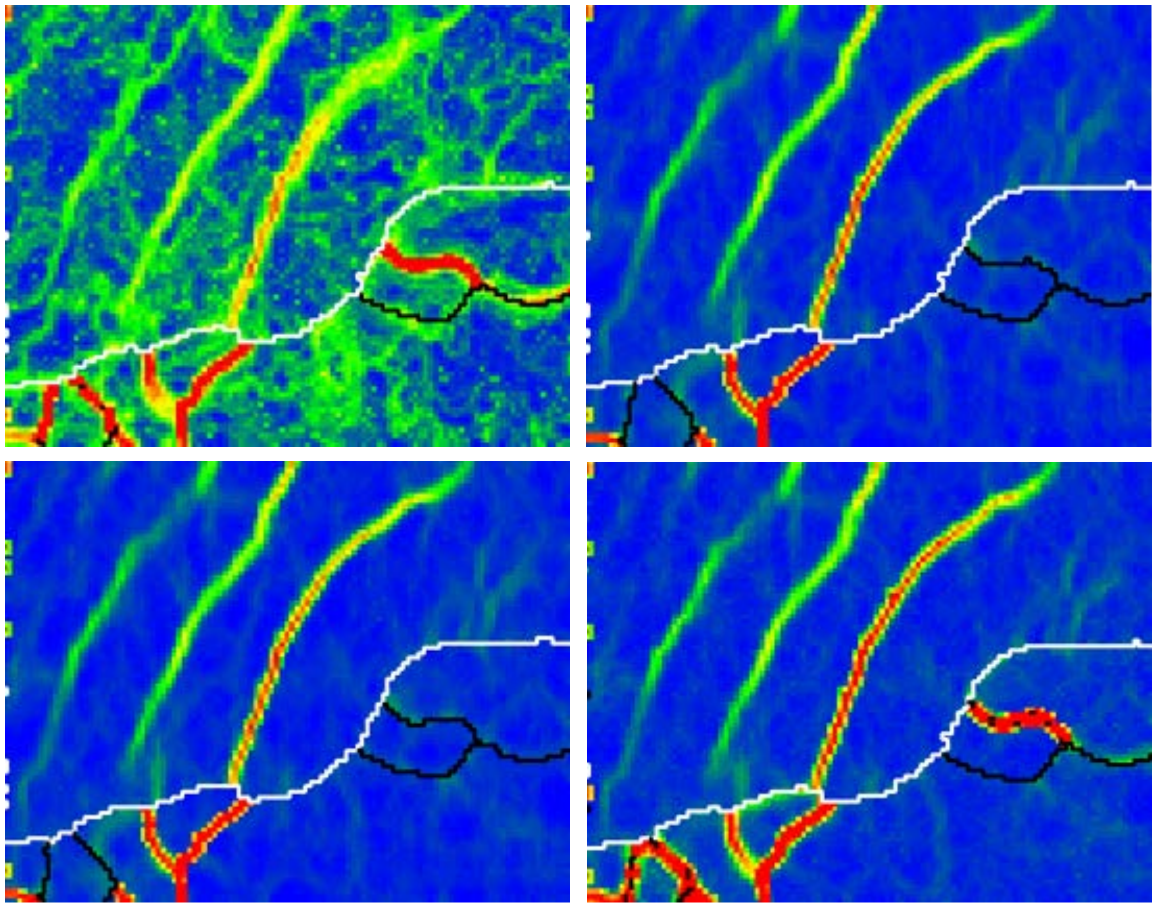








Cross-correlation comparison



Hough

Hough + Refinement

Spherical + Refinement

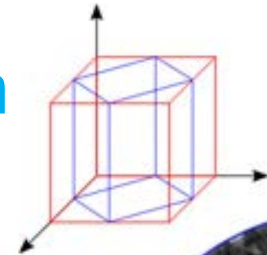
OpenXY
Courtesy Josh Kacher
Georgia Tech University

