USING MATLAB FOR MATERIALS DESIGN: DESCRIBING GRAIN ORIENTATIONS IN METALS

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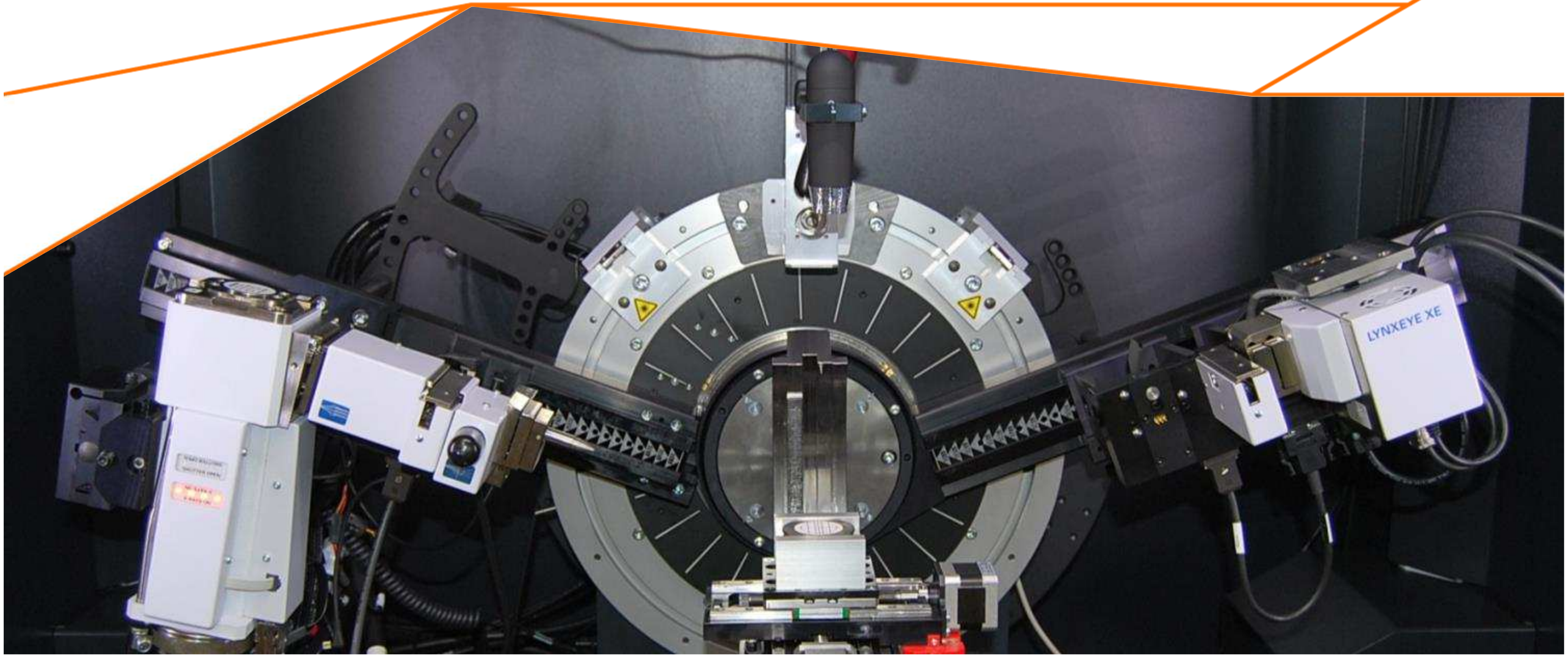

THE SANDVIK GROUP

MACHINING SOLUTIONS

MINING AND CONSTRUCTION

MATERIALS TECHNOLOGY

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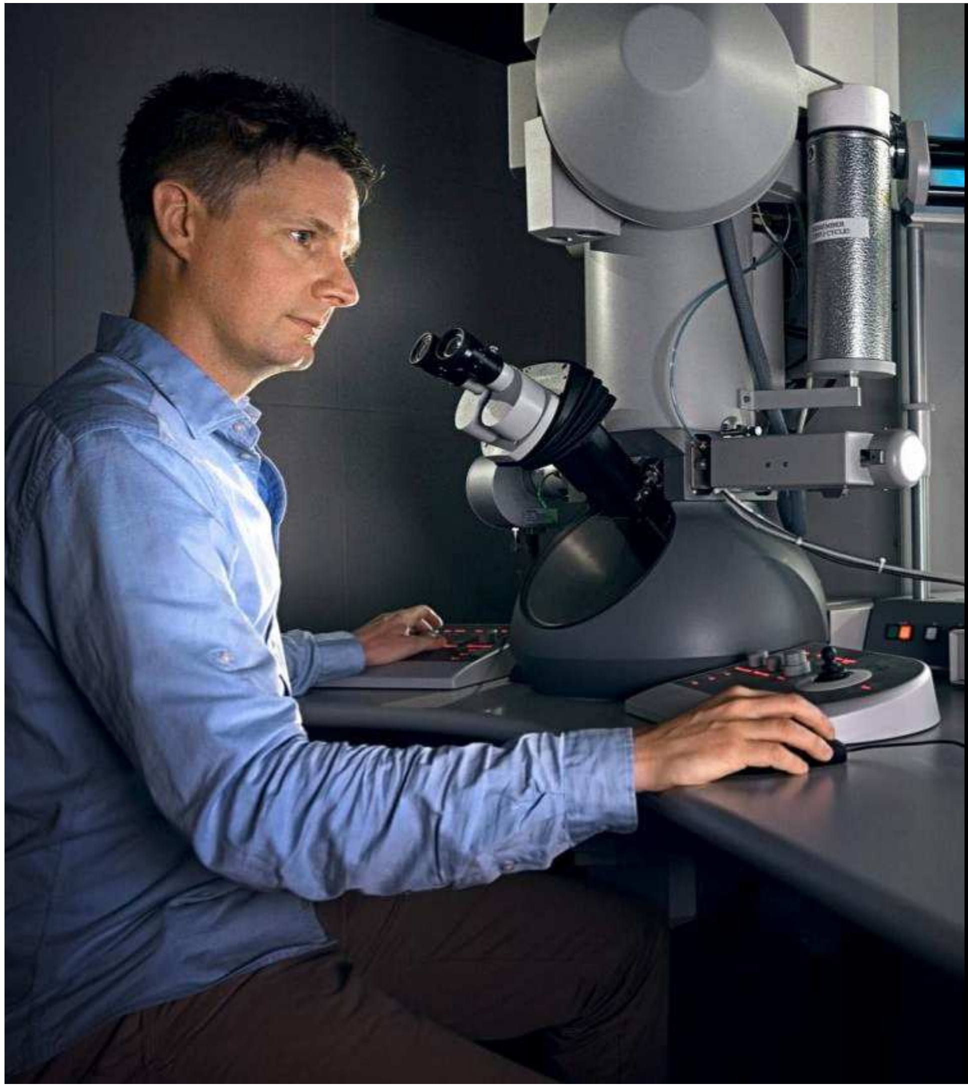

SANDVIK MATERIALS TECHNOLOGY

THE STEEL RESEARCH CENTER IN SANDVIKEN

- ~ 230 staff
- ~ 900 different materials
- Steels
  Ni-base
  Zircaloy
  Titanium
  Powder Metallurgy
  Thin Film Coatings
  ...

