SALZGITTER MANNESMANN FORSCHUNG

A Member of the Salzgitter Group

Application of MTEX in steel research

Dr. Marco Witte

Salzgitter, 20th February 2016

Overview

- Introduction
- Macro Texture Analysis (XRD & EBSD)
  - Elasticity tensor
  - Wave velocities and ultrasound
  - Symmetry of pole figures
- EBSD Analysis
  - Homogeneity
  - IQ Analysis
  - Prior Austenite Grains
- Summary

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Introduction - Salzgitter Mannesmann Forschung

SALZGITTER MANNESMANN FORSCHUNG
Ein Unternehmen der Salzgitter Gruppe

- **One of Europe's leading research institutes in the steel sector**
- Central **research company for steel activities in the Salzgitter Group**

Salzgitter AG

Strip steel BU	Plate/ section steel BU	Energy BU	Trading BU	Technology BU

- **Two powerful locations with close thematic ties and cooperation**
- **Direct connection to Salzgitter AG/CEO**

SZMF is responsible for ensuring the innovation capability and innovation performance in the strip steel, plate/section steel and energy business units

Introduction - SZMF: Concentrated expertise

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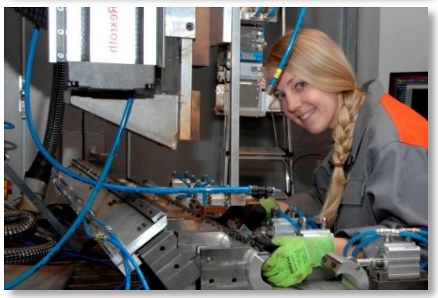
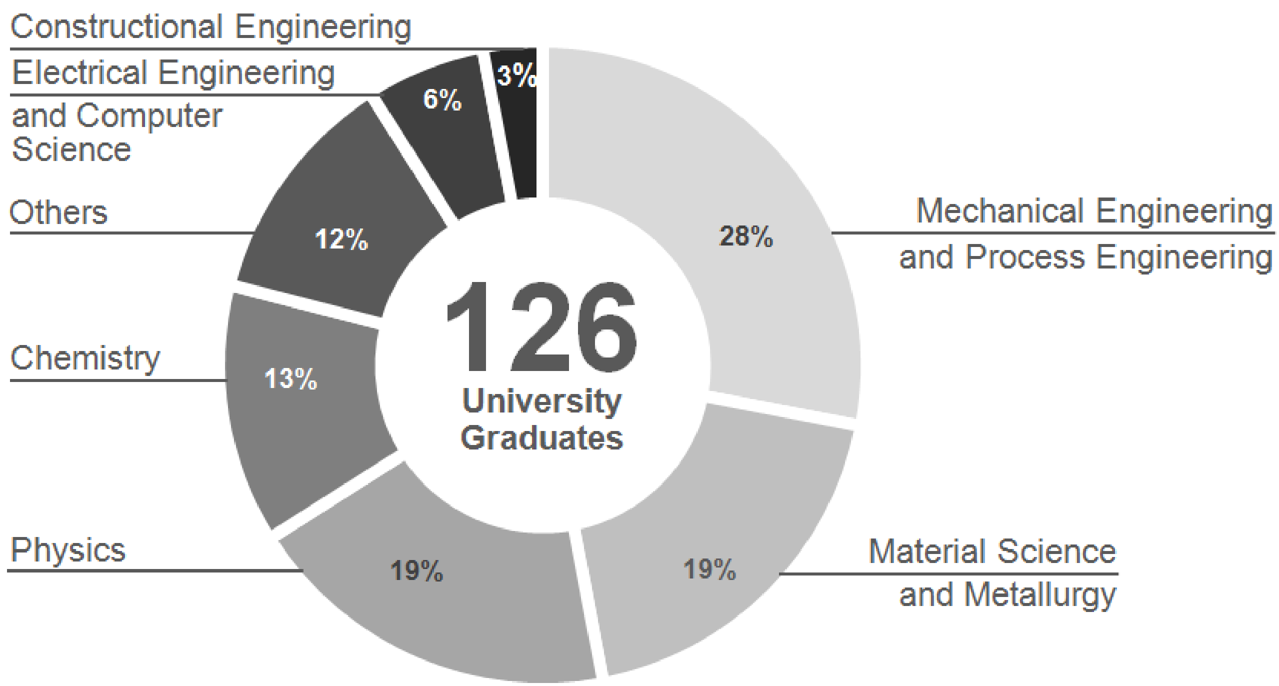
300 employees develop the future of all aspects of steel materials – around 130 members of staff in Salzgitter and 170 in Duisburg



Scientific disciplines

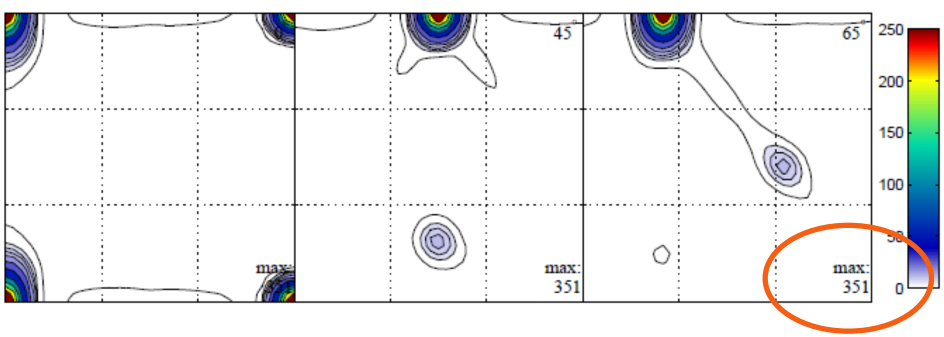


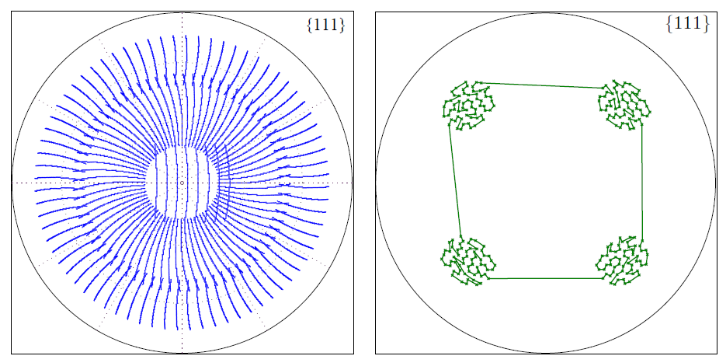
Update 2015-01

My experience with MTEX

2009-2013

- PhD thesis: "**Texture Optimization** of Ni-5at.%W for Coated Conductor Applications" at Institut für Metallkunde und Metallphysik, RWTH Aachen
- Evaluation of very **sharp textures** measured with XRD on **non regular pole figure grids.**
- Project with F. Bachmann: "Development of an Adaptive pole figure measurement technique for sharp textures"
- MTEX made working with textures and orientations fun!





My experience with MTEX

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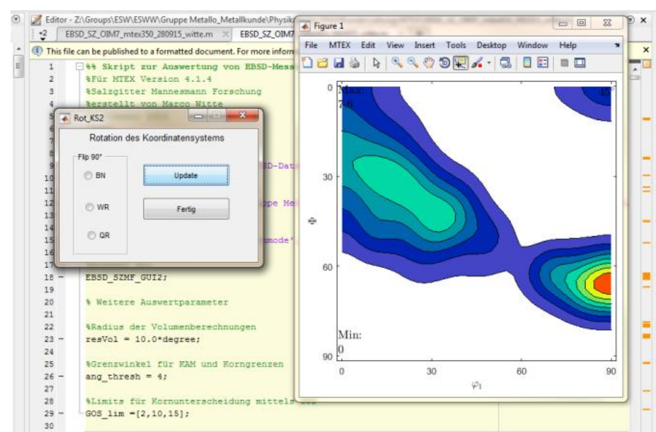
2013-now

- Evaluation of **XRD and EBSD** measurements to assist steel research and process optimization at Salzgitter Mannesmann Forschung.
- **Scripting** makes every day evaluation faster and easier.
- Approach: First evaluate “everything” and see later which information is useful.
- Automated creation of PDF reports
- MTEX still makes a lot of fun!



Overview

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Macro Texture Analysis

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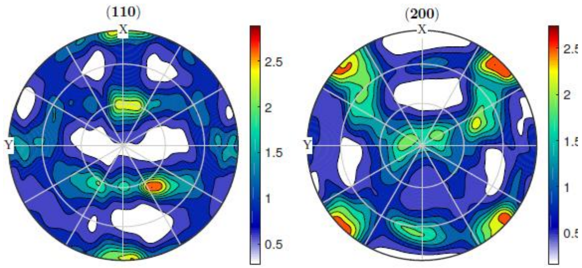
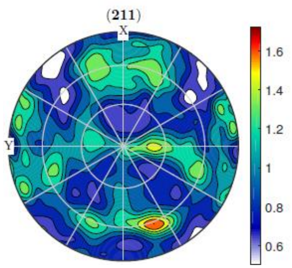
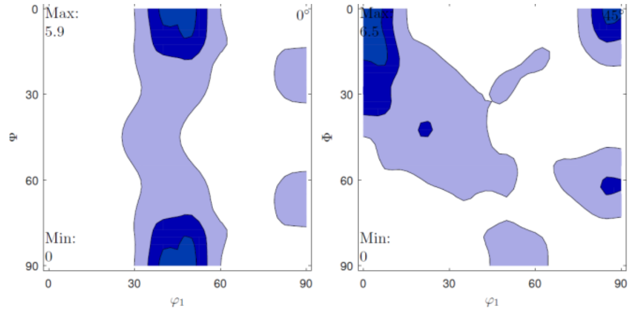
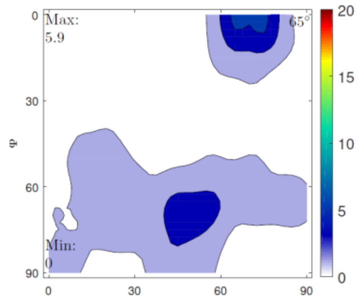
Pole figures, ODF plots, texture index, volume fractions of orientations of interest



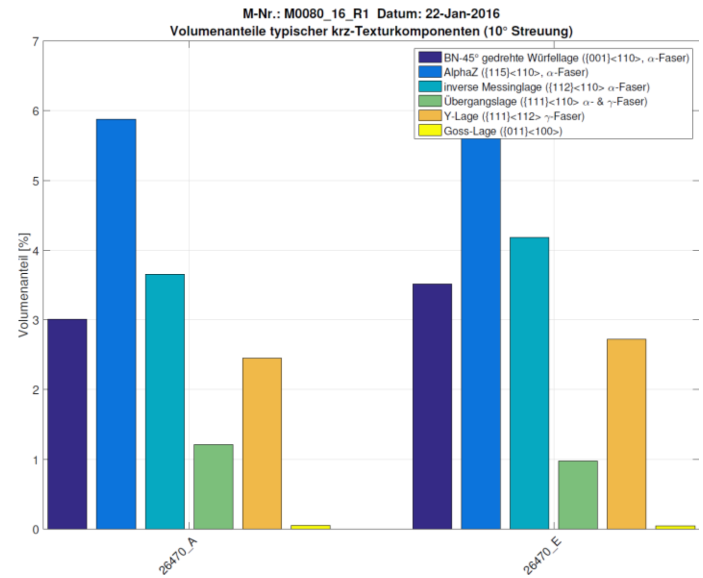


M-Nr.: M0080_16_R1
Datum: 22-Jan-2016
Probe: 26470_A
Aus der ODF zurückgerechnete, vollständige Polfiguren