5-15° > 15°

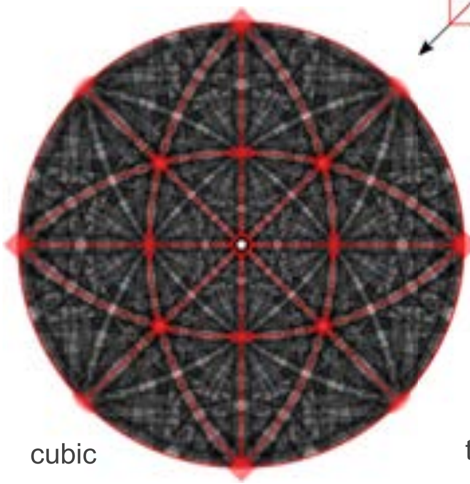
0° 5°

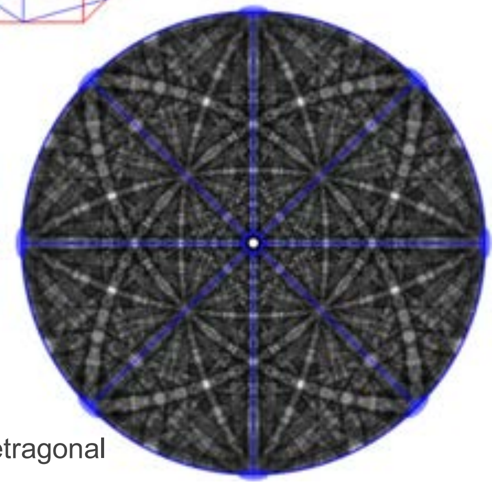
2μm

ZrO_2 pseudosymmetry & phase differentiation



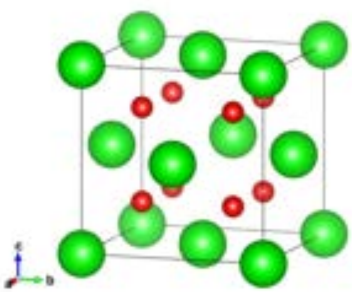
- Tetragonal phase is small strain away from cubic
- Double cell c/a ratio 1.0134
- ~0.7% strain between phases
- Type and severity of pseudo symmetry is predicted well w/ spherical cross correlation

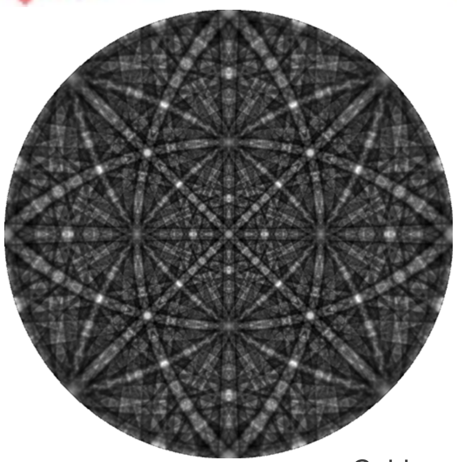


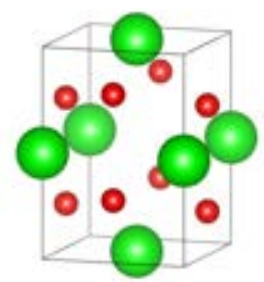


Normalized cross-correlation between the master patterns for each phase for all rotations

Max Cross Correlation	Cubic	Tetragonal
Cubic	60@<111> 29.9%	45@<001> 96.3%
Tetragonal		90@<110> 90.5%

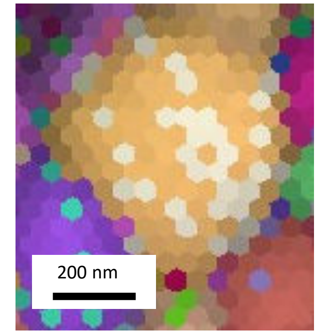




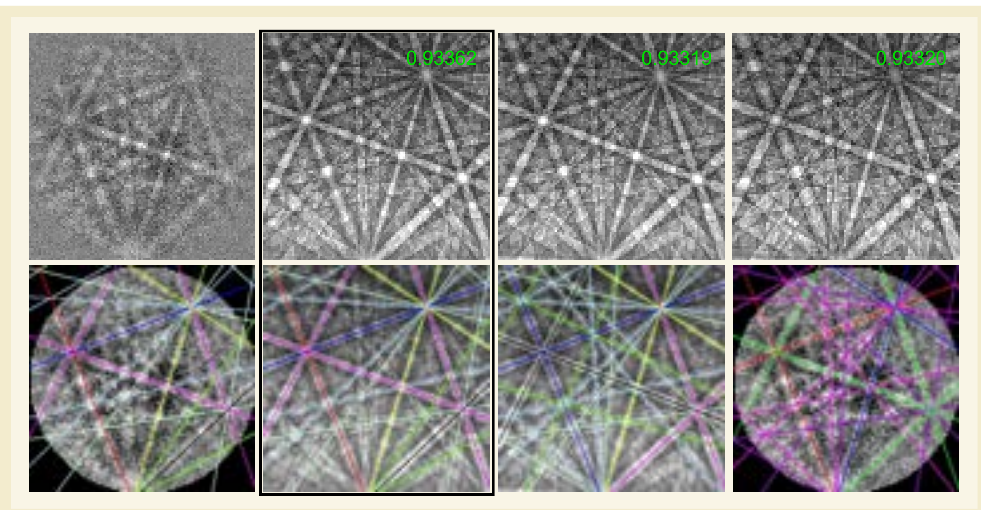


Cubic vs tetragonal rotated 45 @ z

ZrO_2 pseudosymmetry



Experimental Pattern | Tetragonal Orientation | Tetragonal Pseudosymmetric Orientation | Cubic Orientation