ANALYTICAL TASK

- Zircaloy tubes are used for packaging nuclear fuel
- To prevent cracking, it is important to control grain orientation in the tube wall.
- Grain orientation is characterized with X-ray Diffraction
- Data evaluation, quantification and reporting is made with MATLAB + MTEX
- MTEX is an open source toolbox, developed by Prof. Dr. Ralf Hielscher *et al.* at TU-Chemnitz. It contains tools for handling material orientation data:

  https://mtex-toolbox.github.io/

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ZIRCALOY
TUBING FOR NUCLEAR FUEL

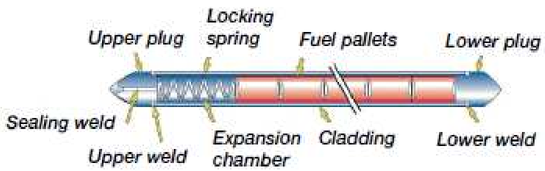
NUCLEAR FUEL RODS IN ZIRCALOY

CHARACTERISTICS

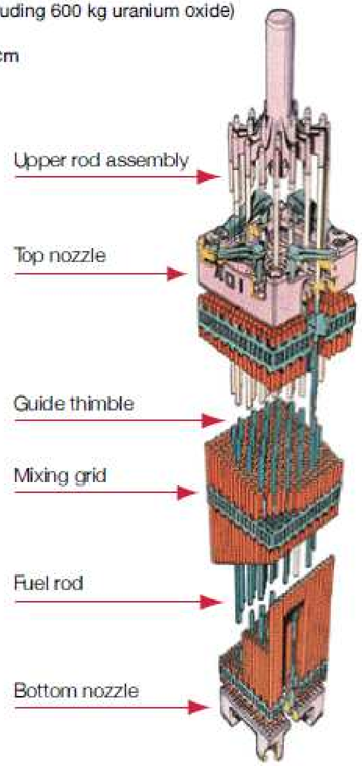
- Right in the reactor core
- Need a material with low neutron capture cross section, hence Zr-Nb alloys
- Good control of impurities
- High demands on the metallurgical process
- High demands on the rolling process

Uranium pellet:
Weight: 7 to 8 g
Height: 10 to 15 mm
Width: 8 to 9 mm

Fuel rod:
Weight: 2,2 kg
Length: 4 m
Width: < 1 cm
contains about 300 pellets



Fuel assembly:
Weight: 800 kg (including 600 kg uranium oxide)
Height: > 5 m
Width: 21 cm x 21 cm
17 x 17 rods



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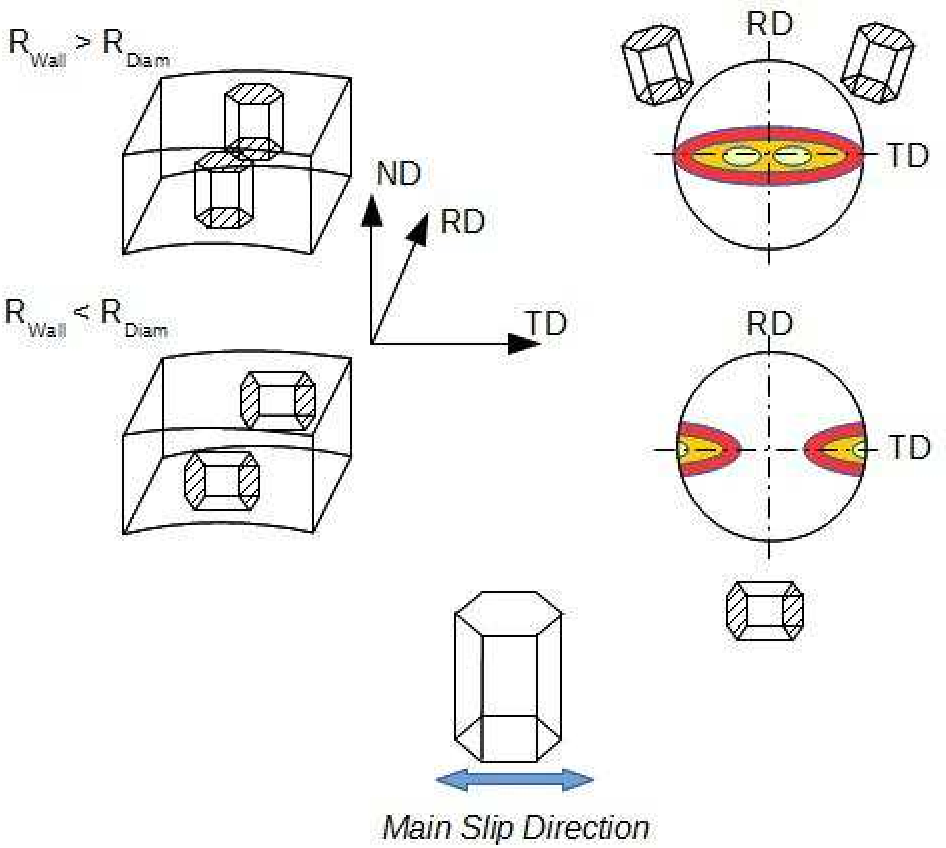
ROLLING TUBES OF HEXAGONAL MATERIALS