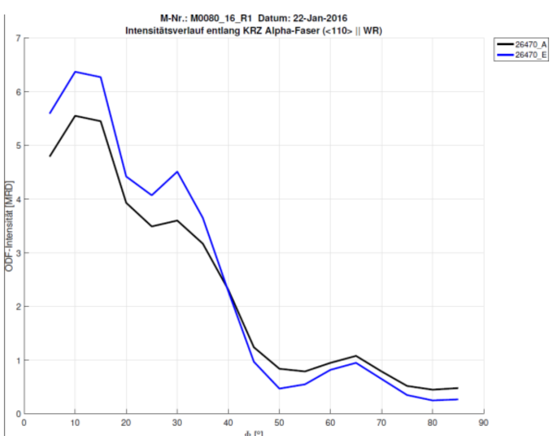


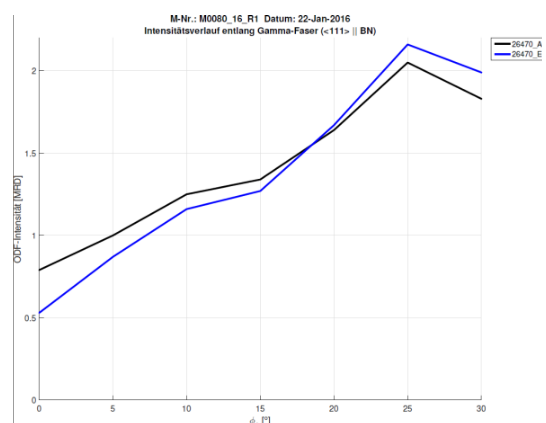


M-Nr.: M0080_16_R1
Datum: 22-Jan-2016
Probe: 26470_E
ODF-Schnitte bei phi2=0°, 45° und 65°.



Texture fibres. For steel: α-fibre ({110} || RD) & γ-fibre ({111} || ND)





Macro Texture Analysis - Elasticity

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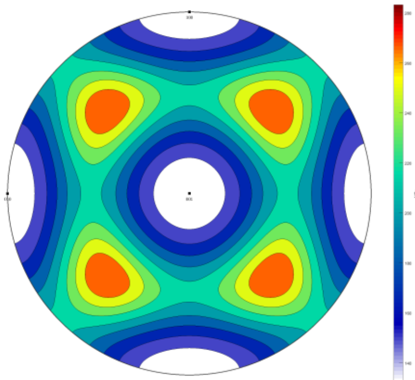
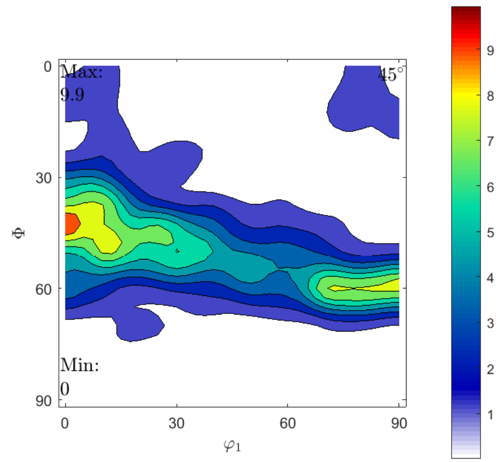
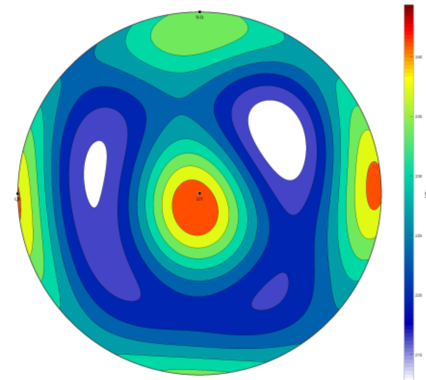


Single crystal elasticity tensor of Iron

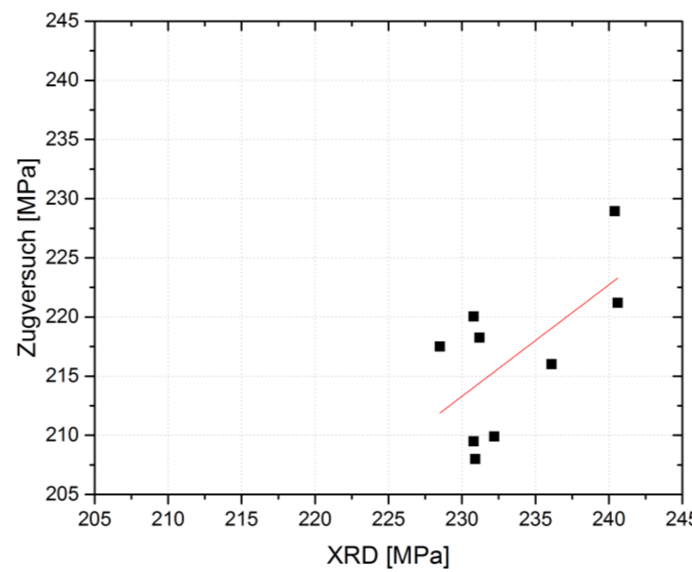


Measured ODF



Effective elasticity tensor

- Comparison with Young's modulus from tensile tests shows **good correlation**.
- Results from texture measurement too high as single crystal **tensor from literature** is for pure iron.



Macro Texture Analysis - Elastic Wave Velocities

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- Can textures be **measured with ultrasound** (online process control)?
- How does **texture composition** (γ-fibre & α-fibre) **affect wave velocities** in certain directions?
- Approach: Calculate the wave velocities for different compositions of fibre textures

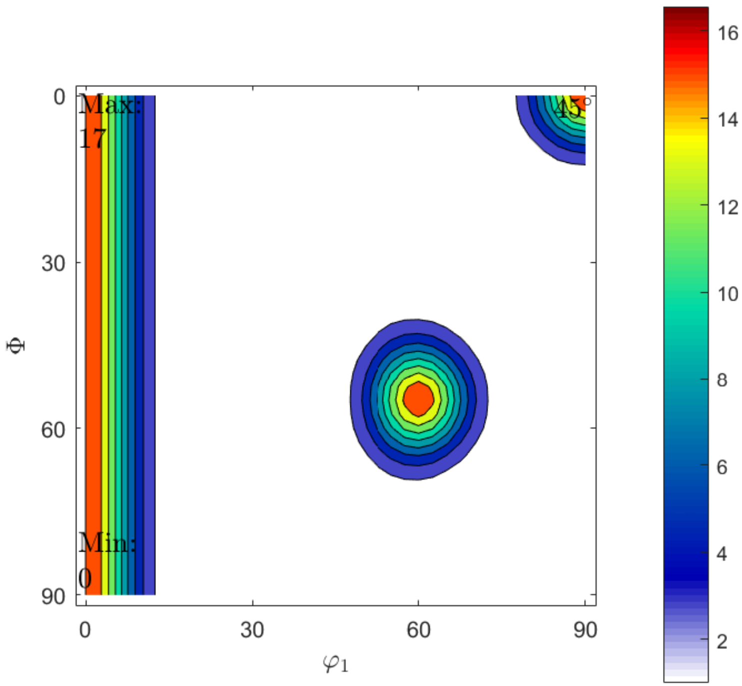
Macro Texture Analysis - Elastic Wave Velocities


```
%crystal symmetry
CS = crystalSymmetry('m-3m');
%specimen symmetry
SS = specimenSymmetry('-1');

%kernel for odfs
psi = deLaValeePoussinKernel('HALFWIDTH',7*degree);

%gamma fibre odf
odf_gamma = fibreODF(Miller(1,1,1,CS),zvector,psi);

%alpha fibre odf
odf_alpha = fibreODF(Miller(1,1,0,CS),xvector,psi);

%fibre fractions to be calculated
x = 0:0.1:1;

%to save velocities
vs1 = cell(1,length(x));

for i = 1:length(x)

%sum of fibre odfs
odf = x(i)*odf_gamma + (1-x(i))*odf_alpha;

%elasticity tensor of ferrite from MPOD
Ein_krist = [[231.4 134.7 134.7 0 0 0];...
  [134.7 231.4 134.7 0 0 0];...
[134.7 134.7 231.4 0 0 0];...
[0 0 0 116.4 0 0];...
[0 0 0 0 116.4 0];...
[0 0 0 0 0  116.4]];

T = tensor(Ein_krist, CS,'name','elastic
stiffness','unit','GPa');
```

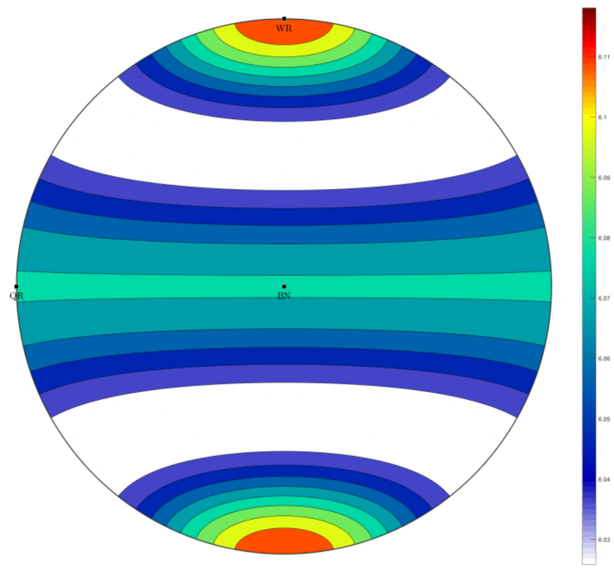
```
%weight tensor with texture
Tmean = calcTensor(odf,T);

%density in g/cm^3
rho = 7.8;

%ultrasound measurement points of s-waves
polar_angle = -35*degree;
azimuth_angle = 0:15:90;
v_shear =
vector3d('polar',polar_angle,azimuth_angle*degree);

%get s-wave velocities
[vp{i},vs1{i}] = velocity(Tmean,v_shear,rho);

%plot s-wave tensor
figure
plot(Tmean,'PlotType','velocity','vs1','density',rho,'compl
ete','upper')

ax = colorbar;
xlabel(ax,'km/s')
saveFigure(['Gamma_' num2str(x(i),2)
'_Elast_Welle_vs1.png'])
close

end
```