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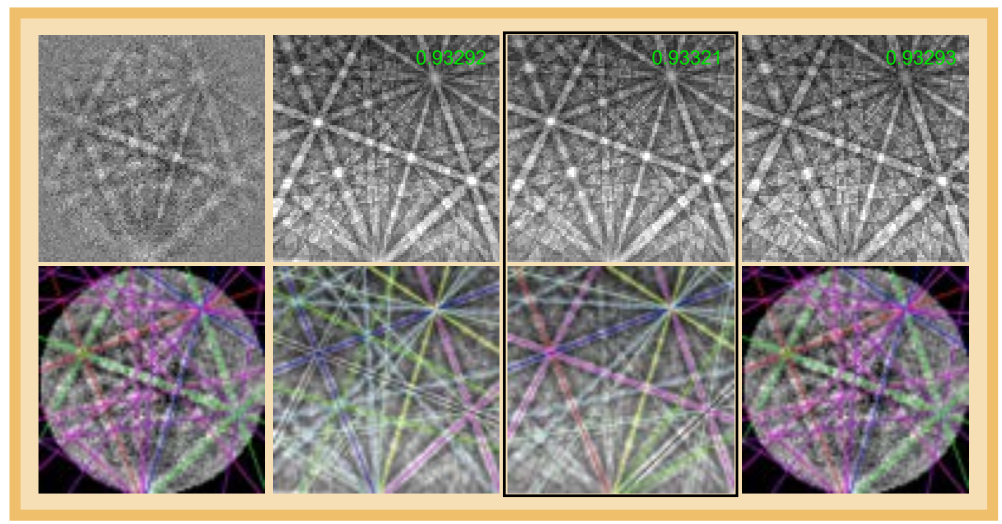

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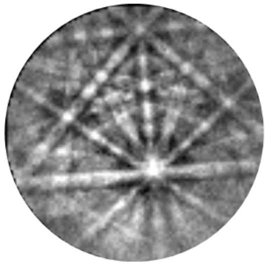
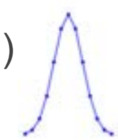
Pseudosymmetry / phase differentiation workflow



Experimental Pattern

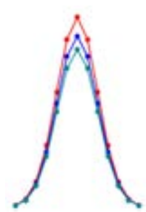
- Find the orientation with the highest normalized dot product (NDP) for the cubic phase (q_c)
  - Refine the cubic orientation (q_c^R)

- Find the orientation with the highest NDP for the tetragonal phase -or-
- Calculate the tetragonal orientation related to the cubic orientation (q_t)
  - Refine the tetragonal orientation (q_t^R)



- Calculate the pseudosymmetric variation of q_t (q_{t2})
  - Refine q_{t2} (q_{t2}^R)

- Select refined orientation with the maximum NDP



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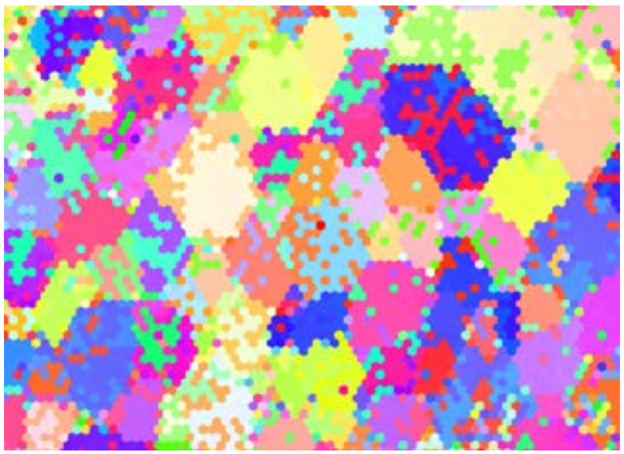
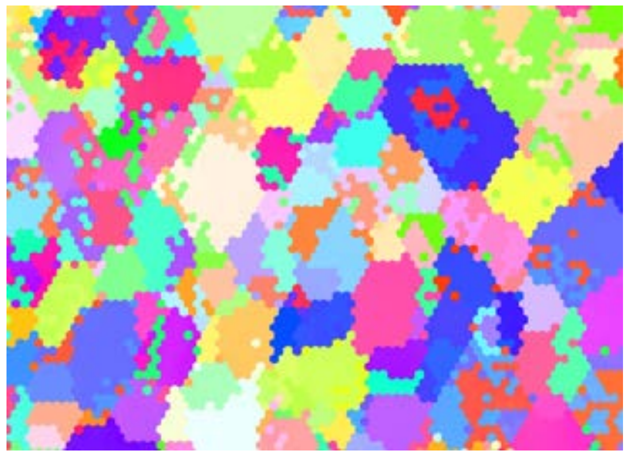
ZrO_2 tetragonal pseudosymmetry results