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TEXTURE MEASUREMENT WITH X-RAY DIFFRACTION (XRD) AND MATLAB

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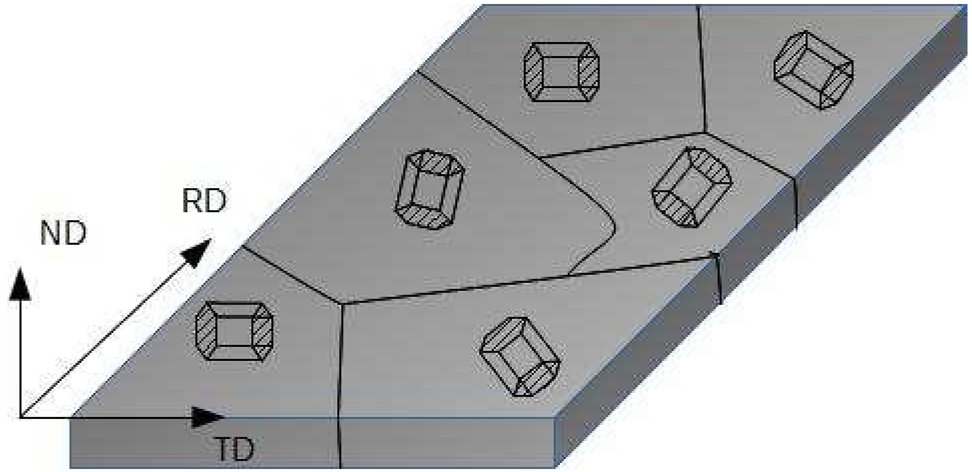
THE ORIENTATION DISTRIBUTION FUNCTION (ODF)

Orientation Distribution (Euler Angles)

- Describes the orientation of all crystals in the sample
- For a cubic system, a 90° x 90° x 90° space is sufficient
- $\frac{dV}{V} = f(g)dg = f(\varphi_1, \Phi, \varphi_2)\frac{sin\Phi}{8\pi^2}d\Phi d\varphi_1 d\varphi_2$



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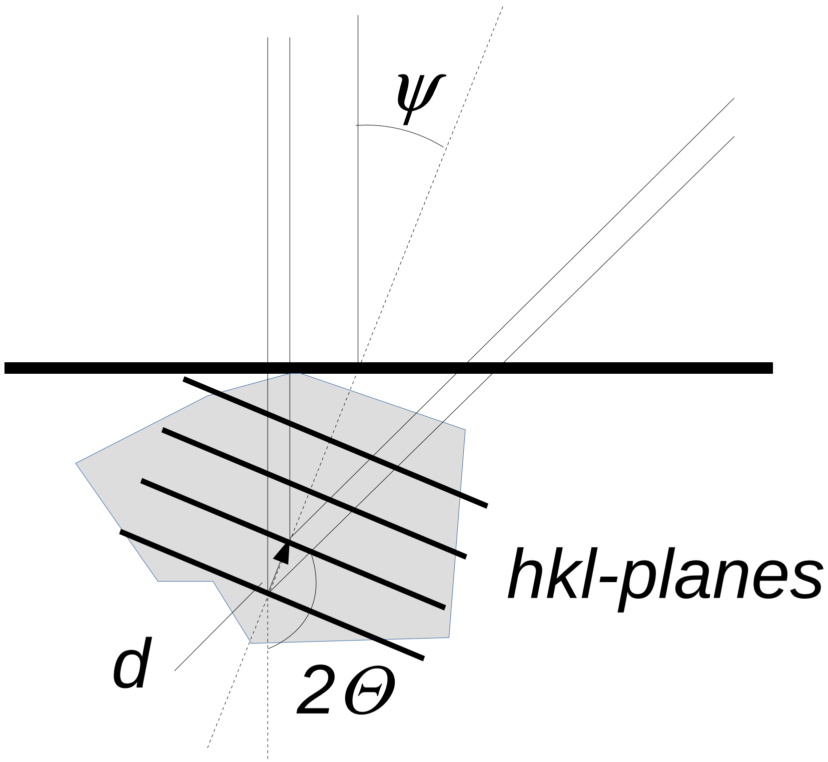
X-Ray Diffraction Measures Lattice Spacing



- Only crystals with the correct orientation fulfills the Bragg condition, i.e.
  $\Delta\boldsymbol{k} = \boldsymbol{G}$, *alternatively*

  $$d = \frac{2sin\theta}{n\lambda}$$

- Record the intensity of each diffraction peak in a 5°x 5° grid on the hemisphere above the sample.

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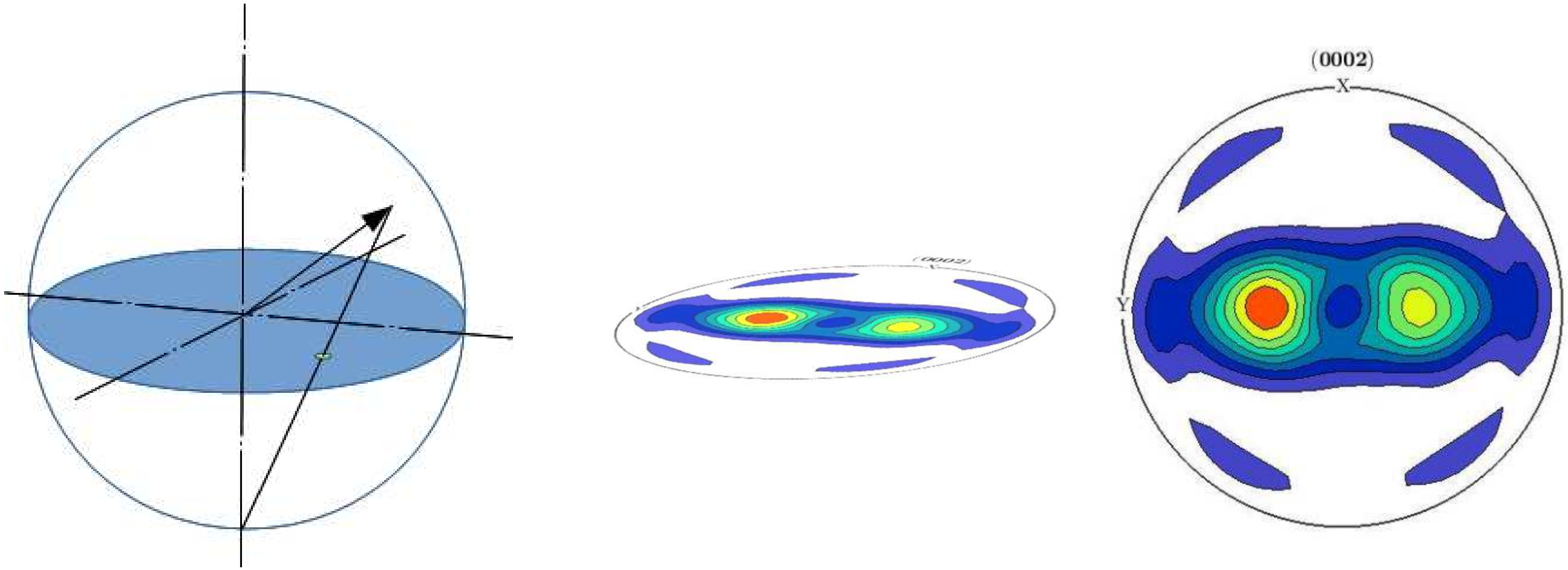
SPHERICAL PROJECTIONS