Texture Analysis - Elastic Wave Velocities

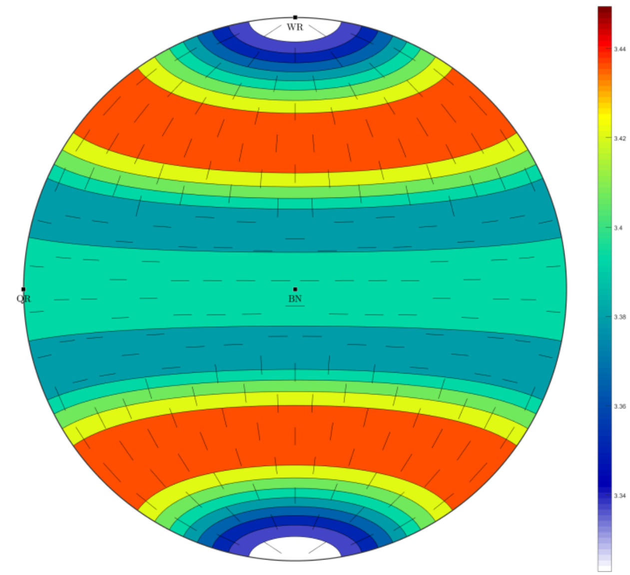
0% γ -fibre & 100 % α -fibre



ODF



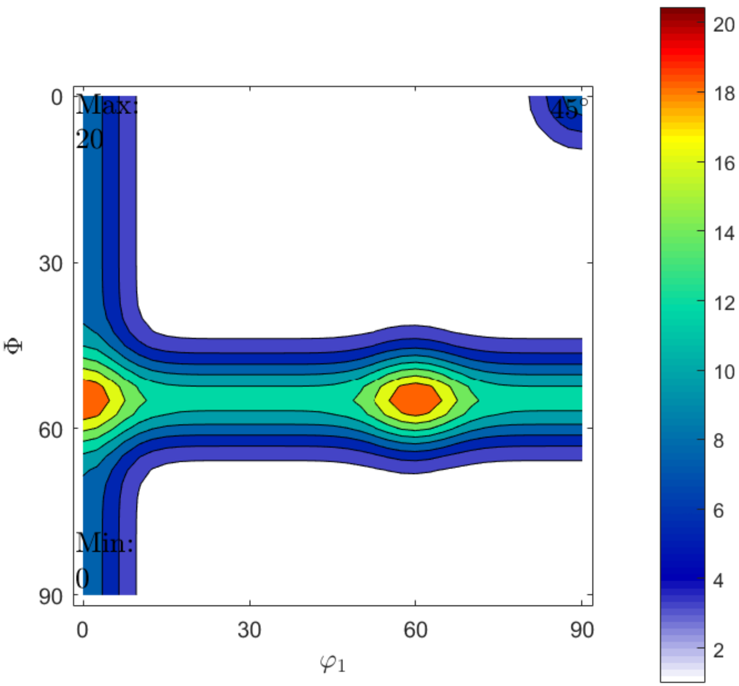
p-wave velocity



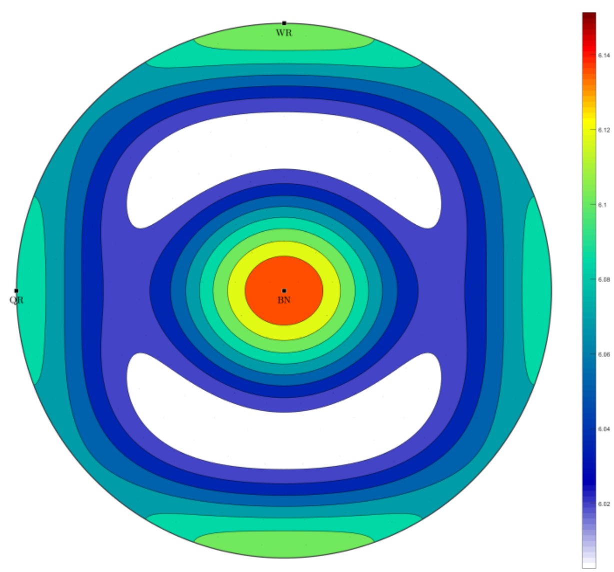
s-wave velocity

Macro Texture Analysis - Elastic Wave Velocities

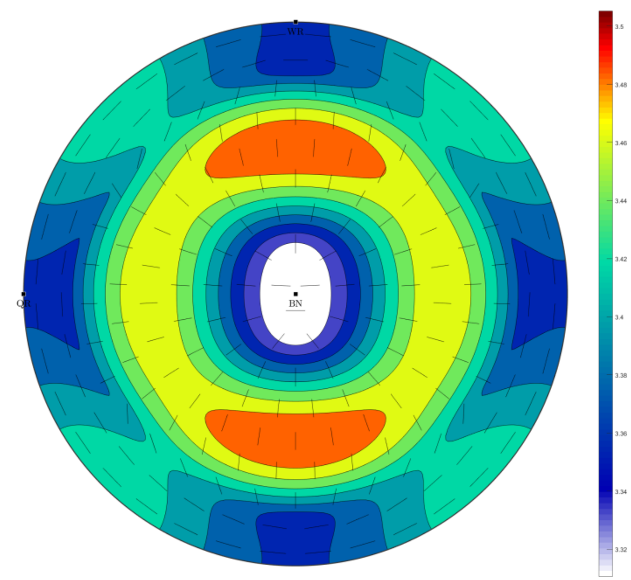
50% γ -fibre & 50 % α -fibre



ODF



p-wave velocity



s-wave velocity

Macro Texture Analysis - Elastic Wave Velocities

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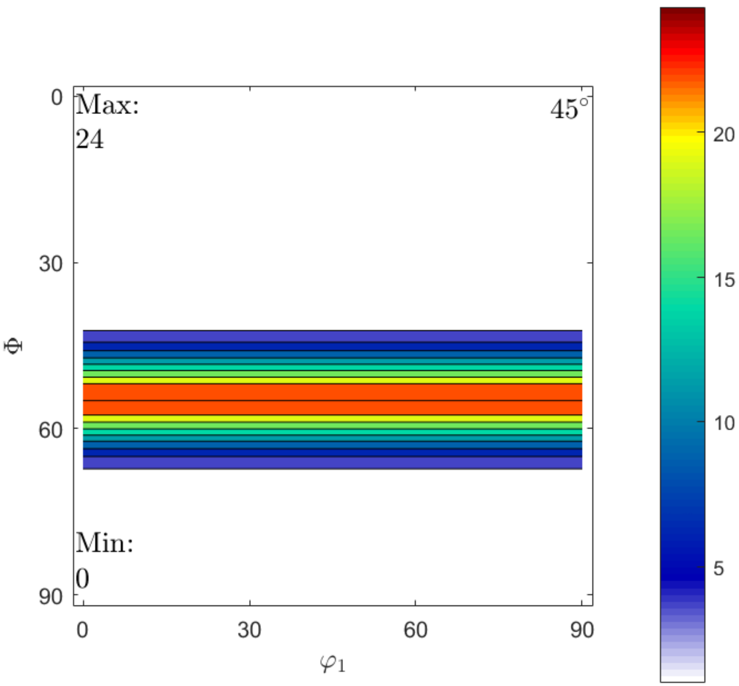
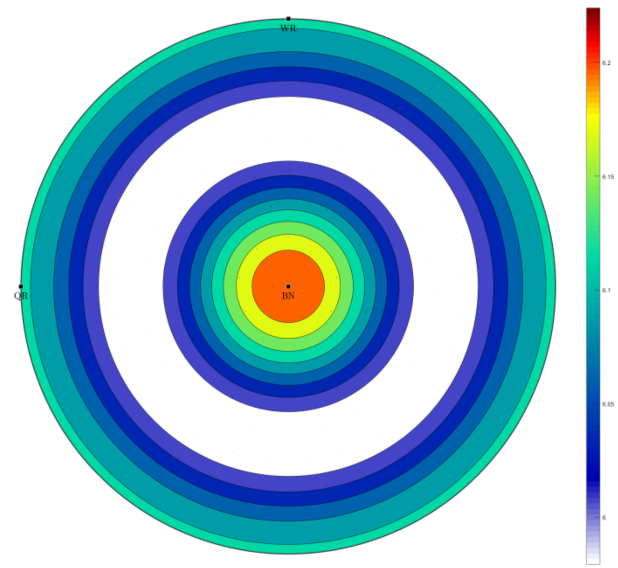
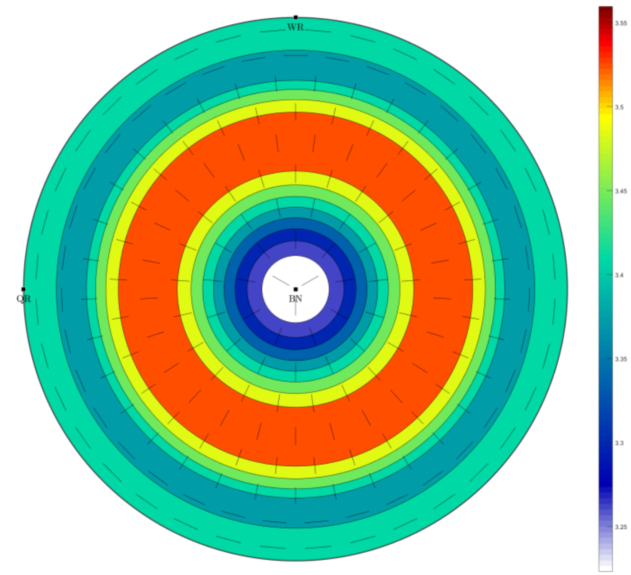
100% γ -fibre & 0 % α -fibre



ODF



p-wave velocity



s-wave velocity

Macro Texture Analysis – Velocity of Sound – p-Waves

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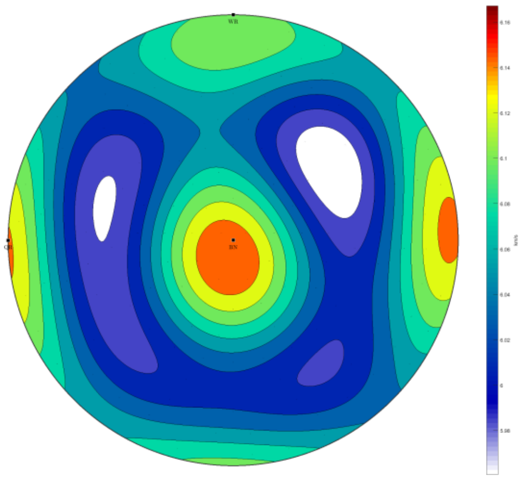
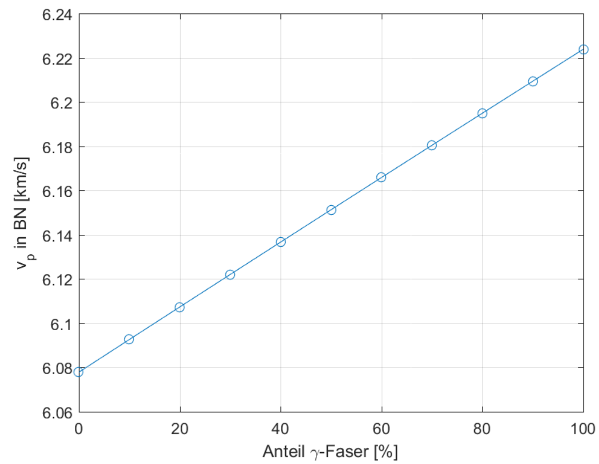
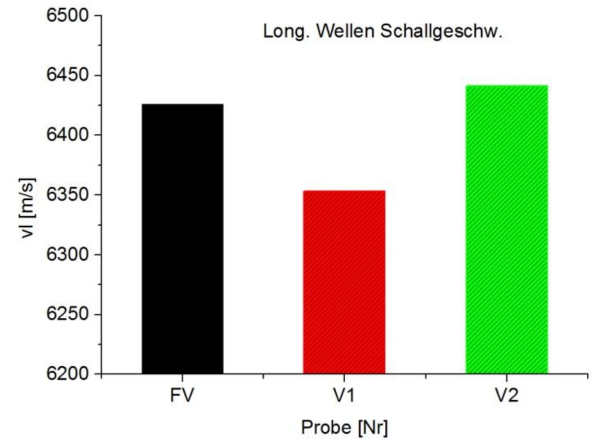


