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Characterisation of texture of strontium hexaferrite with EBSD and XRD

Timmy Reimann, Arne Bochmann, Jörg Töpfer

0. Outline

1. Introduction
2. EBSD results
3. XRD results
4. Evaluation with MTEX
5. Summary
6. Outlook

1. Introduction – field of interest

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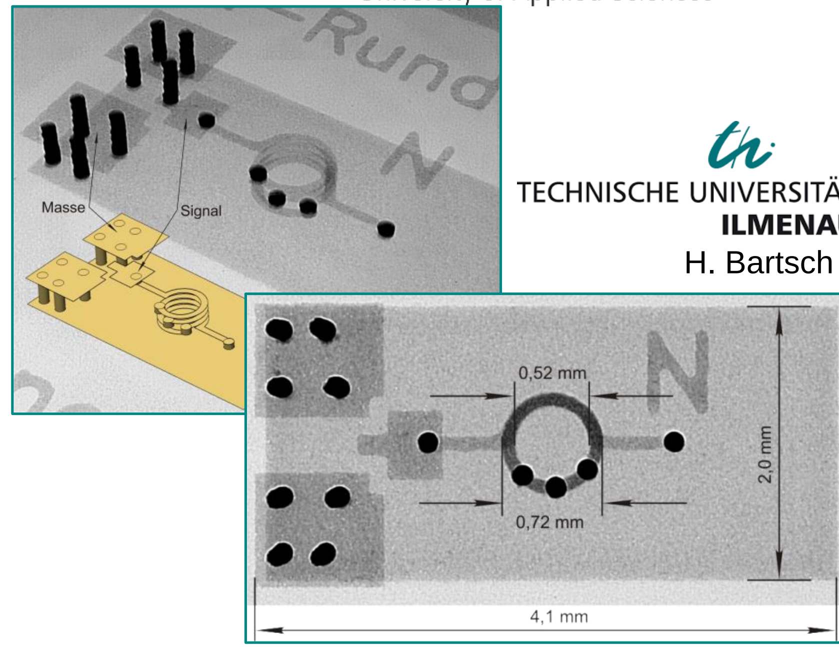
Functional ceramics

- Soft ferrites
  - Mn-Zn ferrites for multilayer inductors
  - M-, Y-, Z-type hexaferrites
- Hard ferrites — TRIDELTA Hartferrite GmbH
  - Sr hexaferrites for permanent magnets
- Dia- and Piezoelectrics — PI Piezo Technology
  - $CaCu_3Ti_4O_{12}$
  - PZT ($PbZrO_3$)
  - Pb free BNBT $(Bi_{0.5}Na_{0.5})TiO_3$ – $BaTiO_3$ and KNN $(K_{0.5}Na_{0.5}NbO_3)$
- Thermoelectrics
  - CCO ($Ca_3Co_4O_9$); $CaMnO_3$
- Low temperature ceramic cofiring (LTCC) devices sintered at 900°C



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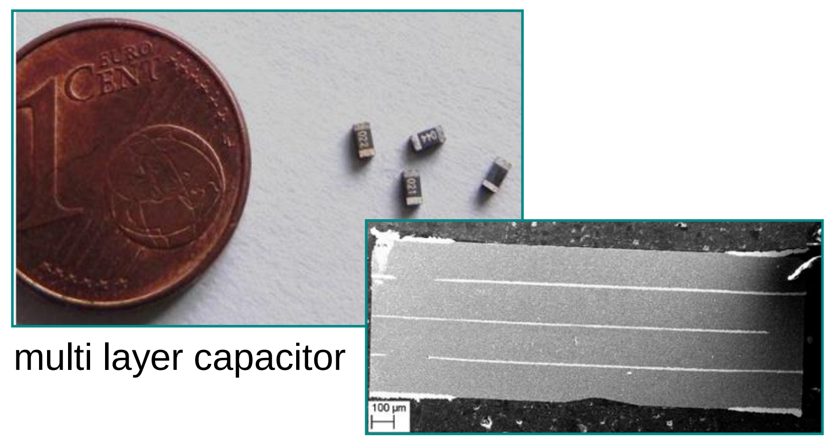
H. Bartsch

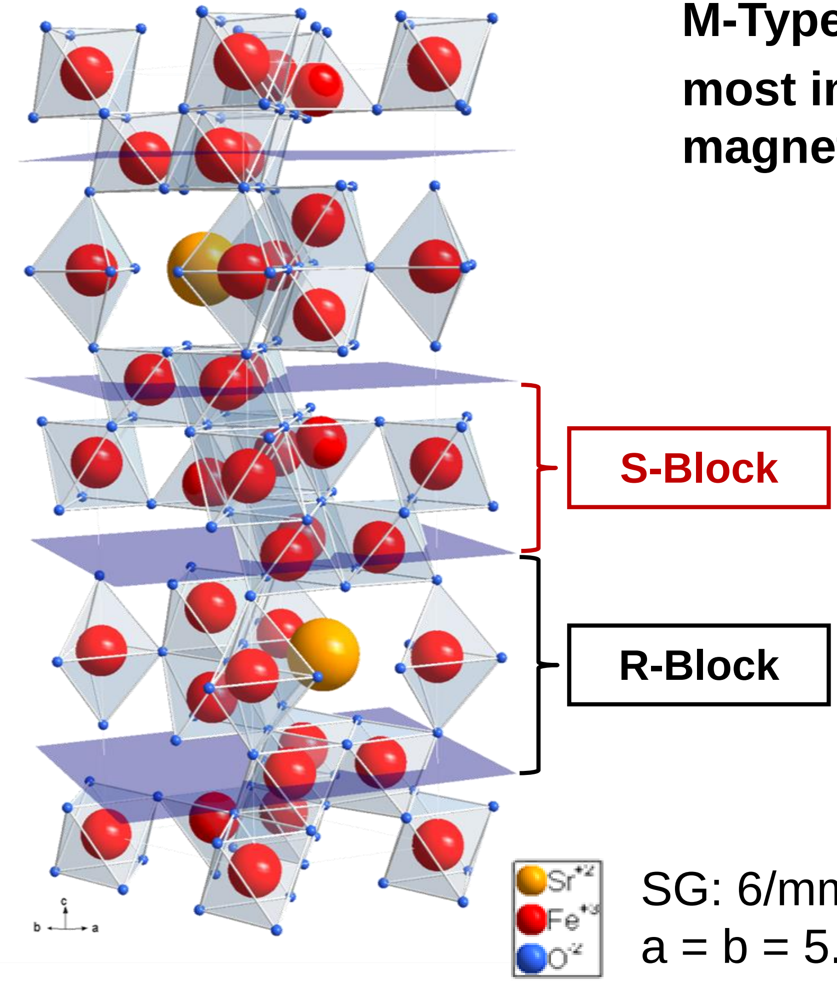
multi layer round coil



multi layer capacitor

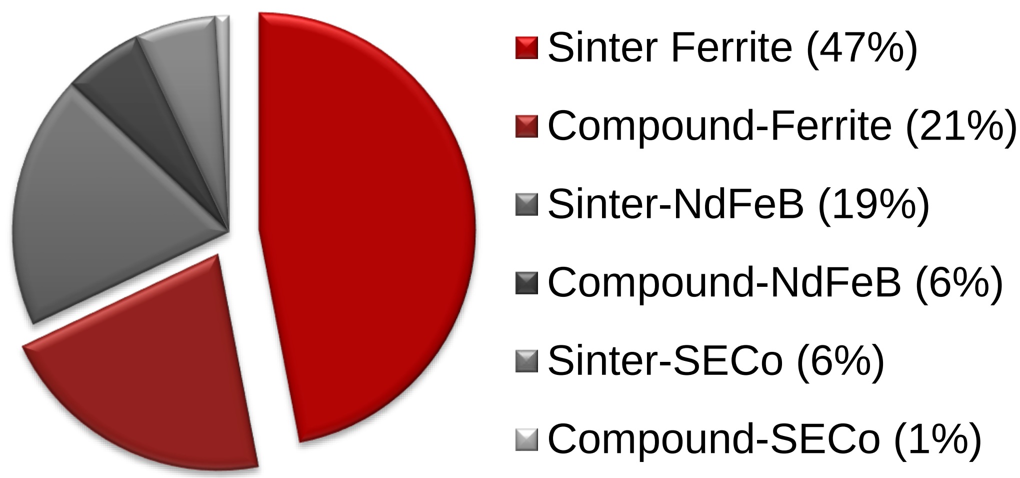
1. Introduction - Hexaferrite

$Sr^{2+}Fe^{3+}_{12}O^{2-}_{19}$



M-Type hexagonal ferrites (Ba/Sr)$Fe_{12}O_{19}$:
most important material group for permant magnets

market share:



SG: 6/mmm
a = b = 5.8836 Å
c = 23.0376 Å

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Data sheet
Maniperm 882 :

flux density B
remanence:

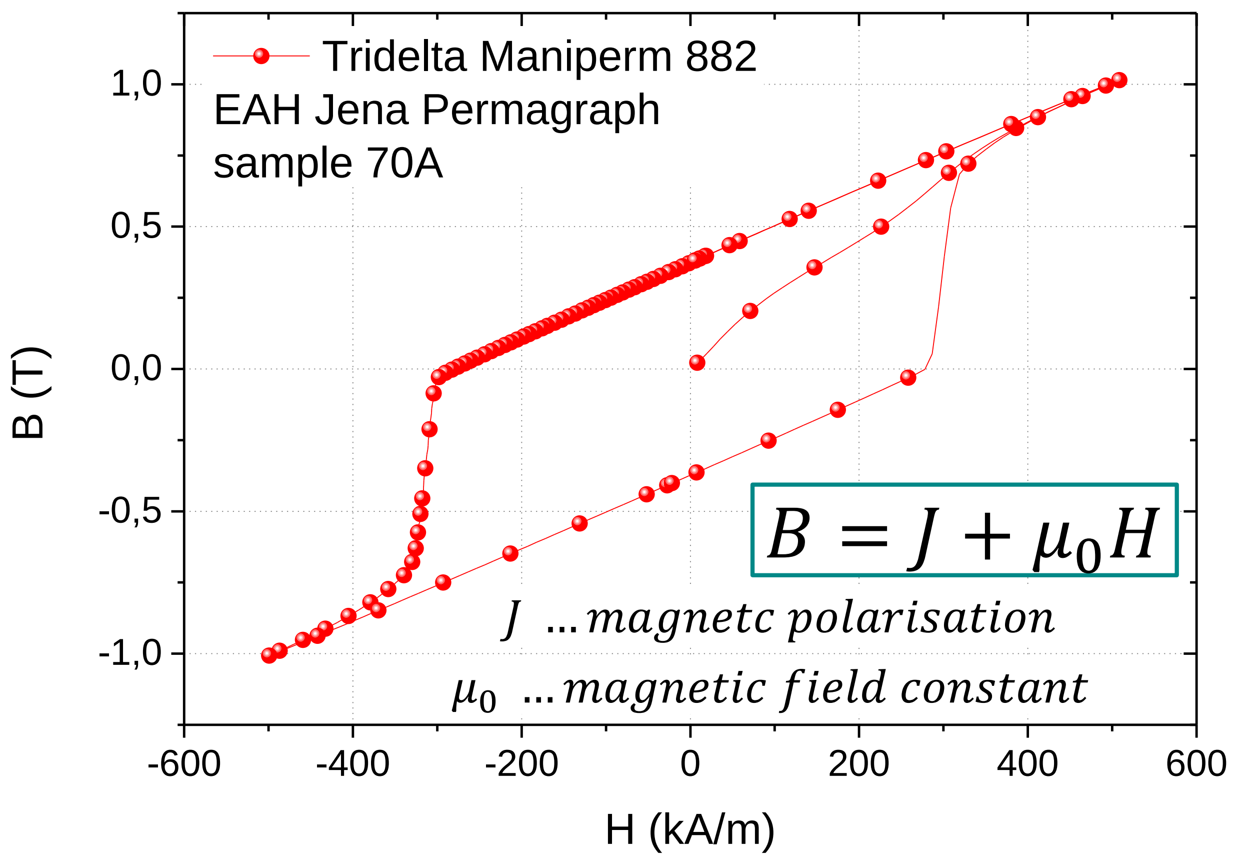
B_R = 405-415 mT

magnetic field H
coercivity:

H_{CB} = 270-240 kA/m

energy product:

$(BH)_{max}$ = 32 kJ/m²



$$B = J + \mu_0 H$$

J ... magnetc polarisation

μ_0 ... magnetic field constant

1. Introduction – sample preparation

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uniaxial pressing with applied magnetic field

$$H = I\,\frac{N}{\sqrt{l^2 + D^2}}$$

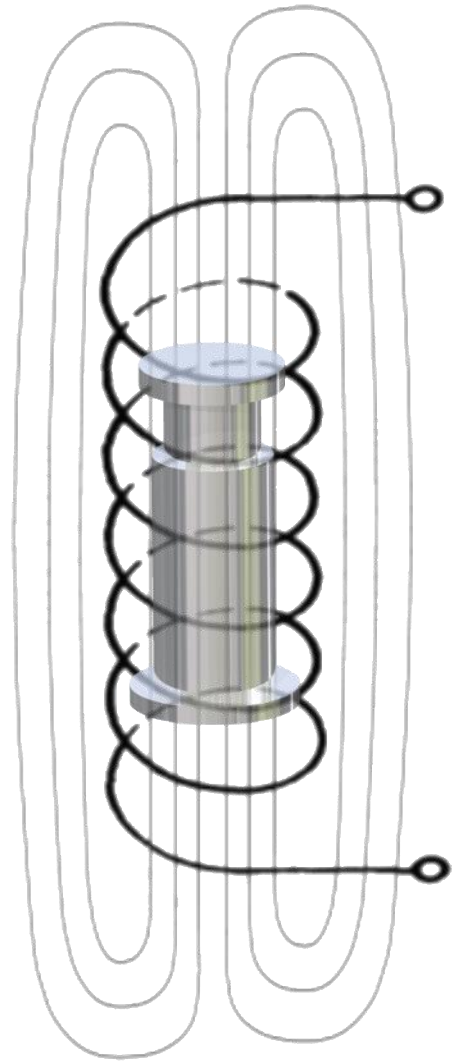
I ... *current*

N ... *number of windings*

l ... *length*

D ... *coil diameter*

current variation:
0 A; 20 A; 40 A; 60 A; 70 A



1. Introduction - sample preparation

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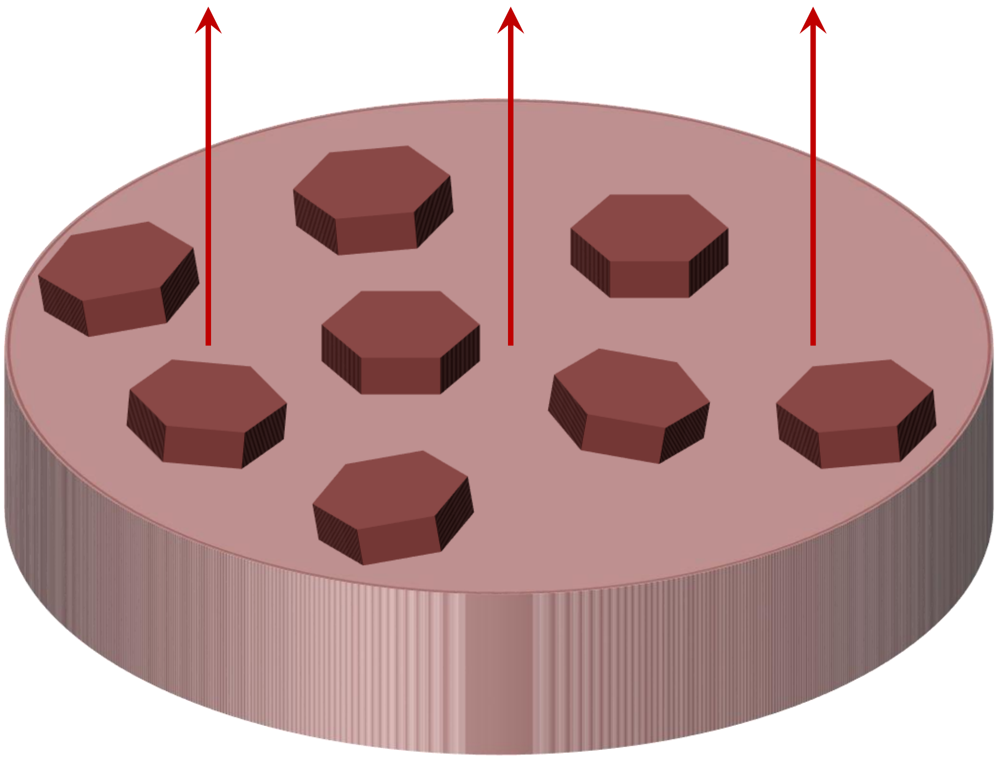
uniaxial pressing with
applied magnetic field