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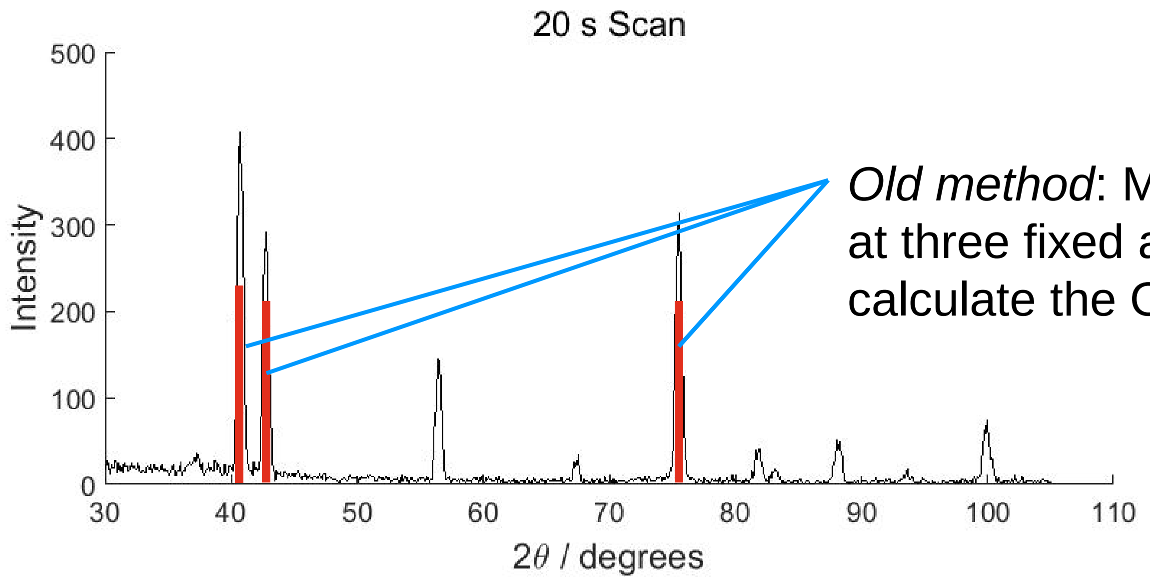
FAST DIFFRACTOGRAM RECORDING



Old method: Measure intensity at three fixed angles to calculate the ODF.

New method: Extract intensities by curve fitting!

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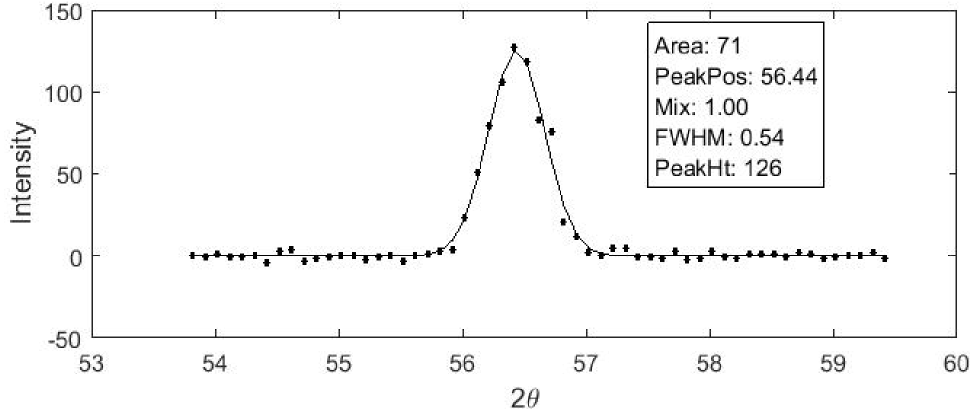
SYNTHETIC LINE PROFILES – PSEUDO VOIGT

$$I(2\theta) = I_{max}\left[me^{-4ln2\left(\frac{2\theta-p}{w}\right)^2} + \frac{1-m}{1+4\left(\frac{2\theta-p}{w}\right)^2}\right]$$

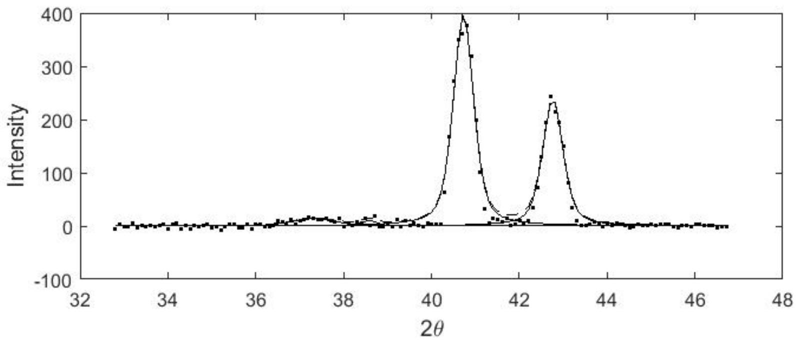
where

I_{max} – peak height
p – peak position
w – full width at half max
m – Gauss / Lorentz mix
2θ – Scale for the x-axis

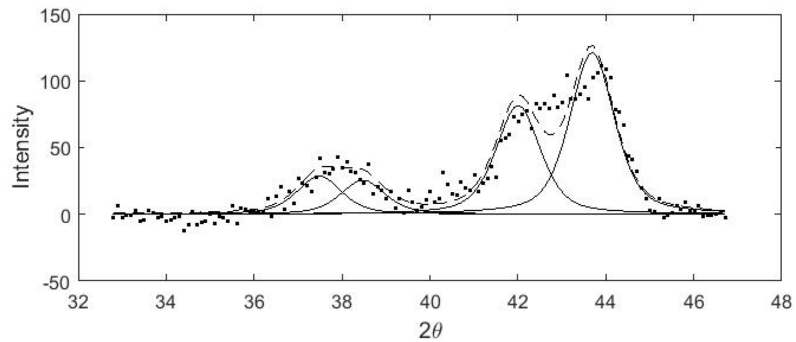


MULTI-PEAK FITTING

No tilt –
diffraction vector normal to sample surface.
OK without fitting.