Distribution of pressure wave velocities v_P calculated from elasticity tensor



Calculated distribution of v_P with increasing amount of γ–fibre (rest α-fibre)



Measured distribution of v_P.
V2: strong γ–fibre
V1: more α-fibre
FV: α-fibre & large grains

- Difference Δv_P = 100 m/s can be expected (measurement error ~20m/s)
- Trend in measurement results not conclusive (errors?)
- More measurements are necessary

M2151_15, IF_Pr915_C6_neu_rotiert

Texture Analysis – Velocity of Sound – s-Waves

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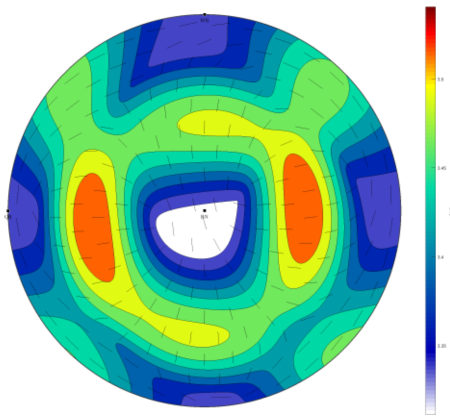
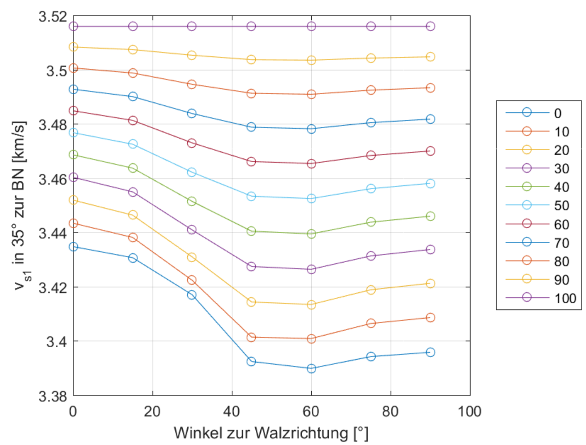
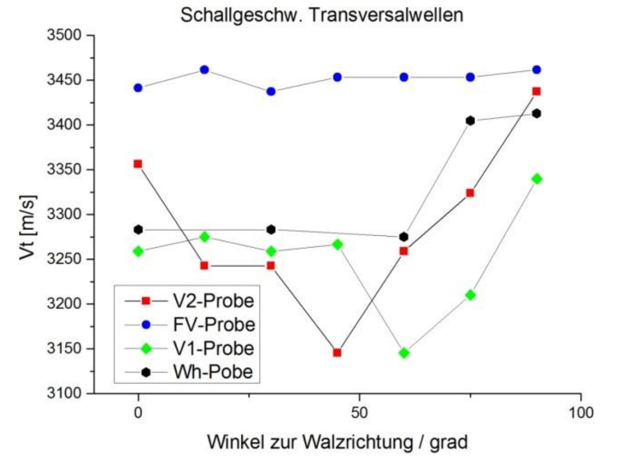


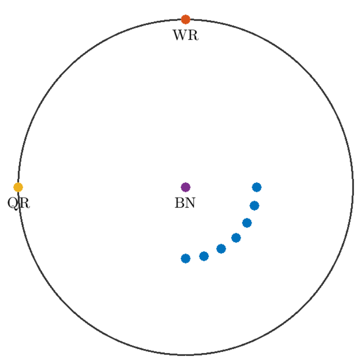
Distribution of shear wave velocities calculated from elasticity tensor



Calculated distribution of v_{S1} with increasing amount of γ–fibre (rest α-fibre)



Measured distribution of v_{S1}.
V2: strong γ–fibre
V1: more α-fibre
FV: α-fibre & large grains

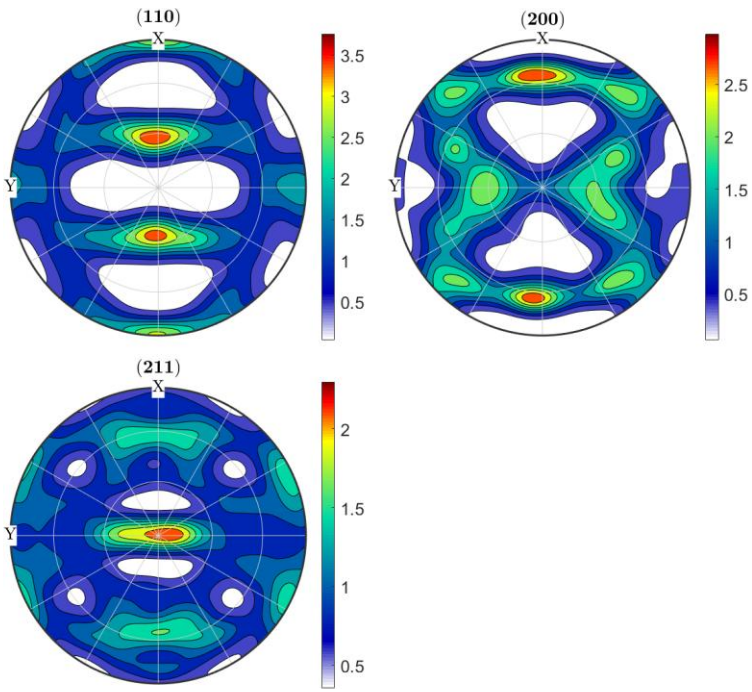


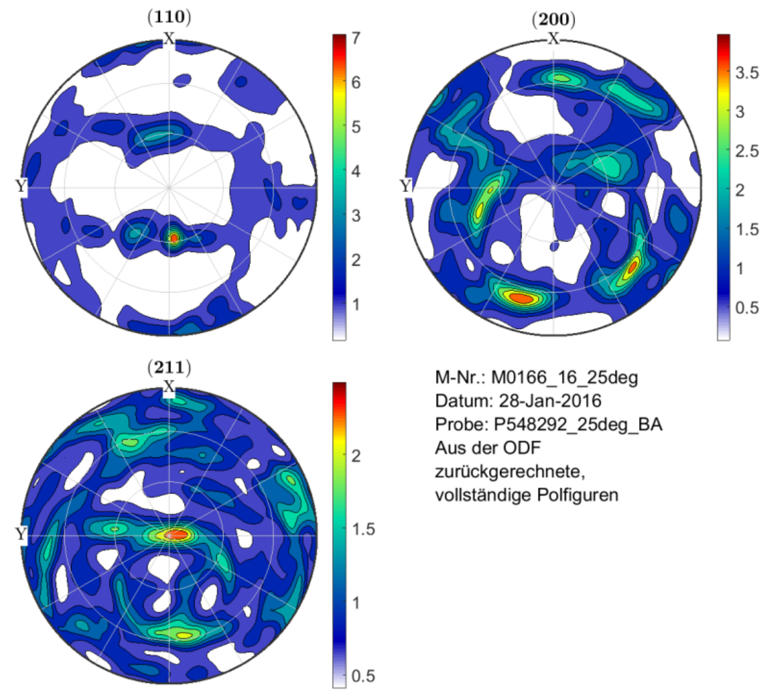
Measurement positions with ultrasound

- Ultrasound measurement difficult due to multiple reflections and sample surfaces
- Only small differences at measurement positions
- Measurement directions not optimal
- Nevertheless comparison with measured velocities possible

M2151_15, IF_Pr915_C6_neu_rotiert

Texture Analysis – Symmetry of pole figures





M-Nr.: M0166_16_25deg
Datum: 28-Jan-2016
Probe: P548292_25deg_BA
Aus der ODF
zurückgerechnete,
vollständige Polfiguren

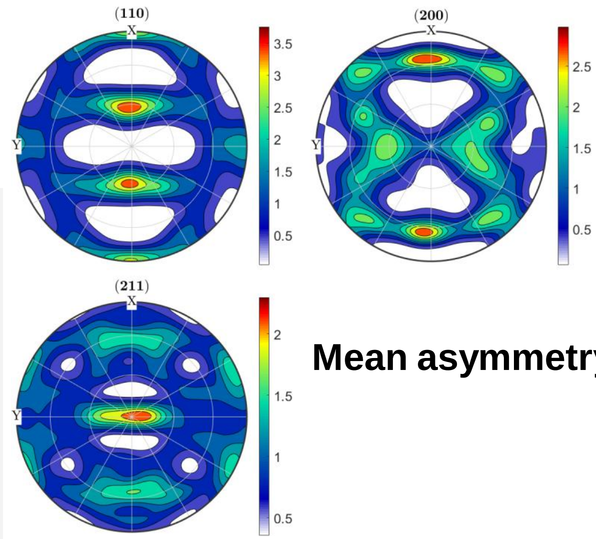
- **Symmetry differences** of pole figures can tell you about:
  - Inhomogeneous deformation, measurement statistic, sample preparation,...
- Orthorhombic sample symmetry should never be imposed! (except for ODF plots...)

M0166_16 & M1990_15, P54829_BA

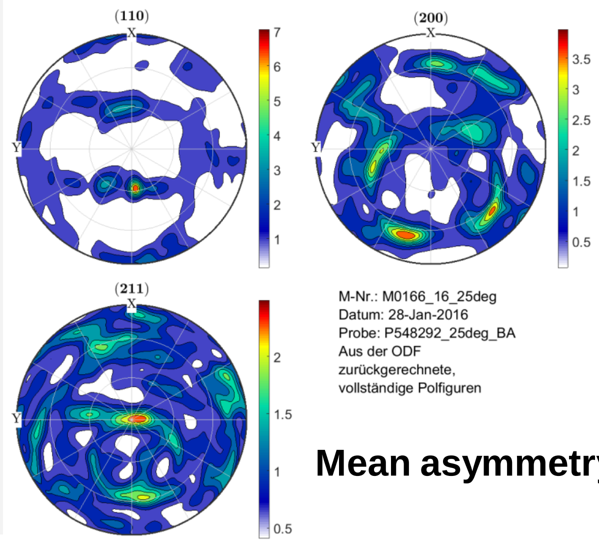
Texture Analysis – Symmetry of pole figures

Approach: Flip pole figures about mirror axes and calculate difference with unflipped pole figure.

```
%Calculate pole figure from ODF
pf_calc = calcPoleFigure(odf,h,'resolution',1*degree,'complete');

%Flip upside-down
rotation_ud = rotation('axis',zvector,'angle',180*degree);

%Flip left-right
rotation_lr = rotation('axis',yvector,'angle',180*degree);

%Do Flip
odf_ud = rotate(odf,rotation_ud);

odf_lr = rotate(odf,rotation_lr);

%flipped pole figures
pf_ud = calcPoleFigure(odf_ud,h,'resolution',1*degree,'complete');

pf_lr = calcPoleFigure(odf_lr,h,'resolution',1*degree,'complete');

%Calculate error between original and flipped pole figures
error_ud = mean(calcError(pf_calc,pf_ud));

error_lr = mean(calcError(pf_calc,pf_lr));
```



Mean asymmetry = 0.074



M-Nr.: M0166_16_25deg
Datum: 28-Jan-2016
Probe: P548292_25deg_BA
Aus der ODF
zurückgerechnete,
vollständige Polfiguren