H field in z direction



orientation of c axis in powder particles of slurry in z direction due to uniaxial magneto-crystalline anistropy of $SrFe_{12}O_{19}$

goal:

increase of remanence in z direction

1. Introduction – magnetic properties

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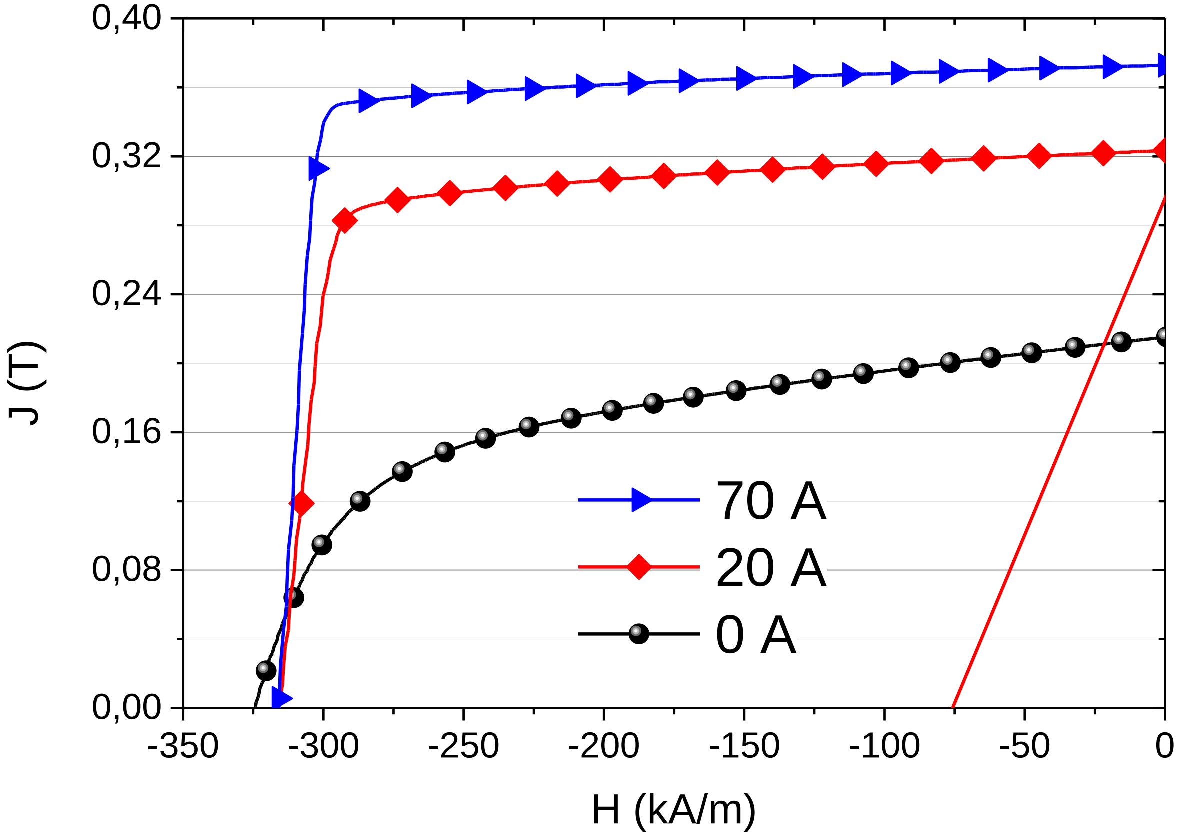
2. quadrant of H – J plot

increase of remanence with increasing H field in pressing process observed



task:

characterisation of texture

$$B = J + \mu_0 H$$

Characterisation of texture

- measurement of texture with EBSD and XRD and evaluation of both data sets with same procedure
- computing ODF
- tetermine vector of main orientation
- calculating amount of fibre texture
- derivation around main orientation and calculating Br according to the ODF

2. EBSD results – 0 A sample

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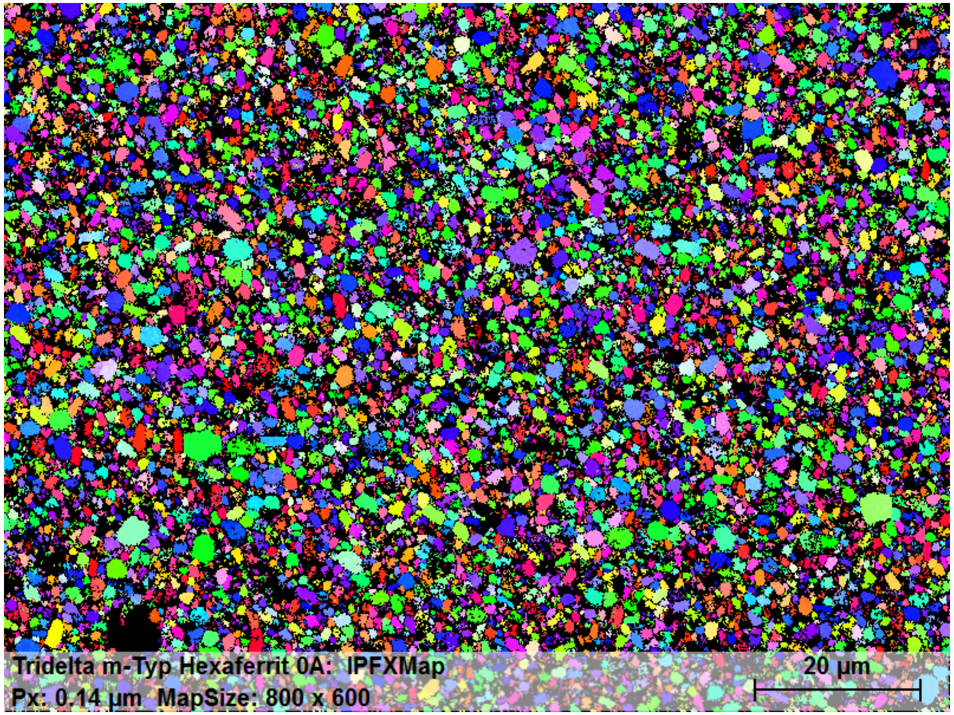
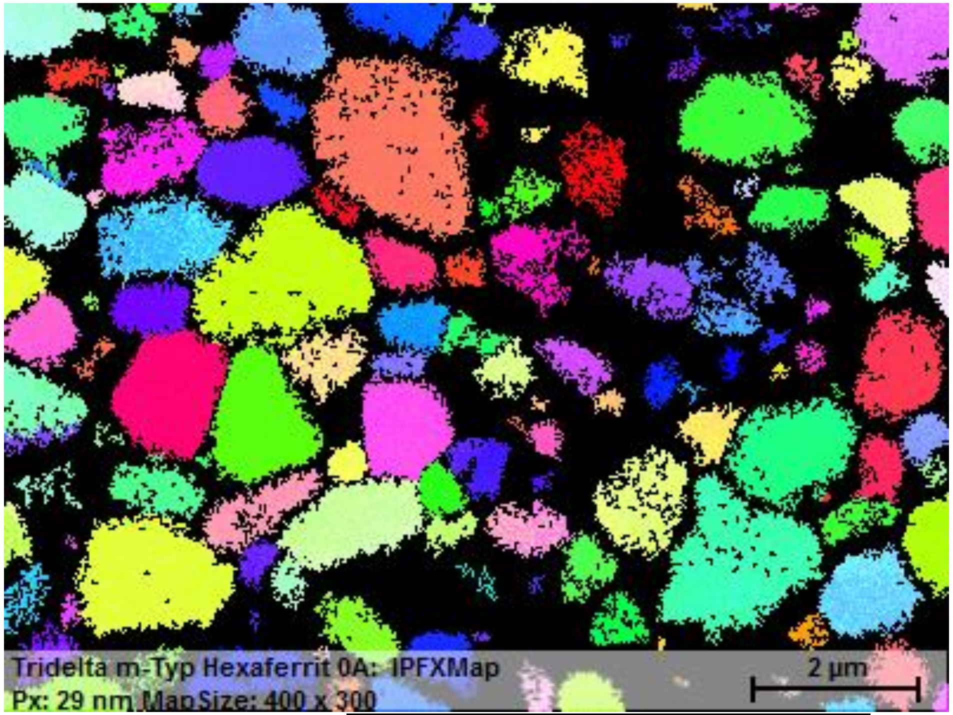
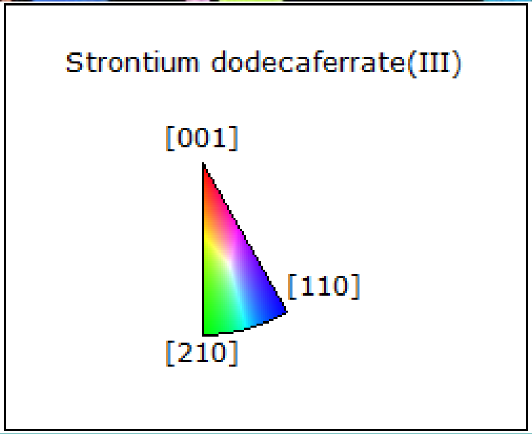
IPFX Map