High tilt –
diffraction vector grazing to sample surface.
Peak fitting necessary!



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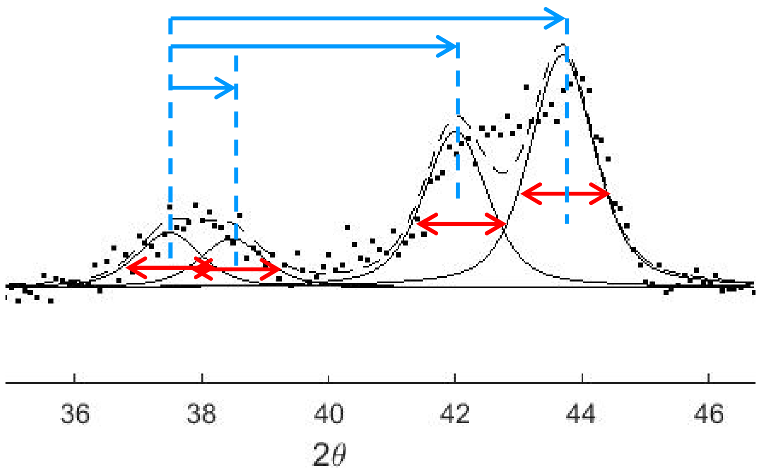
MULTI-PEAK FITTING

- Fix positions relative to each other
- Same width for all peaks
- Set the peak shape to Gaussian
- Intensity is the only completely free variable



Problem: Pass *x* fit variables and (*n*-*x*) fix variables to the solver.

Solution:

- Create a peak shape function:

```
function a = voigtNpeaks(p, xData, fitNdx, fixNdx, fixPrms, nPeaks)
```

- Pass it to the solver (1 peak example):

```
fitNdx =  [1 2];
fixNdx =  [3 4];
fixPrms = [0.9 1];
fitPrms = [1000 43 0.9 1];
```

```
newFitPrms = lsqcurvefit(@voigtNpeaks, fitPrms(fitNdx), x, y, ...
    lb(fitNdx), ub(fitNdx), opt, fitNdx, fixNdx, fixPrms, region.nPeaks);
```

- Finally, re-create the parameter vector inside the peak shape function:

```
p(fitNdx) = fitPrms;
p(fixNdx) = fixPrms;
```

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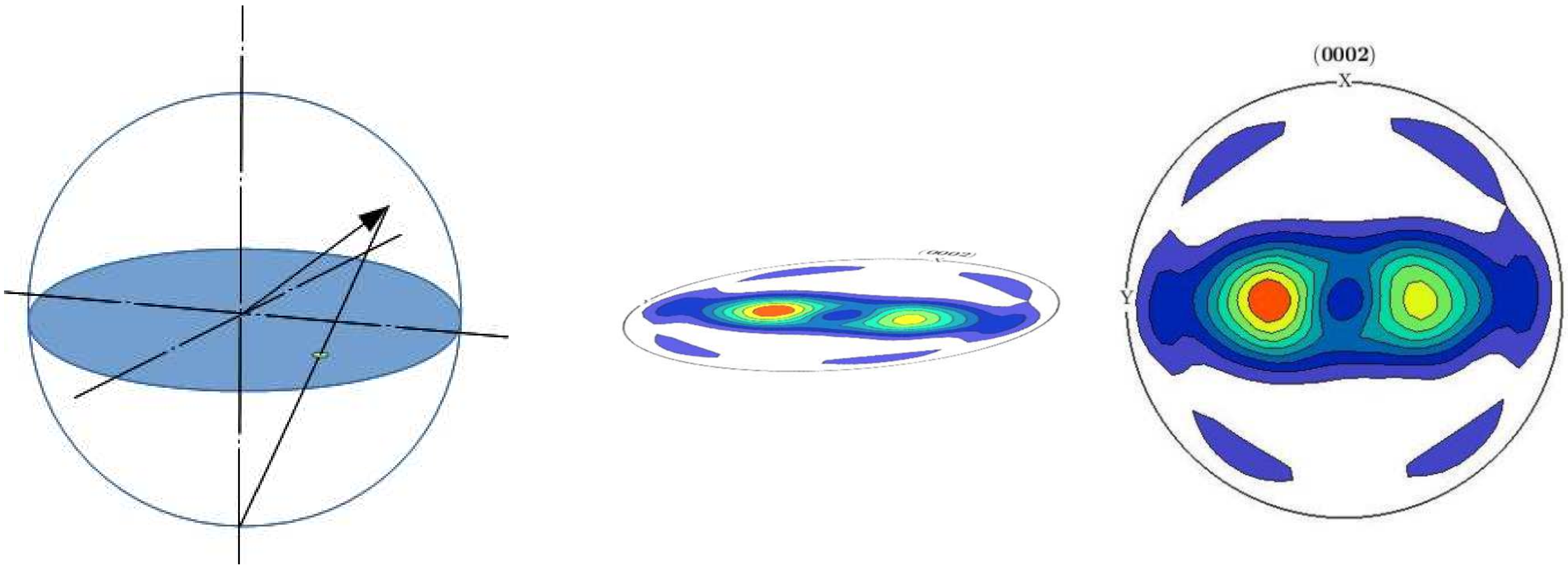
SPHERICAL PROJECTIONS



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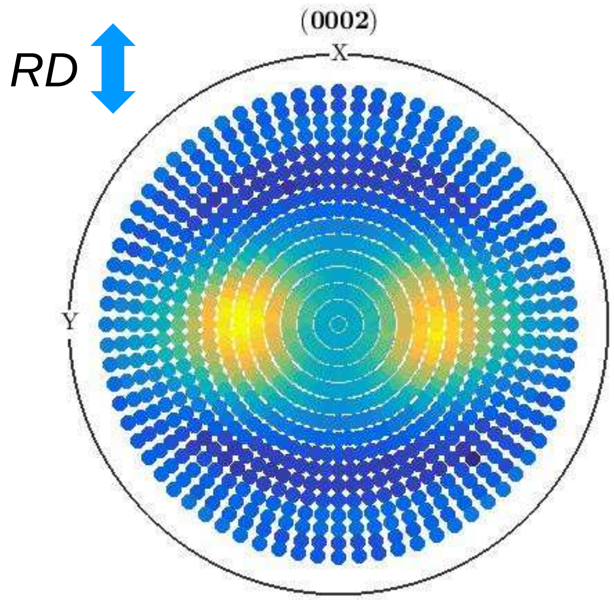
MTEX CODE SAMPLE – PLOTTING

```
% Set plotting convention
plotx2north;

% Define crystal plane(s)
hkls = { ...
        Miller(0, 0, 0, 2, cs), ...
    };

%Define the grid
r = vector3d('polar', theta, psi);

% Crystal symmetry, Zr
cs = crystalSymmetry('6/mmm', [3.225 3.225 5.225], 'X||a*', 'Y||b', 'Z||c');

% Specimen symmetry
ss = specimenSymmetry('-1');

% Create and plot a pole figure
pf = PoleFigure(hkls{1}, r, poleInts(:, i), 'CS', cs, 'SS', ss);
plot(pf({2}));
```