Mean asymmetry = 0.214

M0166_16 & M1990_15, P54829_BA

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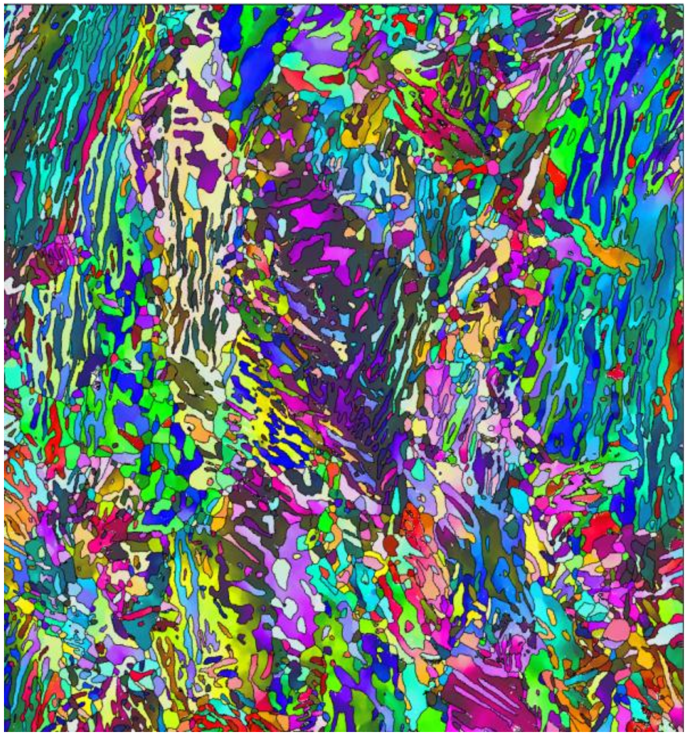
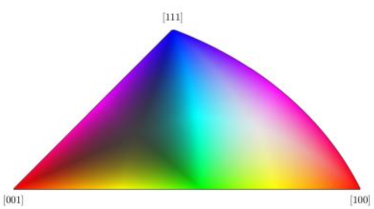
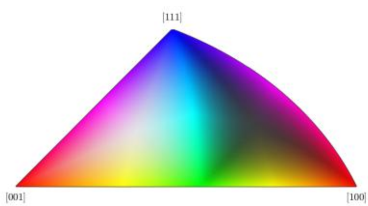
EBSD Analysis

Orientation map





BCC



FCC

Phase map

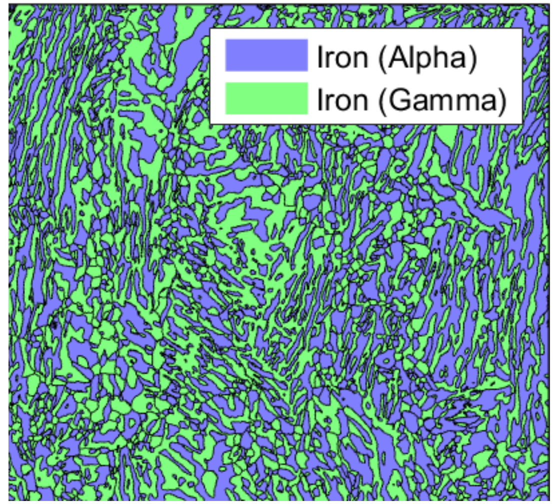
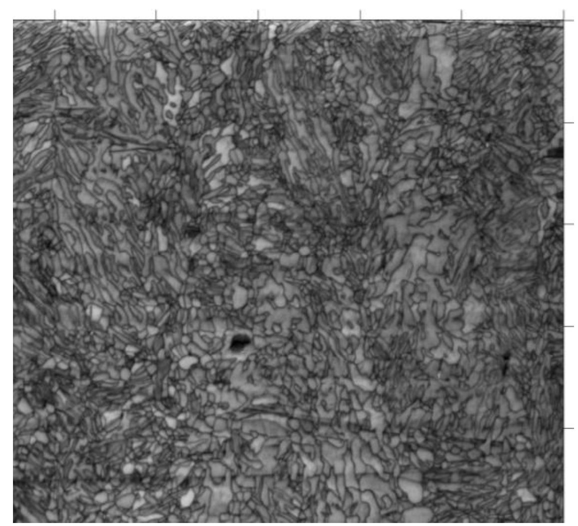


Image Quality map



EBSD Analysis

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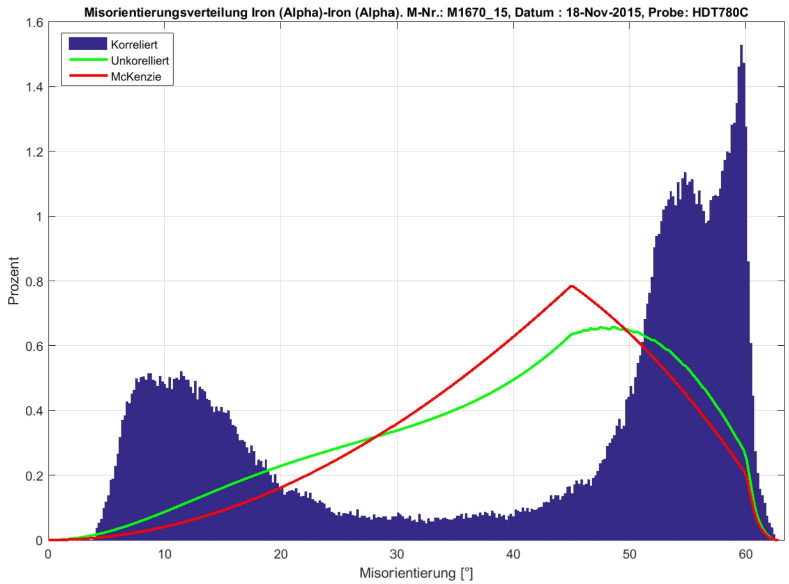
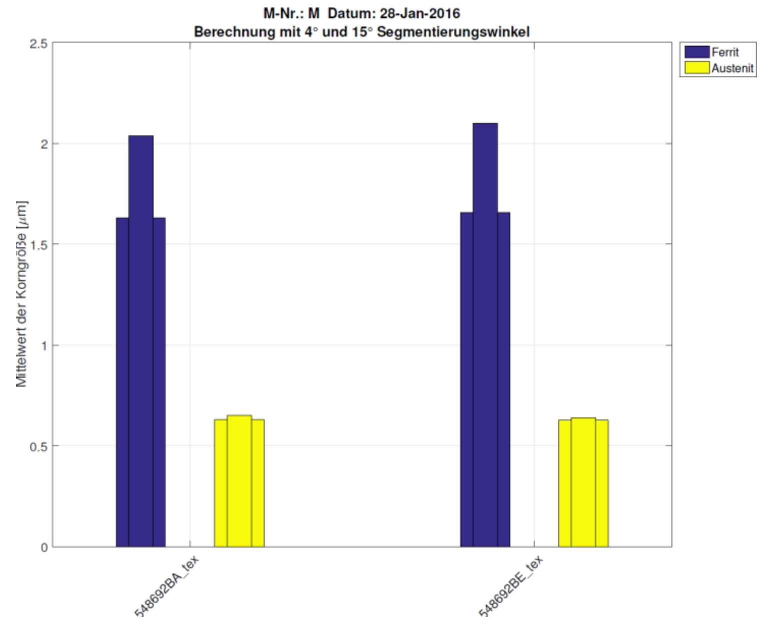
Misorientation angle distribution



- Typical distribution of bainitic microstructure
- Useful for **phase discrimination** ferrite-bainite-martensite
- Uncorrelated distribution (green) and random distribution (red)

Grain size

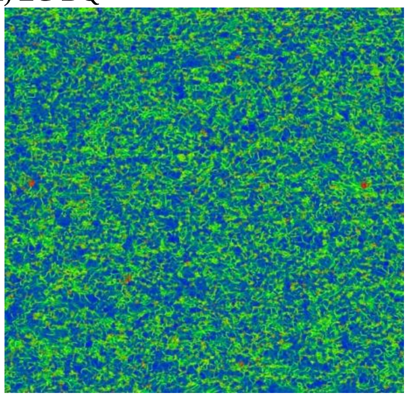


- For every phase
- For 4° and 15° segmentation angle (LAGB – HAGB)

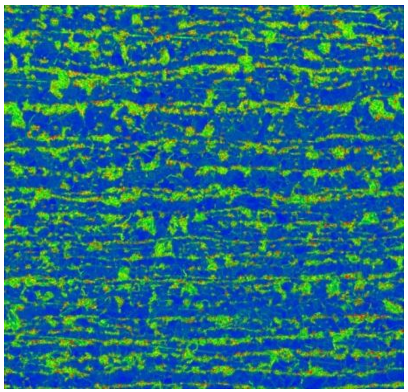
EBSD Analysis – Homogeniety

Kernel average misorientation maps of multi phase steels produced with different cooling conditions

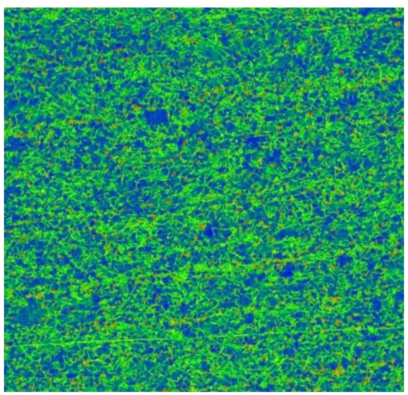
a) LC-DQ



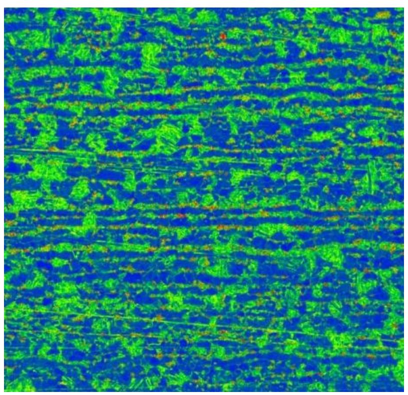
b) HC-DQ



c) LC-IQ

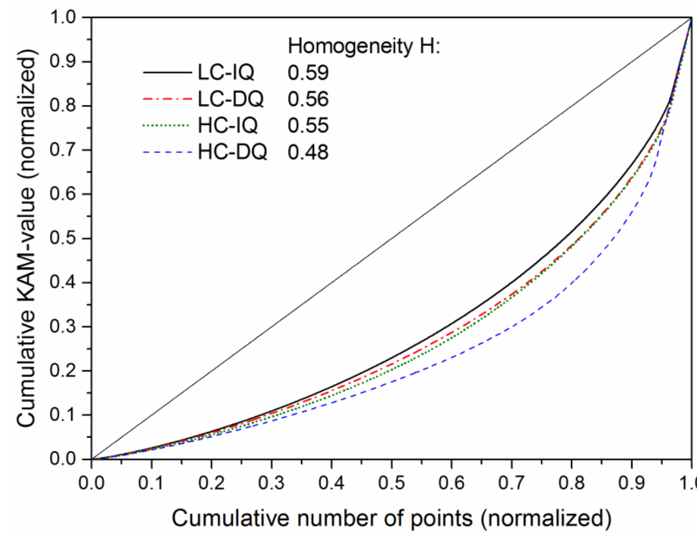


d) HC-IQ



Lorenz curves of KAM distribution

[Rossi et. al., 2014 *Pract. Met.*, *51*(3), 180-199]