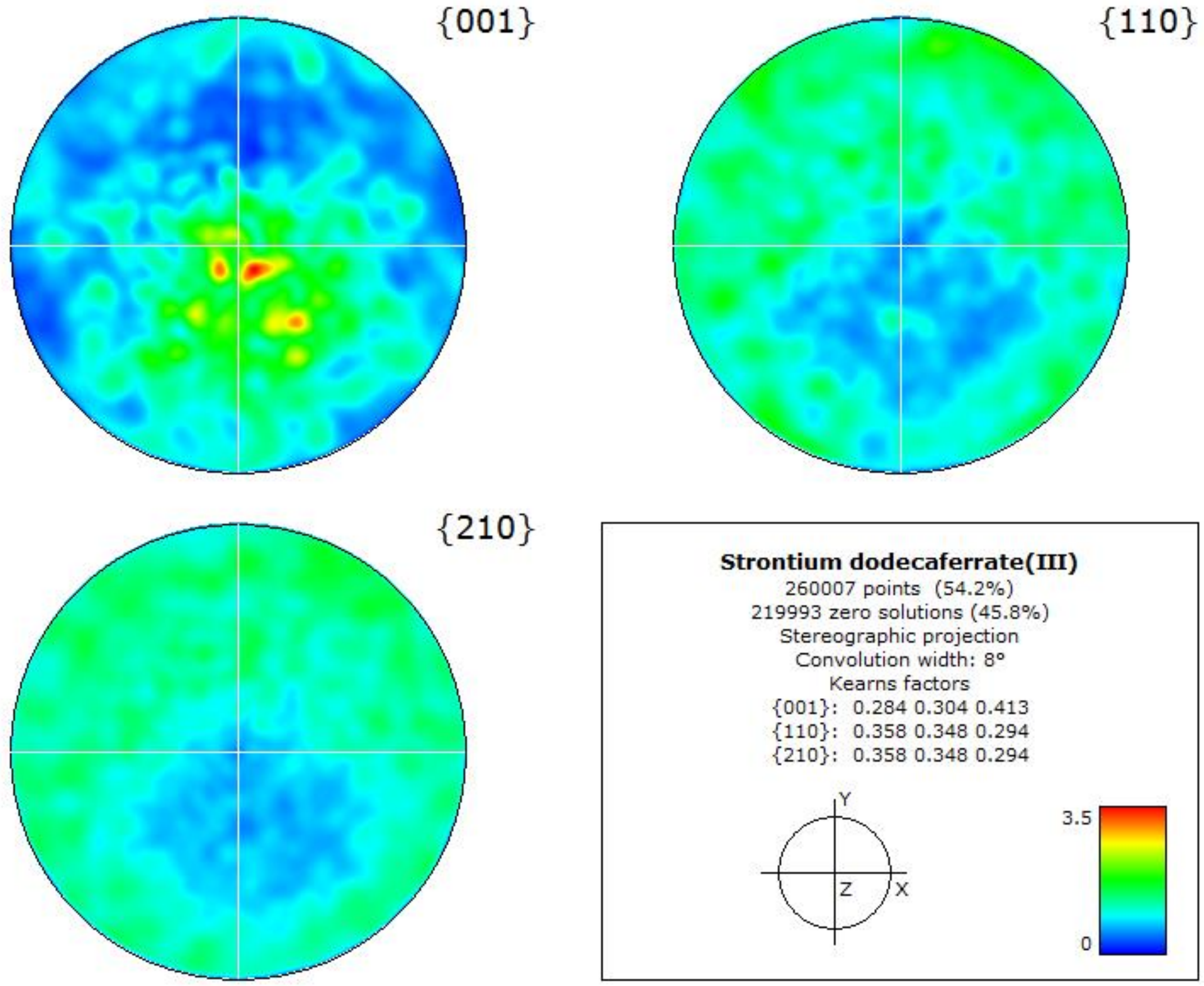




112 x 84 µm



2. EBSD results – 0 A sample



2. EBSD results – 20 A sample

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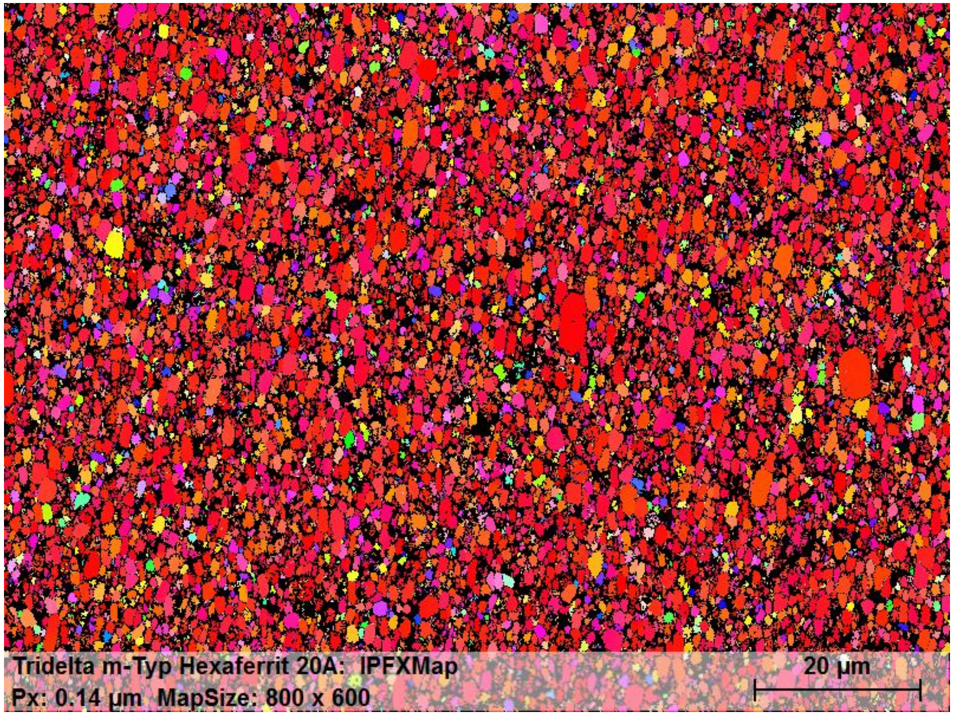