https://mtex-toolbox.github.io/

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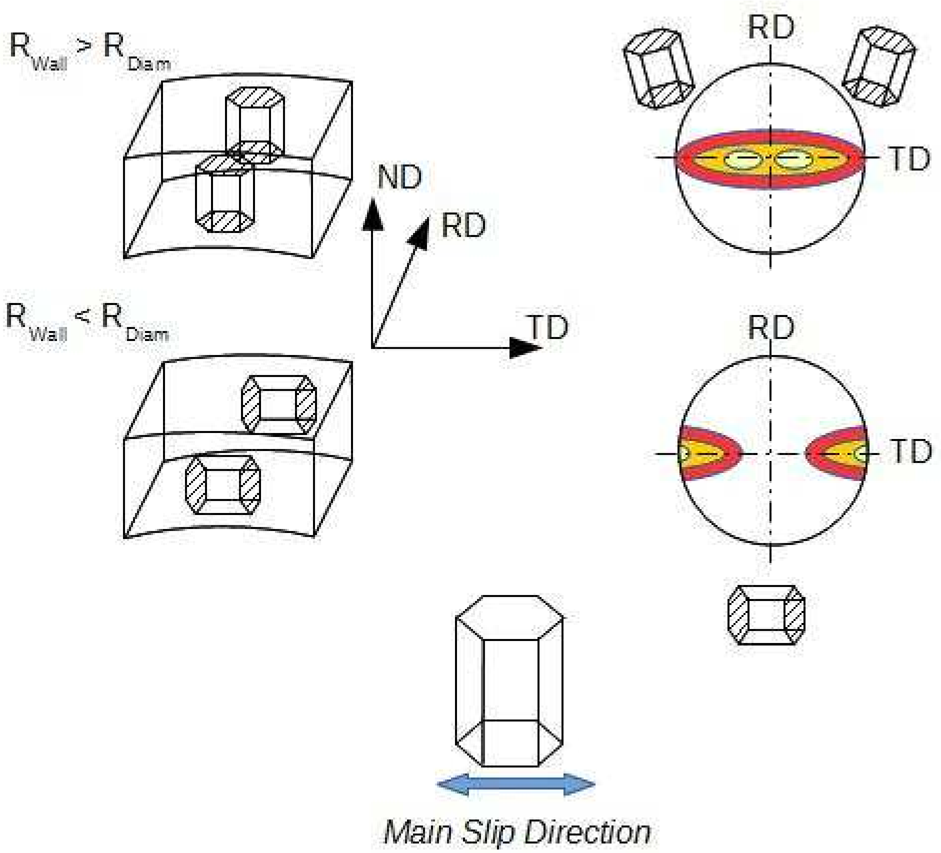
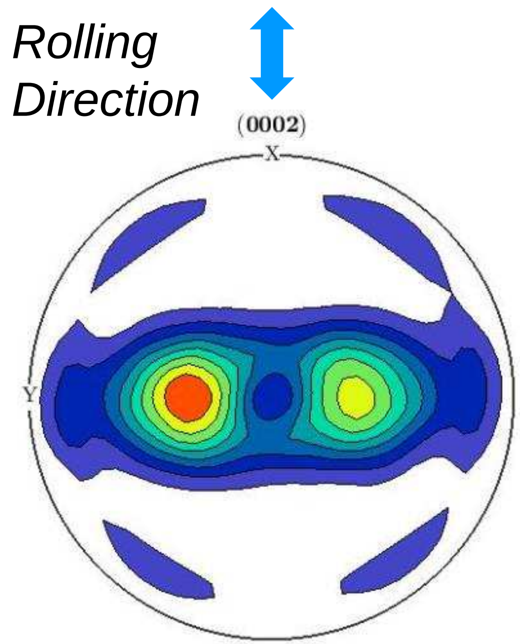
MEASUREMENT - THEORY





Calculated From Measurements via the ODF

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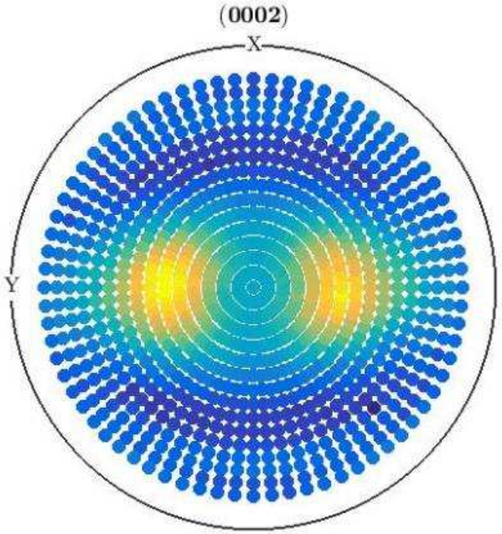
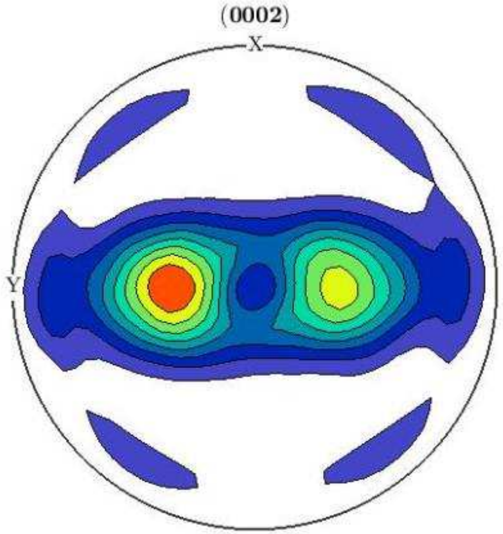
Measured Intensities