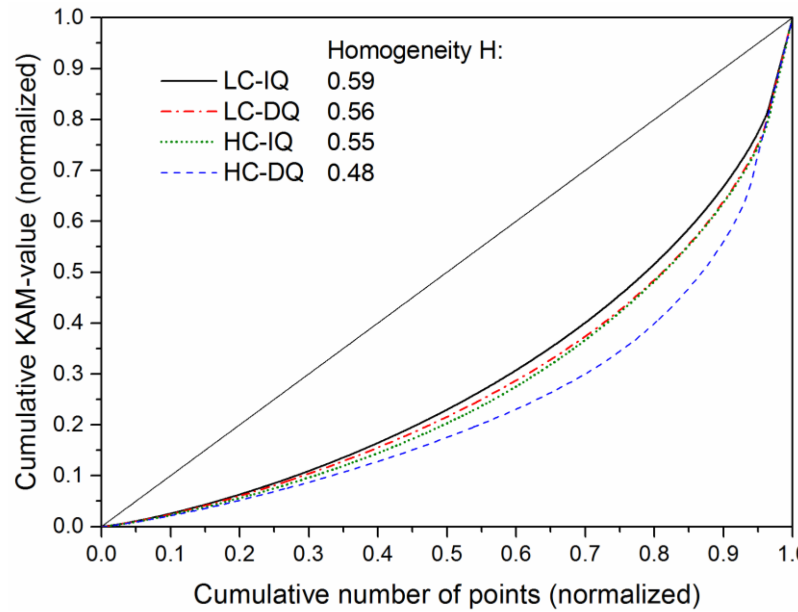
- **Homogeneous distribution** of dislocations (KAM) is important for **crack resistance**.
- Can be characterized by **one value H**, the area under the Lorenz curve.
- Statistical error ΔH ≈ 0.002

EBSD Analysis – Homogeniety

Calculation

```
%KAM values
KAM_1 = KAM(ebsd,'threshold',4*degree,'order',1)./degree;

%calculate cumulative KAM values
cum_sum_kam = cumsum(sort(snip(KAM_1,nan)))…
/sum(snip(KAM_1,nan));

%calculate number of cumulative KAM values
cum_x = (1:numel(cum_sum_kam))./numel(cum_sum_kam);

%calculate homogeniety
homo_kam = 2*trapz(cum_x, cum_sum_kam);
```

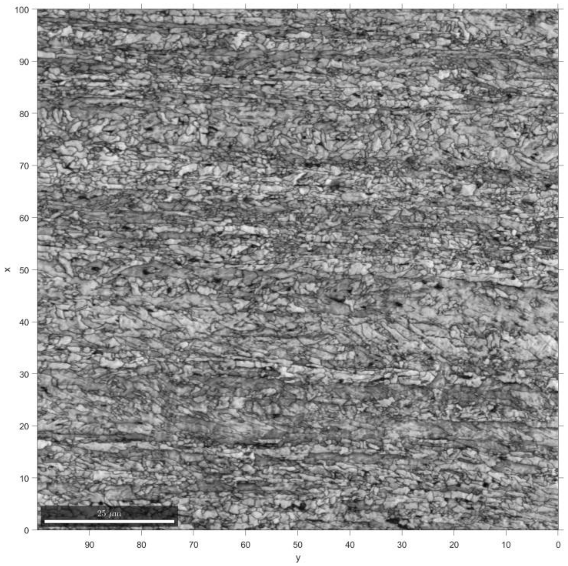
Lorenz curves of KAM distribution



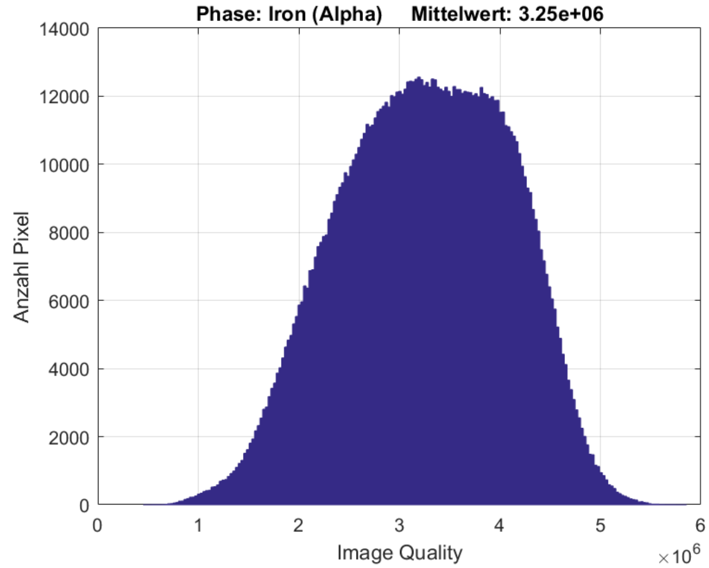
- **Quantitative** description of Homogeneity
- Can easily be applied **to all kind of distributions**, e.g. grain size, particles,...
- Combined homogeneities can be used, e.g. $H = H_{size} * H_{shape}$

EBSD Analysis – IQ distribution

IQ map



IQ histogram

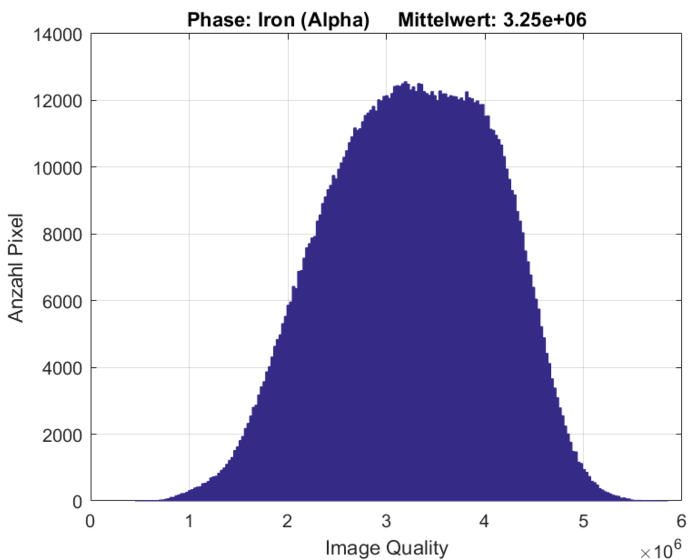


- Image Quality (IQ) describes the contrast of the Kikuchi-Patterns
- It is reduced by lattice distortions like dislocations, grain boundaries, micro strains, ...
- IQ histogram of bcc iron may be the sum of two distributions. One with high IQ values (low distortion) and one with low IQ values (high distortion).
- Phase quantification Ferrit – Bainite – (Martensite)

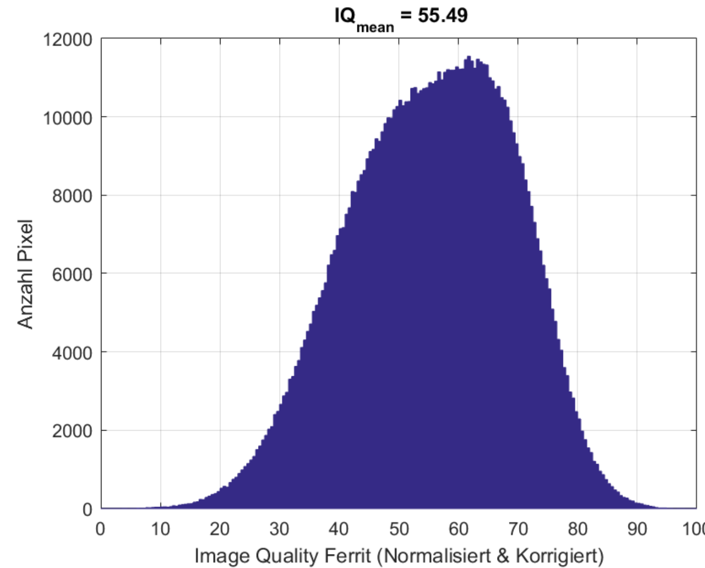
EBSD Analysis – IQ distribution

Remove points at grain boundaries

```
%IDs of ebsd measurements at grain boundaries
ids = grains.boundary.ebsdId;
ids = unique(ids(ids>0));

%vector with boundary points
isGrainBoundaryEBSD =
sparse(ids,1,true,ebsd.size(1),ebsd.size(2));

%exclude points at grain boundaries
ebsd_iq = ebsd(~isGrainBoundaryEBSD);


%normalize IQ
iq_cor = ebsd.iq;
iq_cor_n = 100 .* (iq_cor -min(iq_cor))/(max(iq_cor)-
min(iq_cor));
```



Grain boundary points removed



EBSD Analysis – IQ distribution

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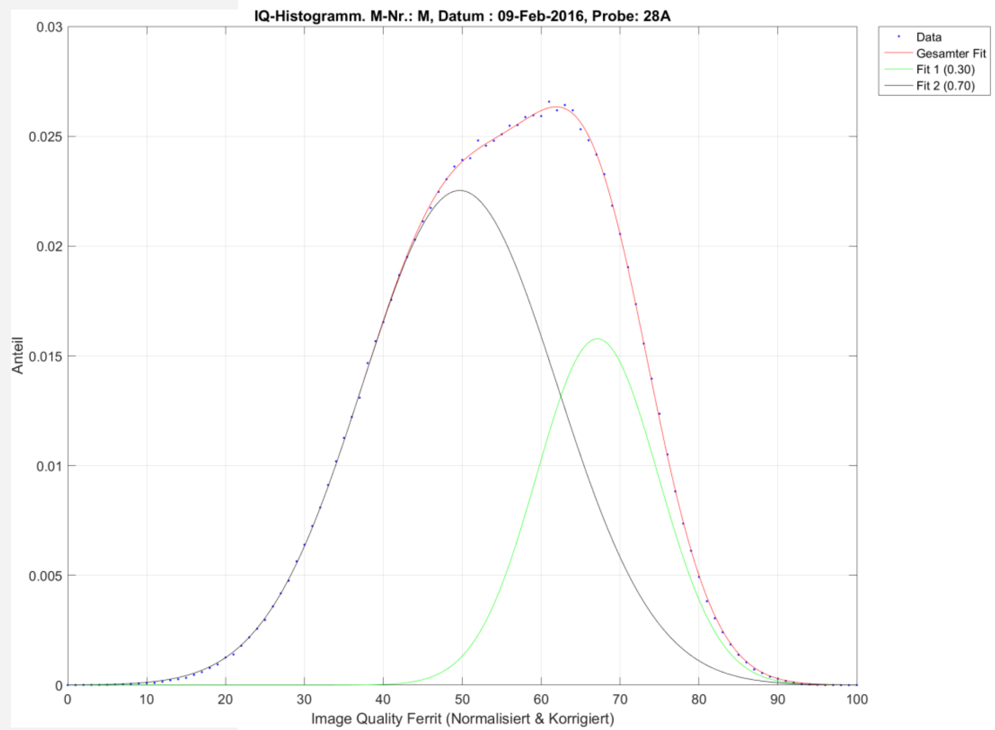
Fit with two Gaussian functions (Curve Fitting Toolbox)

```
%define binning
bins = 0:1:100;

%histogram counts
n = histc(iq_cor_n,bins);

n = n/sum(n);

load enso;
%two Gauss peaks, start conditions
options = fitoptions('gauss2','StartPoint',[0.1 65 10 0.1 40 10]);

%fit
f = fit(bins', n,'gauss2',options);

%integrate results
fun_1 = @(x) f.a1.*exp(-((x-f.b1)./f.c1).^2);
fun_2 = @(x) f.a2.*exp(-((x-f.b2)./f.c2).^2);

fit_area_1 = integral(fun_1,0,100);
fit_area_2 = integral(fun_2,0,100);

%plot result
figure
plot(f,0:1:100,n), hold on
fplot(fun_1,[0 100],'color','green')
fplot(fun_2,[0 100],'color','black')
xlabel('Image Quality Ferrit (Normalisiert & Korrigiert)','FontSize', 14)
ylabel('Anteil','FontSize', 14)
legend('Data','Gesamter Fit',sprintf('Fit 1 (%.2f)',fit_area_1),sprintf('Fit 2
(%.2f)',fit_area_2),'Location','NorthEastOutside')
grid on
set(gca,'FontSize', 14)
saveFigure('IQ_fit_histogramm.png')
hold off
```