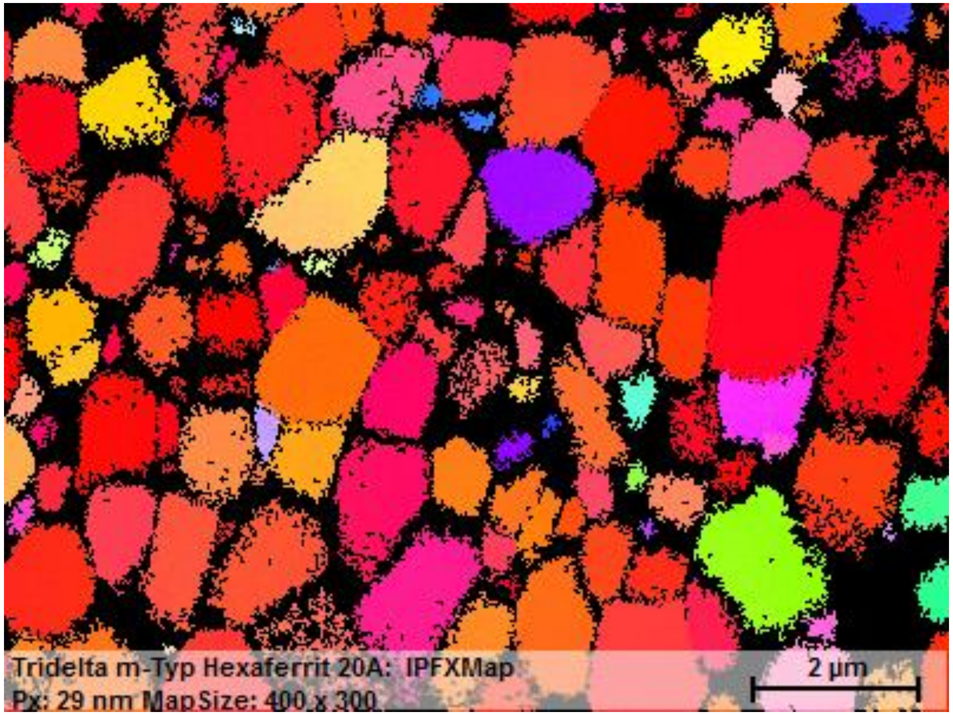
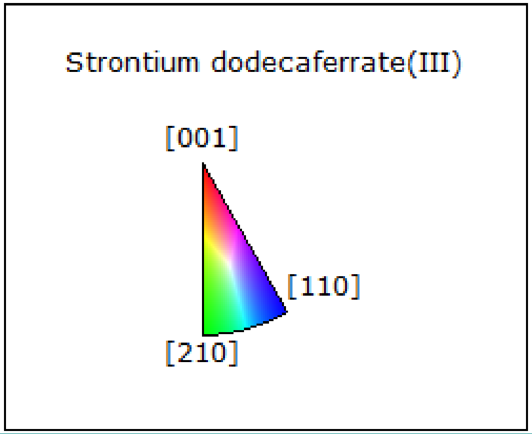
IPFX Map







2. EBSD results – 20 A sample

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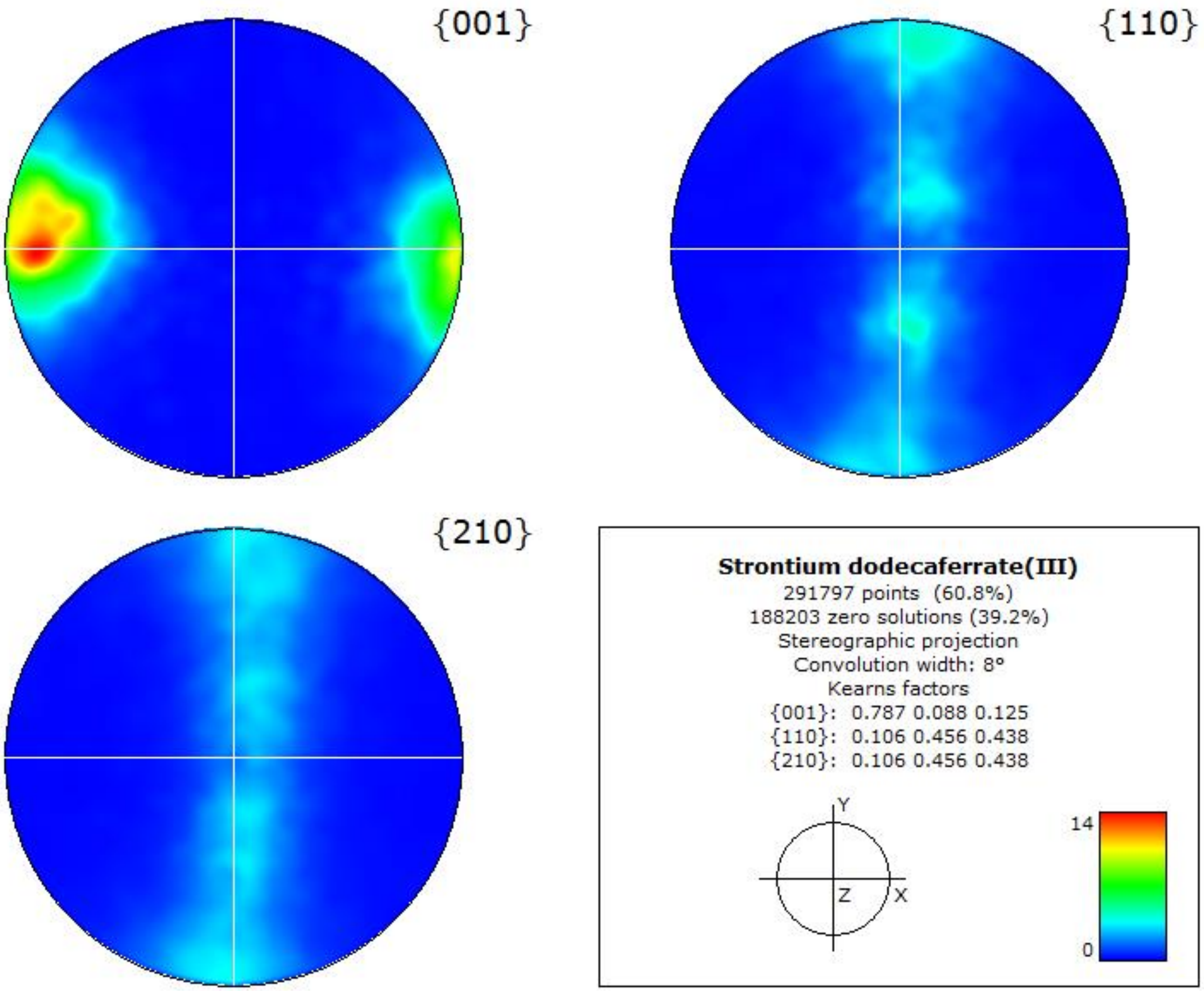