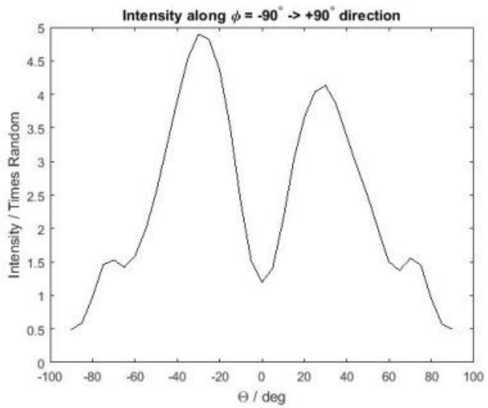


Calculated Intensities



Extracted Line Profile

Kearn's vector:
[0.52 0.33 0.16]



REPORTING

MATLAB Advantages:

- Need complete pole figure to calculate custom KPI:s
- Through *mtex-ODF*, we have access to full pole figures.
- Possibility to run the integrated intensities method in a complete evaluation script with the *Publish* function.
- To our knowledge, no commercial implementation exists of the integrated intensities approach.

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EBSD
ELECTRON BACKSCATTER DIFFRACTION

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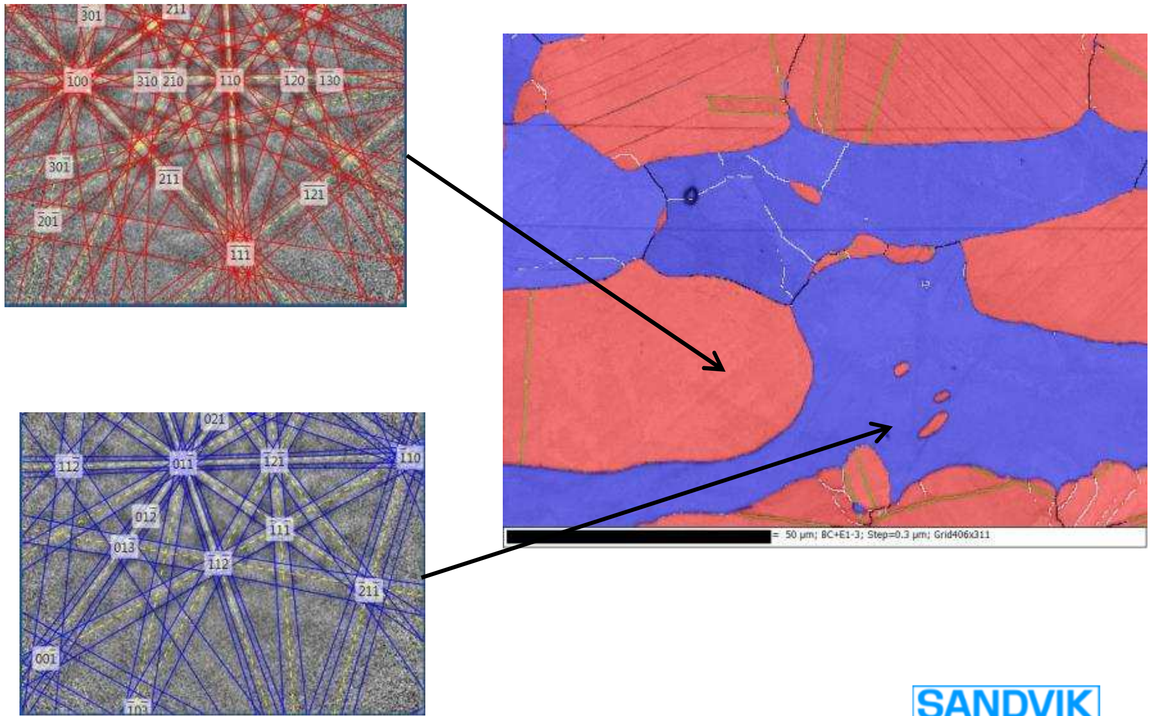
SCANNING ELECTRON MICROSCOPY - ELECTRON BACKSCATTER DIFFRACTION

Each pixel in the image is a Kikuchi pattern

Automatic indexing of each pixel

The indexing gives the orientation of the grain in each point

These orientations are then exported to MATLAB.



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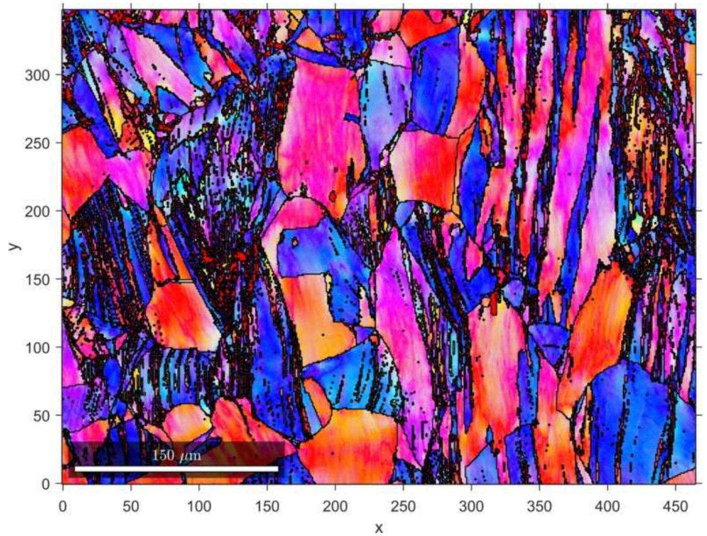
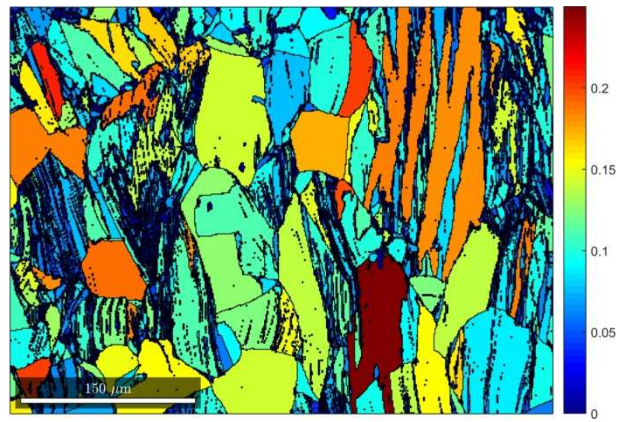
MATLAB EBSD MAPPING