IQ histogram



EBSD Analysis – IQ distribution

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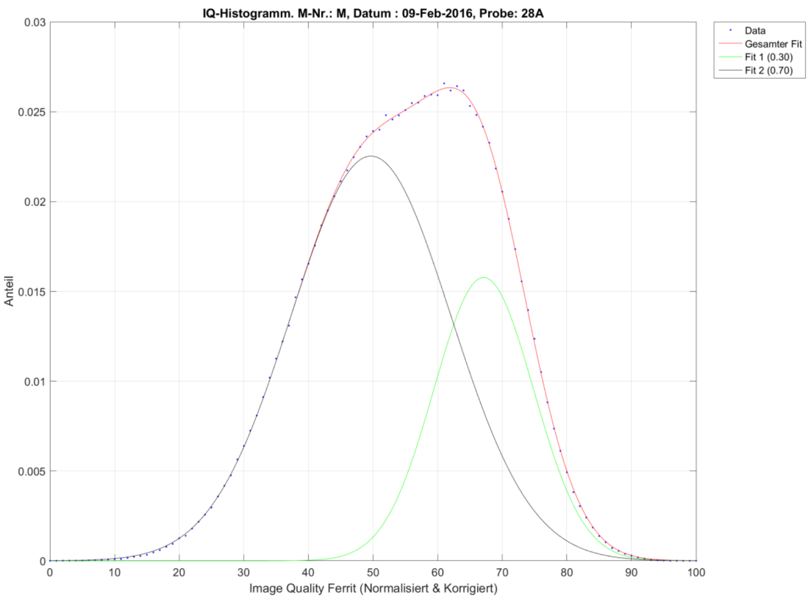
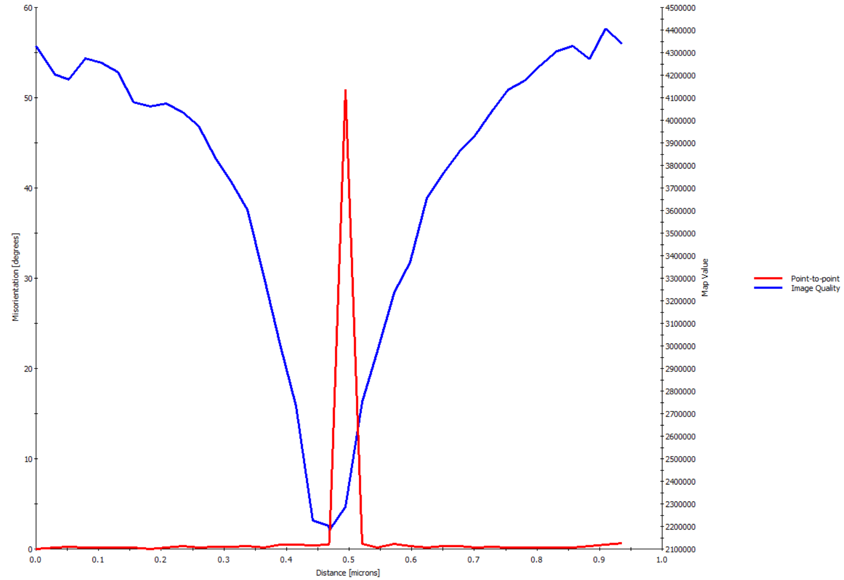
IQ histogram



Drop of IQ at grain boundary

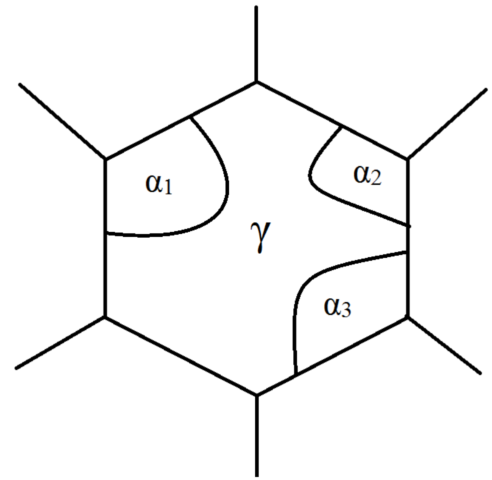


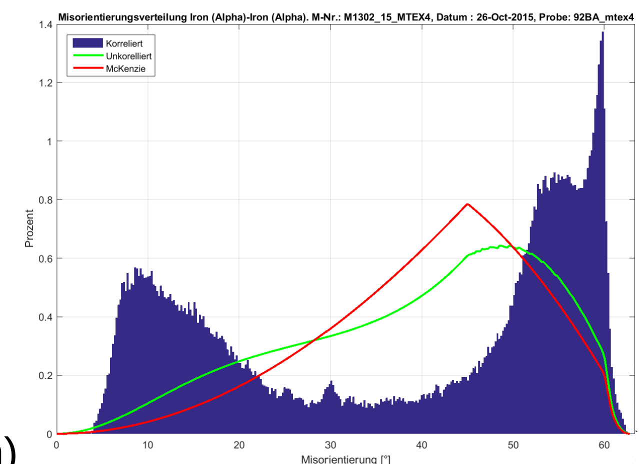
- Result: 70% Bainite, 30% Ferrite
- Reminder: IQ values are strongly influenced by sample preparation and measurement conditions
- **Effect of grain boundaries** to reduce IQ **extends ~300nm**, thus grain boundary efffect may not be completely eliminated.
- Results are hence somewhat biased and give no spatial information

EBSD Analysis – Prior Austenite Grains

- The **size** of the final α–Fe microstructure is **determined by the prior austenite grain size**.
- The prior austenite grain size can be directly influenced by rolling temperature, amount of deformation during hot rolling and microchemistry.
- But it is very **difficult to measure** this austenite grain size in-situ.
- After hot rolling the **phase transformation** austenite (γ-Fe) to α–Fe occurs with certain **orientation relationships** between parent and daughter grains.
  - Kurdimov-Sachs (K.-S.): 90° rotation about <1,1,2>
  - Nishiyama-Wassermann (N.-W.): 95.3° rotation about <3,6,2>
  - The observed orientation relationships are somewhere in between these two.
  - Not all symmetrical equivalent relationships occur (variant selection)

→ Determination of the fcc γ-Fe **from EBSD measurements** of the bcc α–Fe microstructure



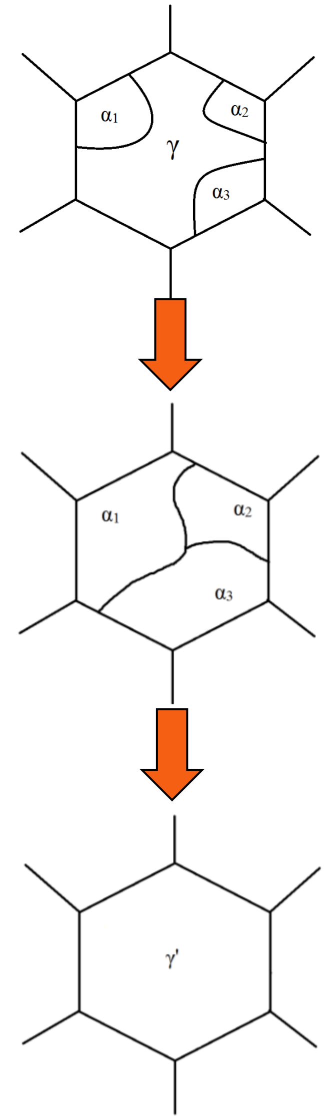


EBSD Analysis – Prior Austenite Grains

- Approach: **Delete all grain boundaries** that deviate less than 5° from K.-S. and N.-W.

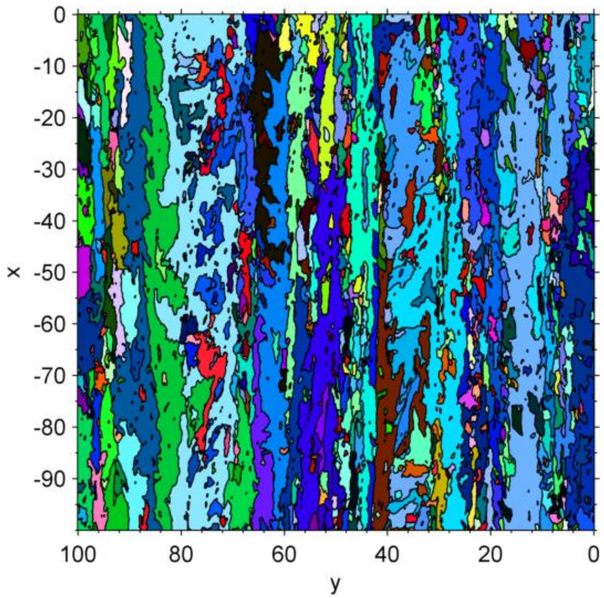
```
%Kurdimov-Sachs misorientation
ori_KS = orientation('axis',Miller(1,1,2,CS),'angle',90*degree,CS,CS);

%Nishiyama-Wassermann misorientation
ori_NW = orientation('axis',Miller(3,6,2,CS),'angle',95.3*degree,CS,CS);

%CSL3 misorientation
ori_twin = orientation('axis',Miller(1,1,1,CS),'angle',60*degree,CS,CS);

%grain boundaries of phase 1 (ferrite)
gb= grains.boundary('1','1');

%indices of grain boundaries to delete
ind_5deg = angle(gb.misorientation,ori_KS) <5*degree |...
           angle(gb.misorientation,ori_NW) <5*degree |...
           angle(gb.misorientation,ori_twin) < 5*degree;

%merge grains
[grains_merge_5deg, grains_merge_5deg_parent_id] =
merge(grains,gb(ind_5deg));
```

- CSL3 boundaries occur at martensitic transformation.
- To recover the austenite structure from martensite CSL3 boundaries are also deleted.