2. EBSD results – 70 A sample

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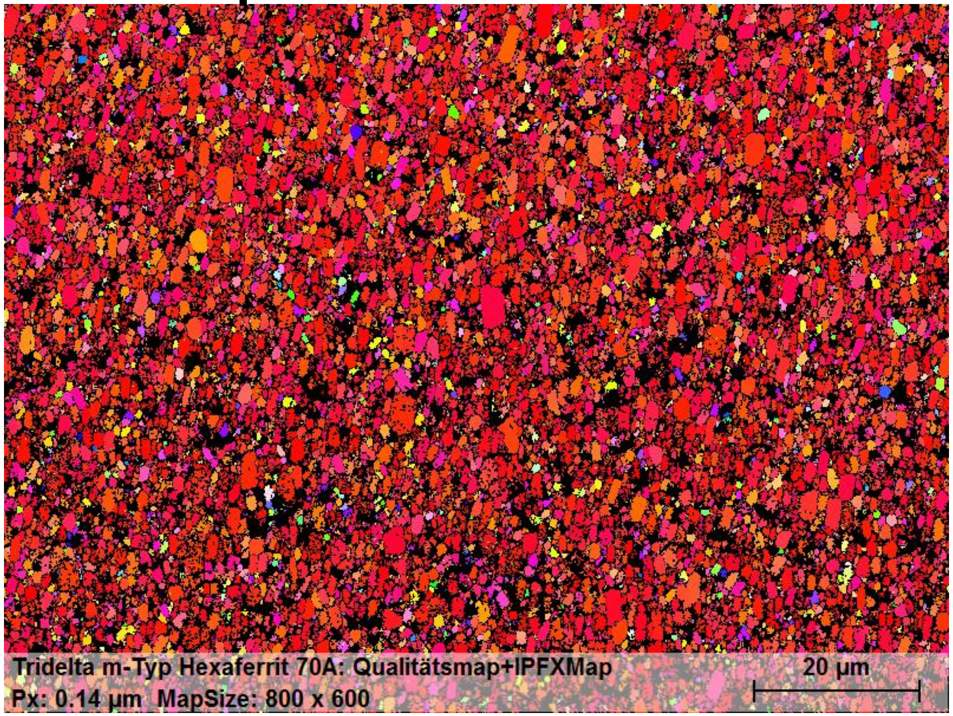
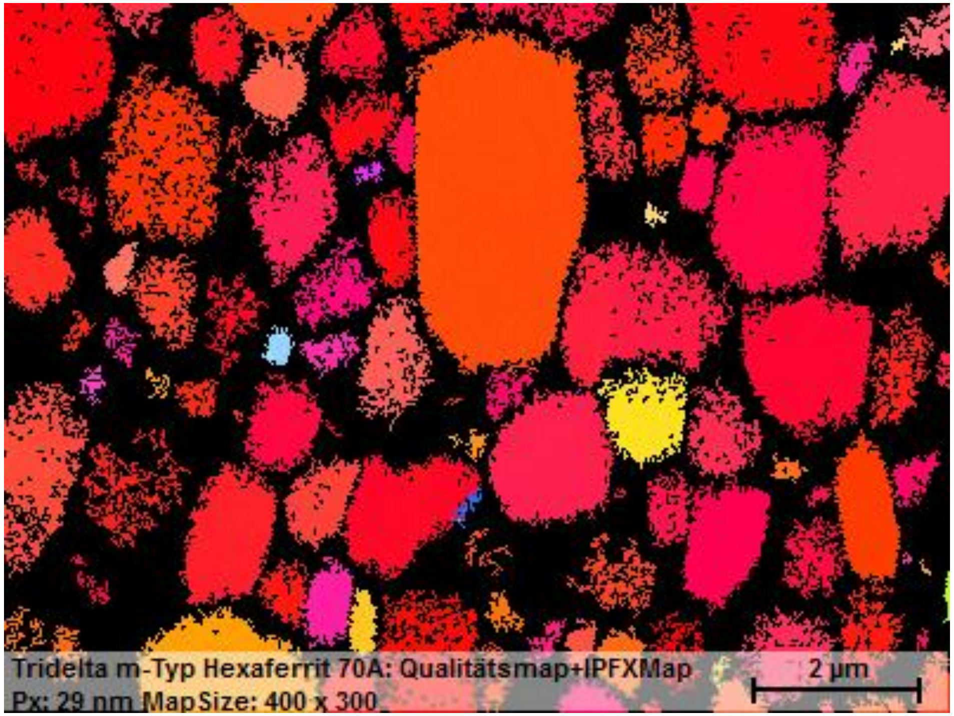
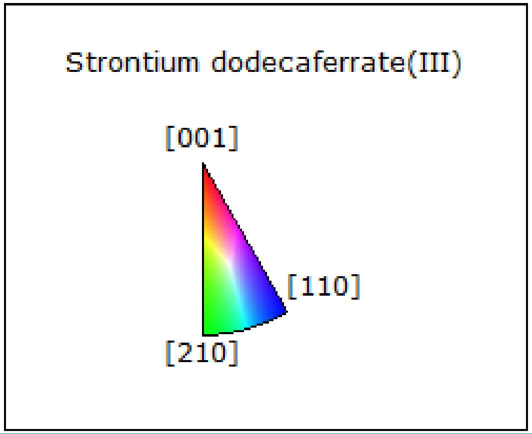
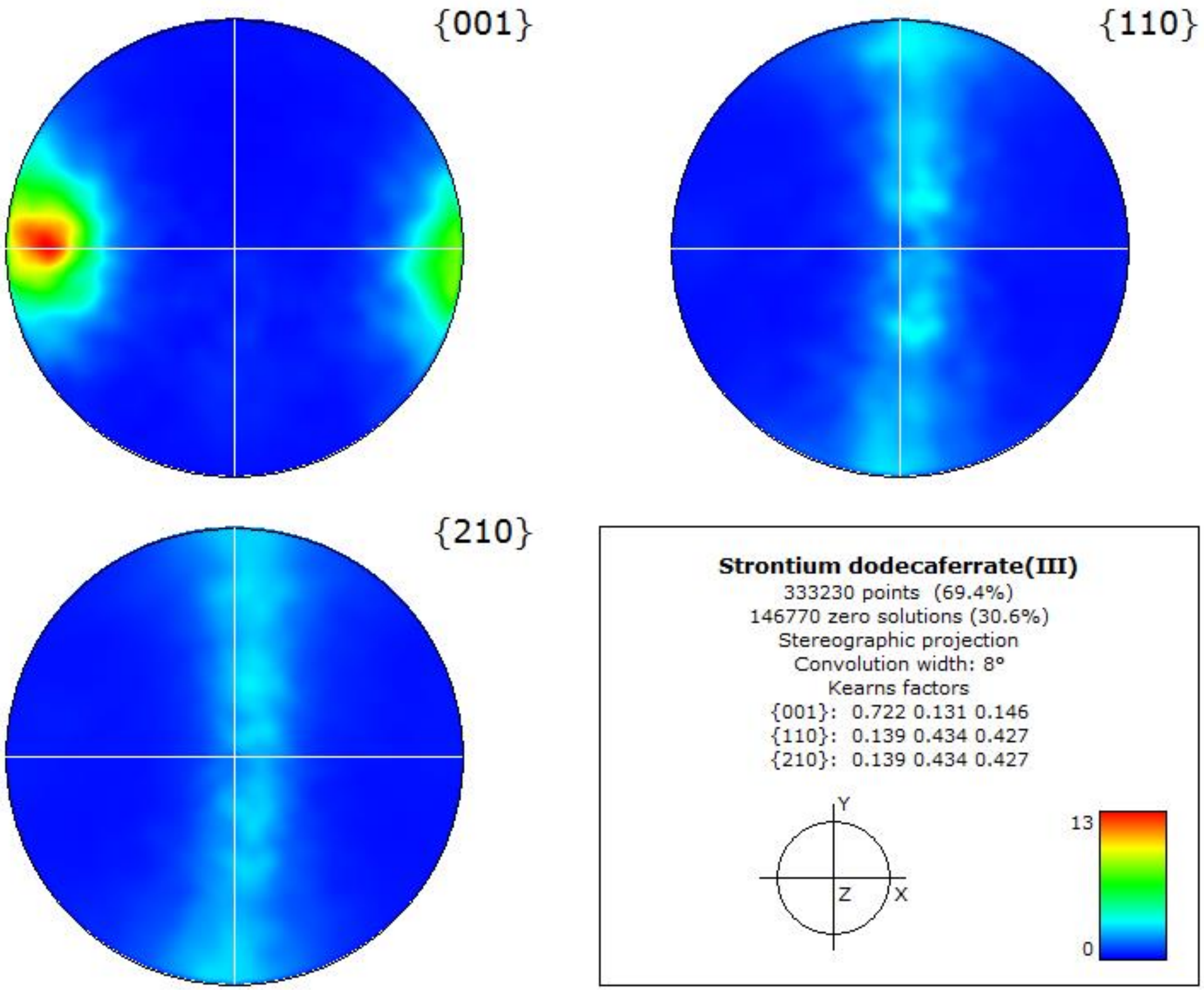
IPFX Map