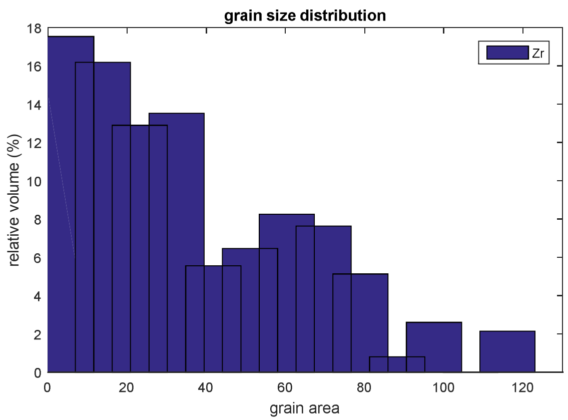
Identify grains:

Colorize pointwise by orientation

Colorize grains by orientation spread



Grain size histograms:



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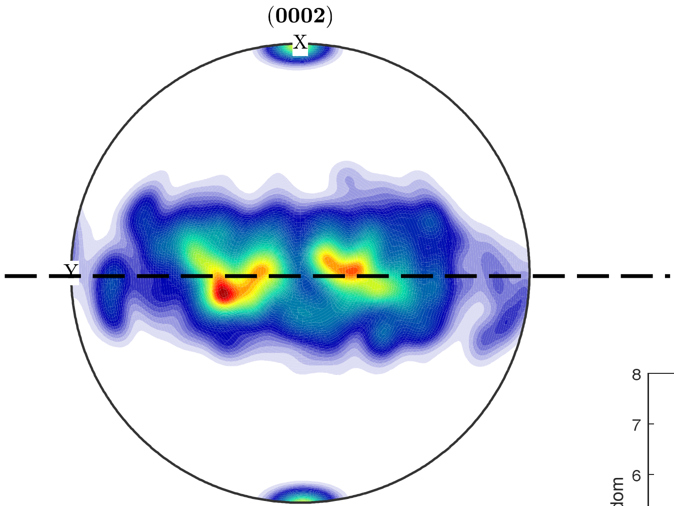
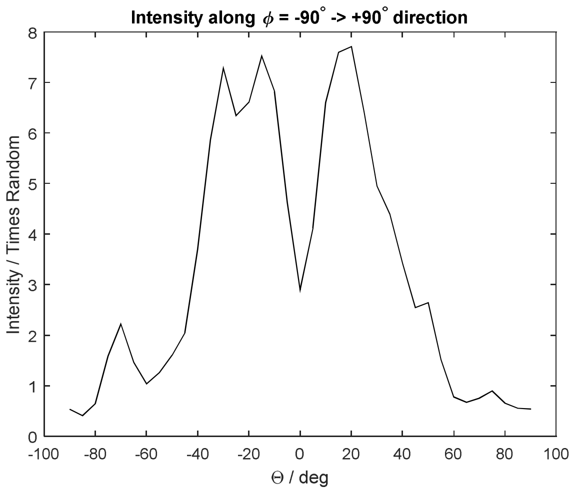
EXTRACT POLE FIGURES



EBSD compares well with XRD data,
although XRD samples a much larger area

Bonuses:
Lateral information
Possibility to extract information on individual grains



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RECRYSTALLIZED FRACTION

A compound criterion for recrystallization:

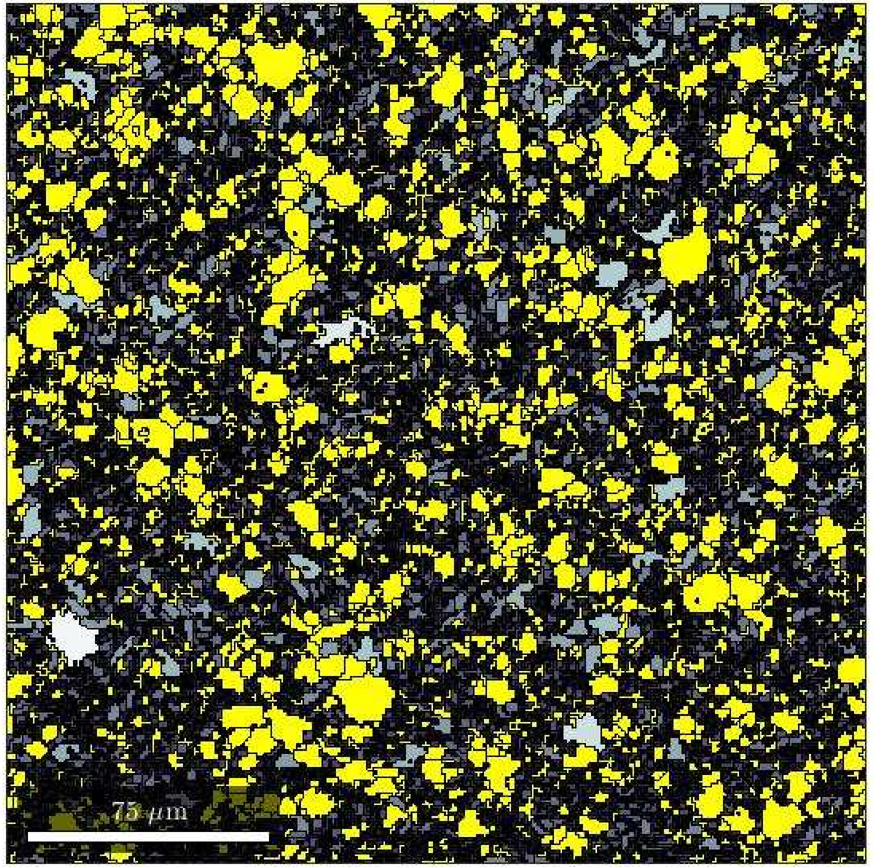
$(A > A_{crit}) \text{ AND } (GOS < GOS_{crit})$

A = Grain Area
GOS = Grain Orientation Spread

The underlying gray scale is by bone-gray by grain size.

This type of combined evaluation criteria is very easily implemented in MATLAB.

In this case, RexFrac (yellow) = 35%



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WHY MATLAB / MTEX?

- MTEX provides the Orientation Distribution Function
  - Easier (and better) than coding the spherical harmonics approximation ourselves.
- MTEX User Community within the Sandvik Group
- Compared to vendor specific software:
  - The integrated intensities method by curve fitting is not available commercially.
  - Access to many mathematical and plotting tools within the environment.

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CONCLUSIONS

- MATLAB / MTEX was used for
  - Automated curve fitting, typically >10 000 fits / measurement
  - Creating a custom script for calculating Key Performance Indicators for Zircaloy tubing.
  - Scripted EBSD evaluation with complex criteria.

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