Hough

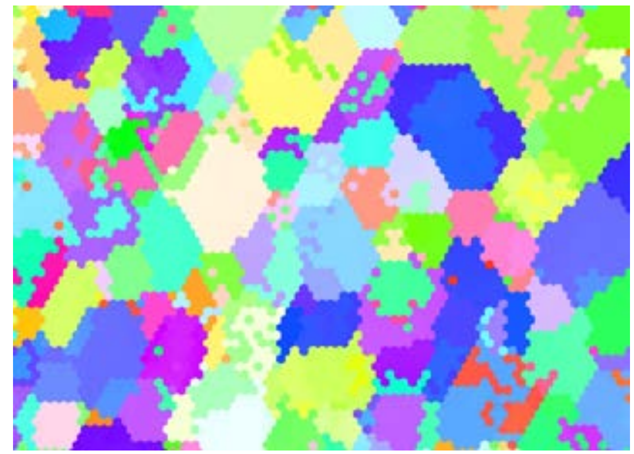


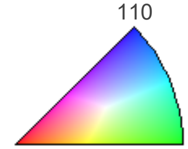
μm

Spherical



Spherical + Pseudosymmetry Analysis

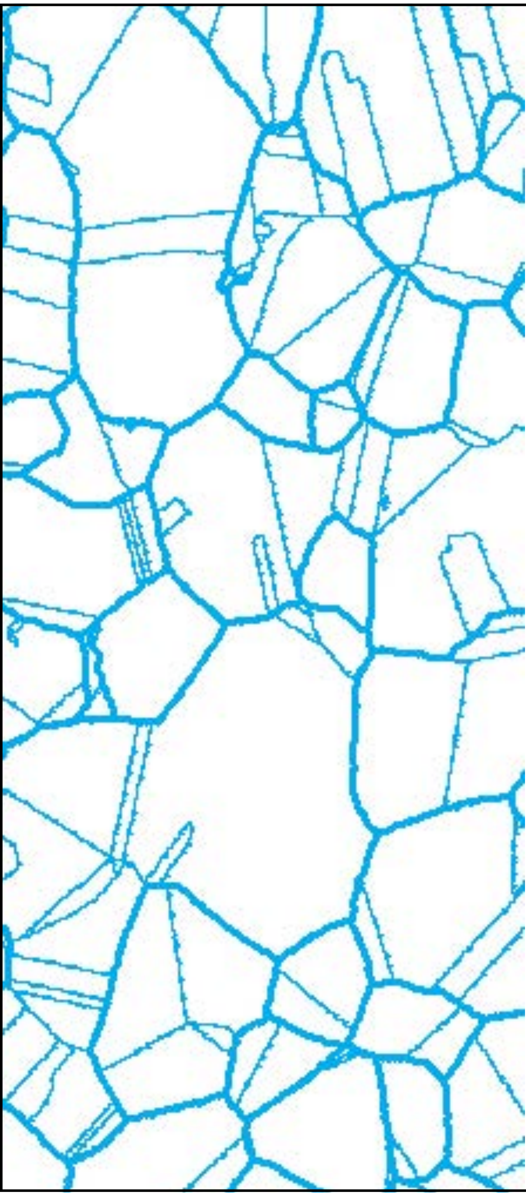




Summary – orientation refinement

Refinement (OIM Matrix)

- Can start with Hough, DI, or SI results
- Improved angular resolution
  - Improved GNDs and KAMs
  - Rivaling HR-EBSD orientation precision (but no lattice distortion measurement)
- Master pattern and recorded patterns required
- Yet to explore:
  - Effects of pattern size and quality
  - Effects of lattice distortion (HR-EBSD)
  - …



Thank you for your attention