2. EBSD results – 70 A sample



2. EBSD results – ODF (MTEX)

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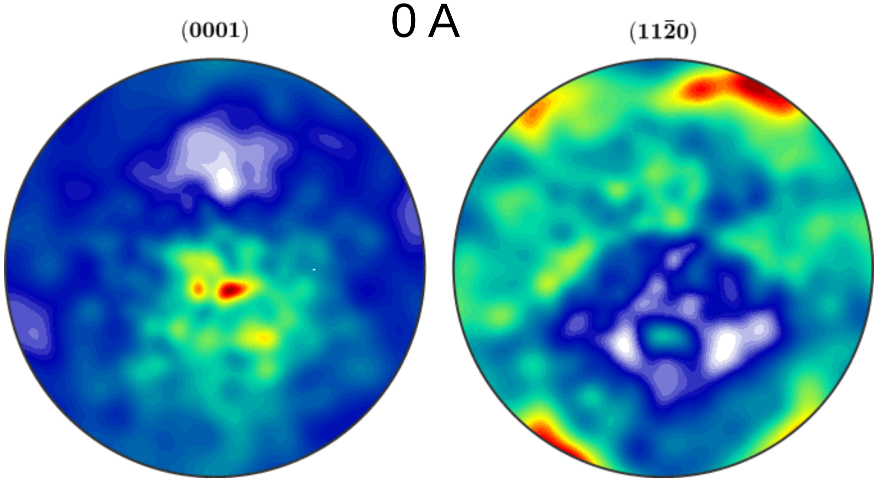
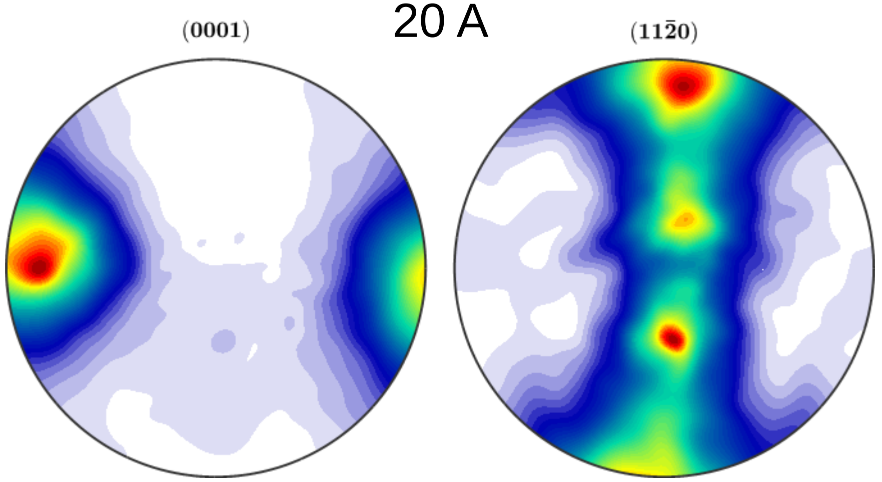
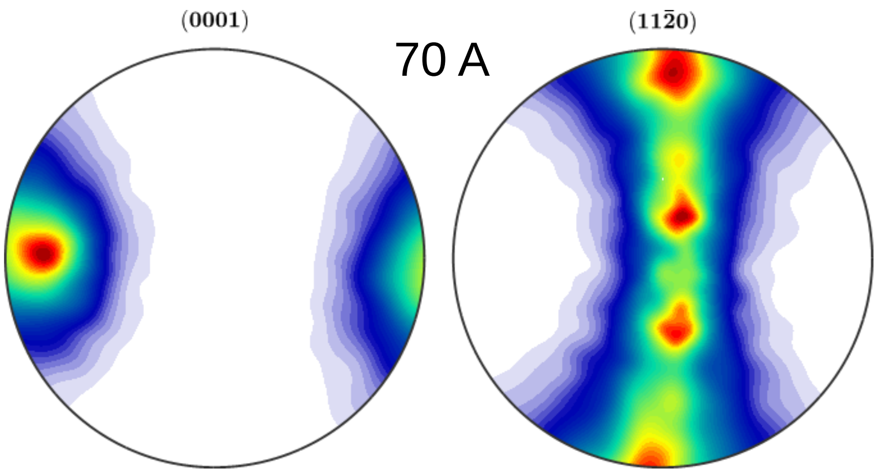
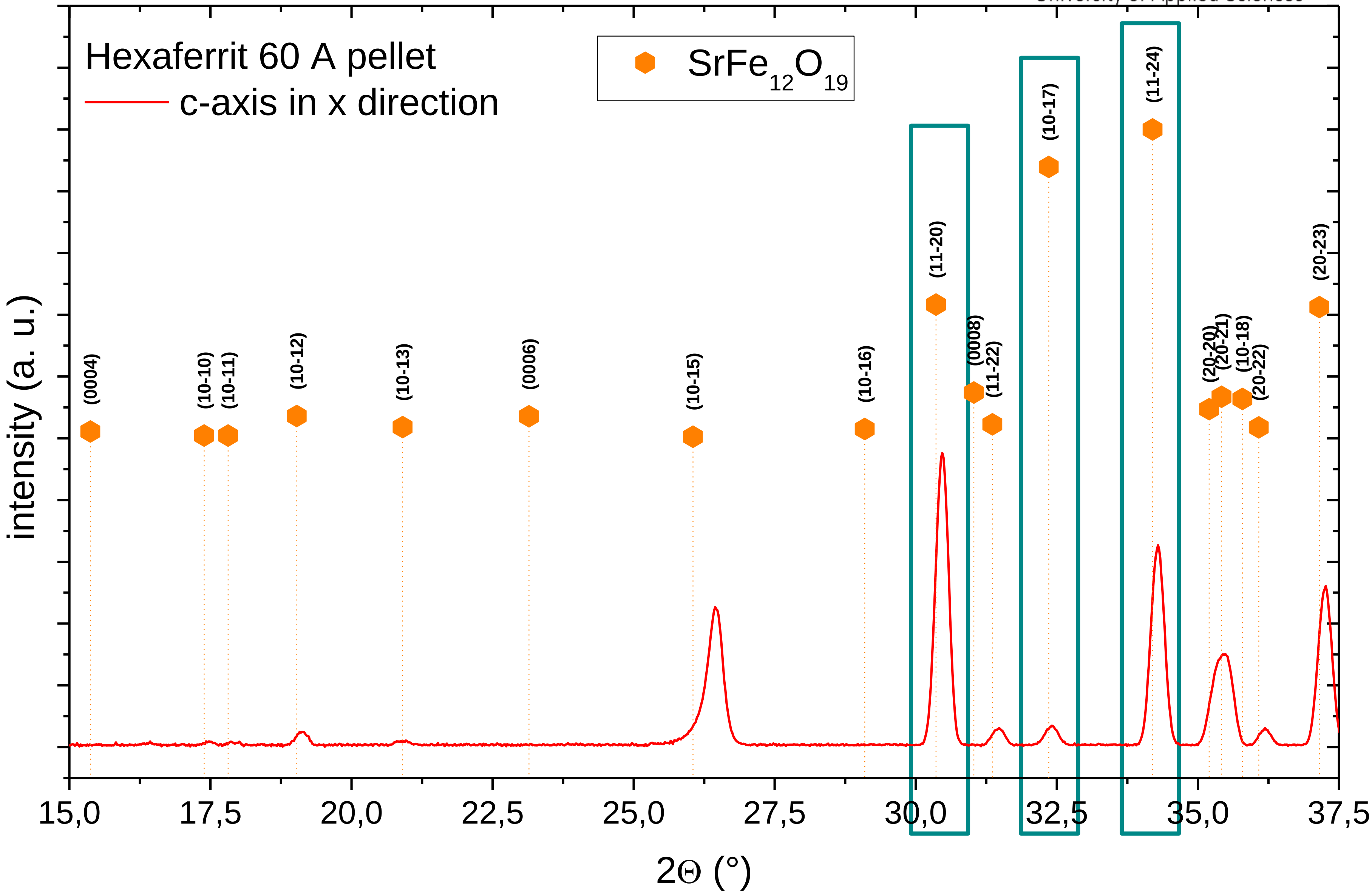
To calculate the ODFs given in the pole figures below a kernel function with a halfwidth of 5 ° was used.