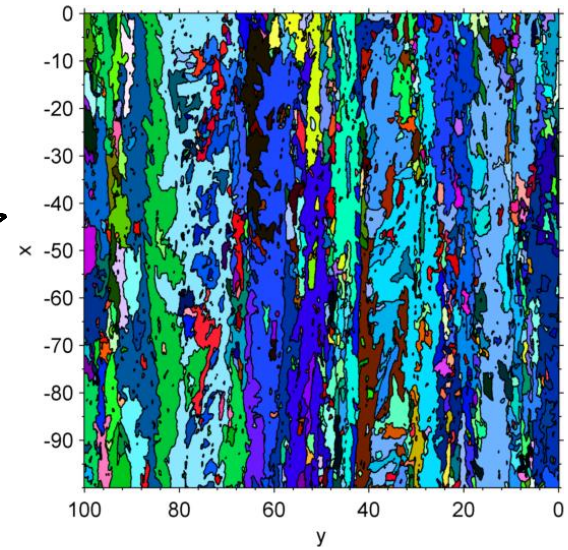


EBSD Analysis – Prior Austenite Grains

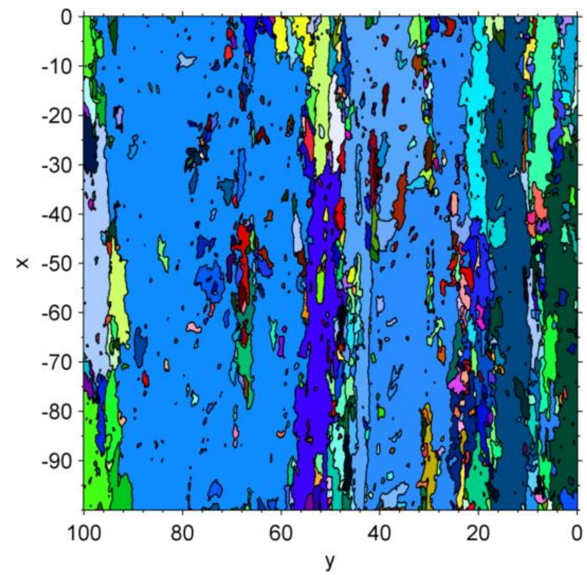
Test with K.-S. and arbitrary misorientation



35.3° about <1,2,3>
5° tolerance



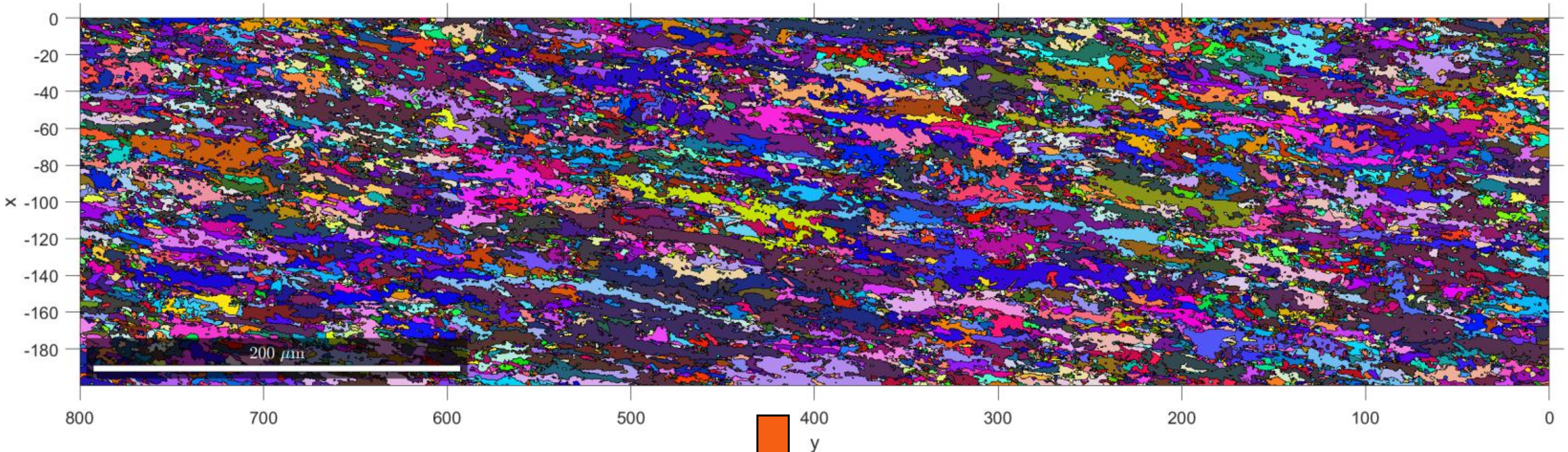
K.-S.
5° tolerance

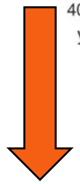


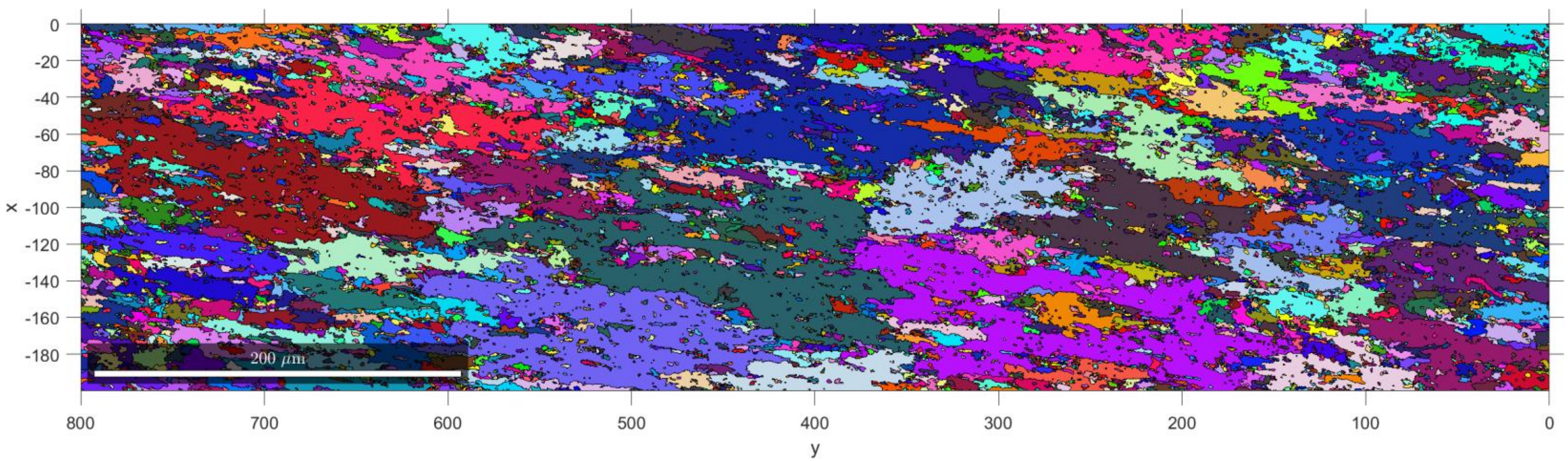
- Only with correct orientation relationship merging occurs.
- Measurement field to small for austenite microstructure

0707EE_1800µm

EBSD Analysis – Prior Austenite Grains

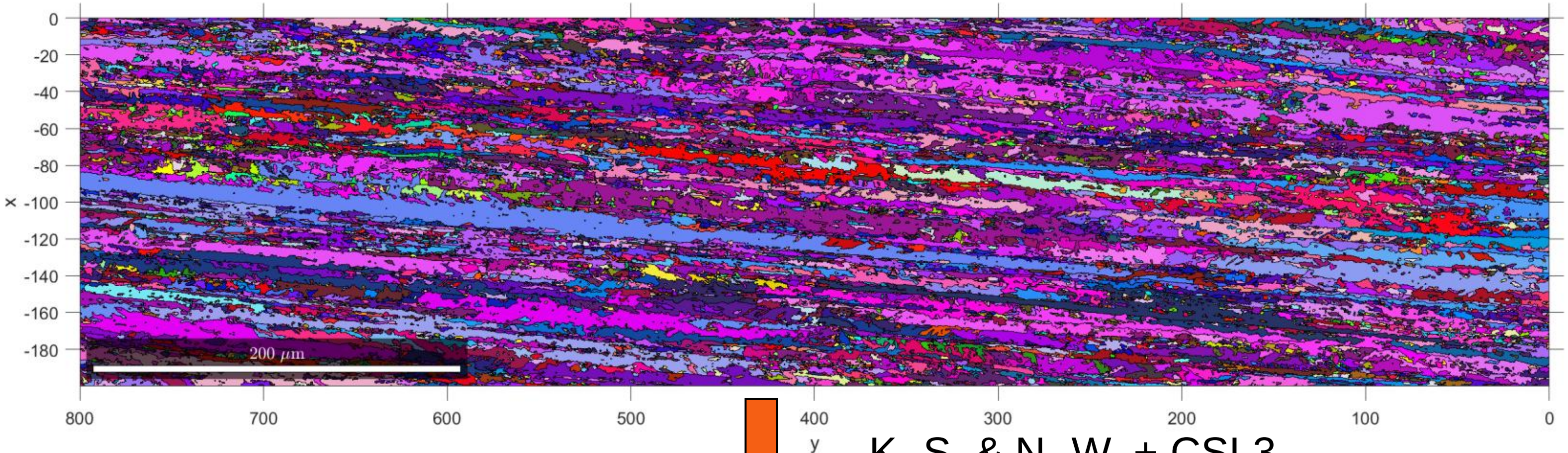




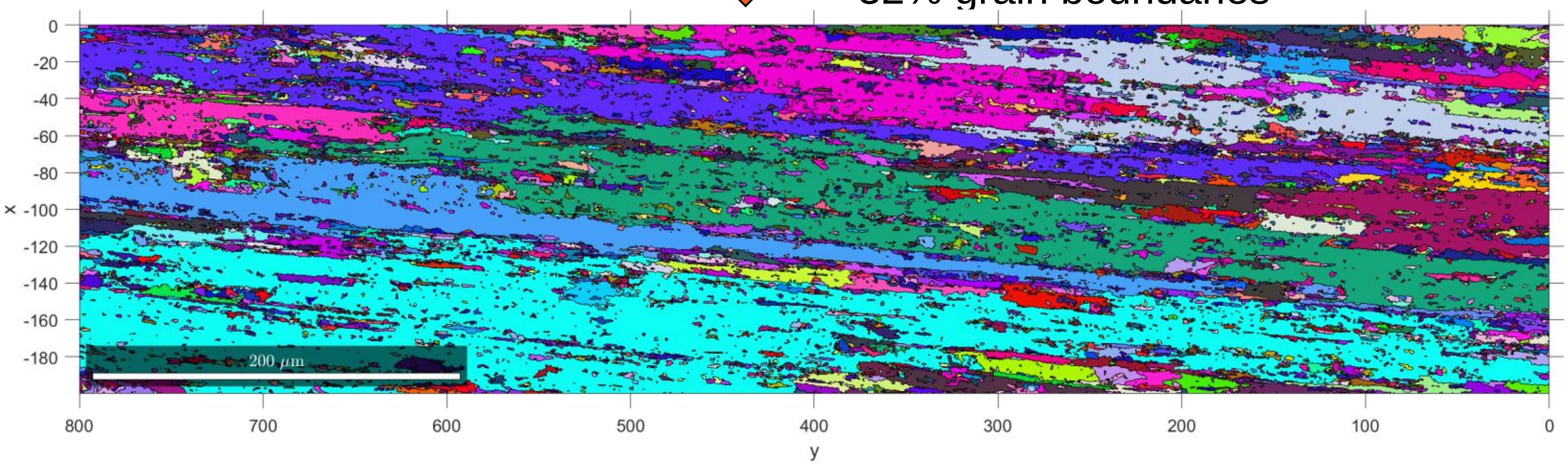


Recrystallized grain structure. Prior austenite grain size ~200 µm

EBSD Analysis – Prior Austenite Grains



K.-S. & N.-W. + CSL3
5° tolerance
-32% grain boundaries



- Elongated grain structure → No recrystallization before transformation
- Measurement field still too small…

Summary

- Using MTEX scripts can automate XRD and EBSD evaluation.
- Implementation into Matlab gives access to large amount of analysis tools.
- Examples shown
  - Elasticity tensor
  - Wave velocities and ultrasound
  - Symmetry of pole figures
  - Homogeneity
  - IQ Analysis
  - Prior Austenite Grains

No matter what you have planned ...

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Thank you for your attention!