3. XRD results



3. XRD results – Bruker Multex

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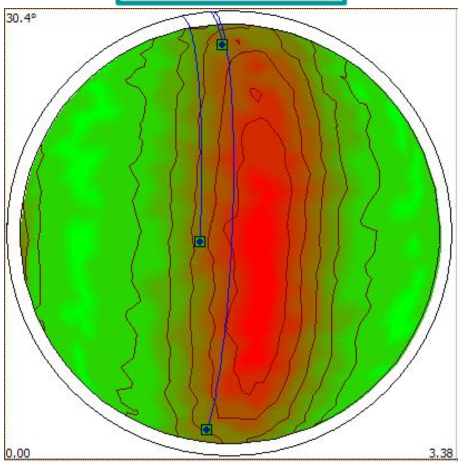
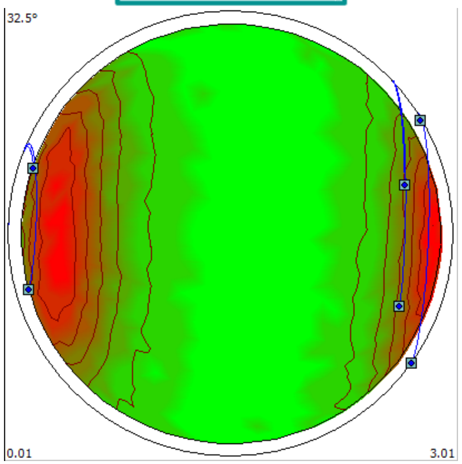
Polfigures

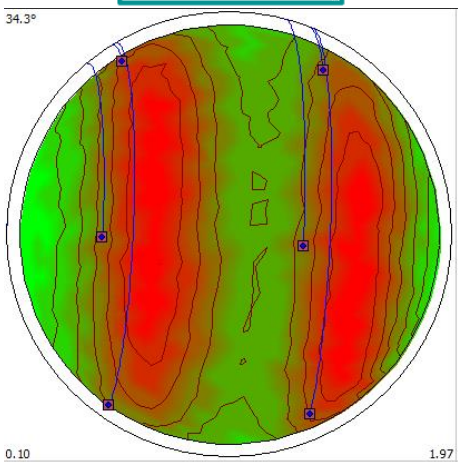
$(10\bar{1}0)$



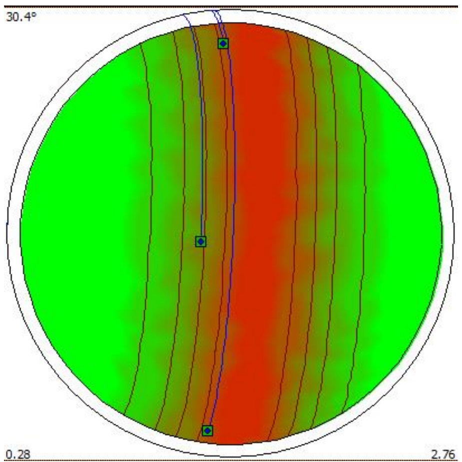
$(10\bar{1}7)$

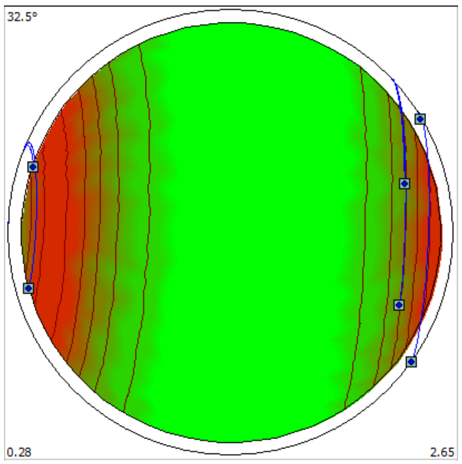


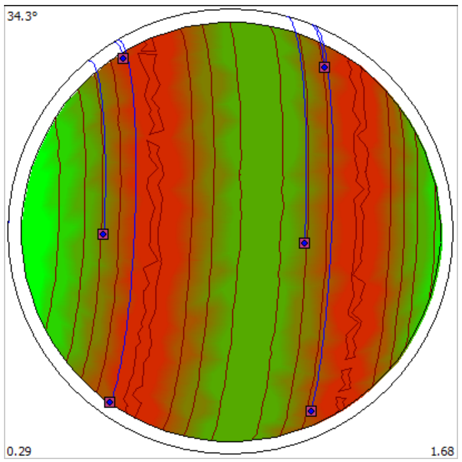
$(11\bar{2}4)$



Calculated polfigure for fibre texture







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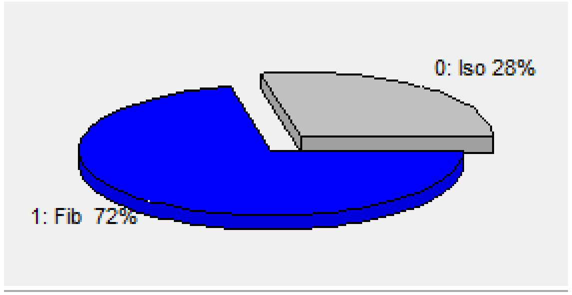
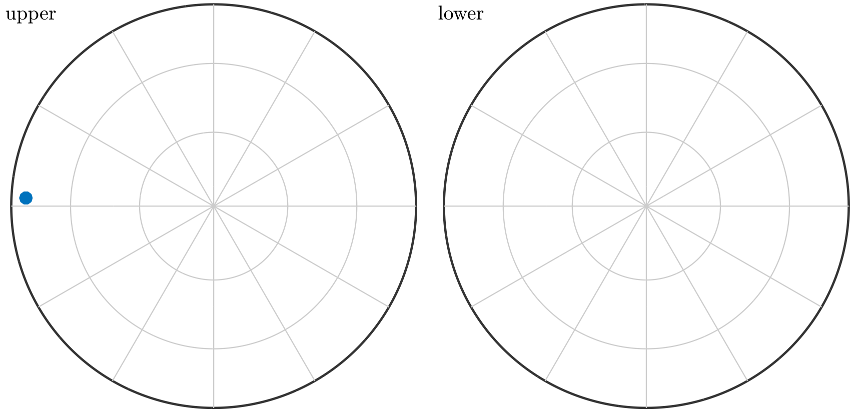
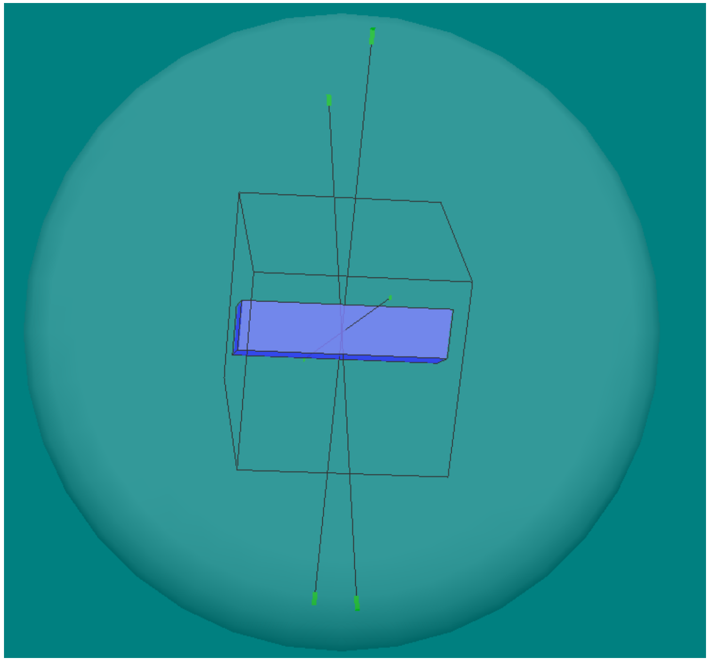
phases



rotation axis f



rotation axis
halfwidth: 30°
f.polar = 82,32°
f.azimuth = 177,49°



4. Evaluation with MTEX

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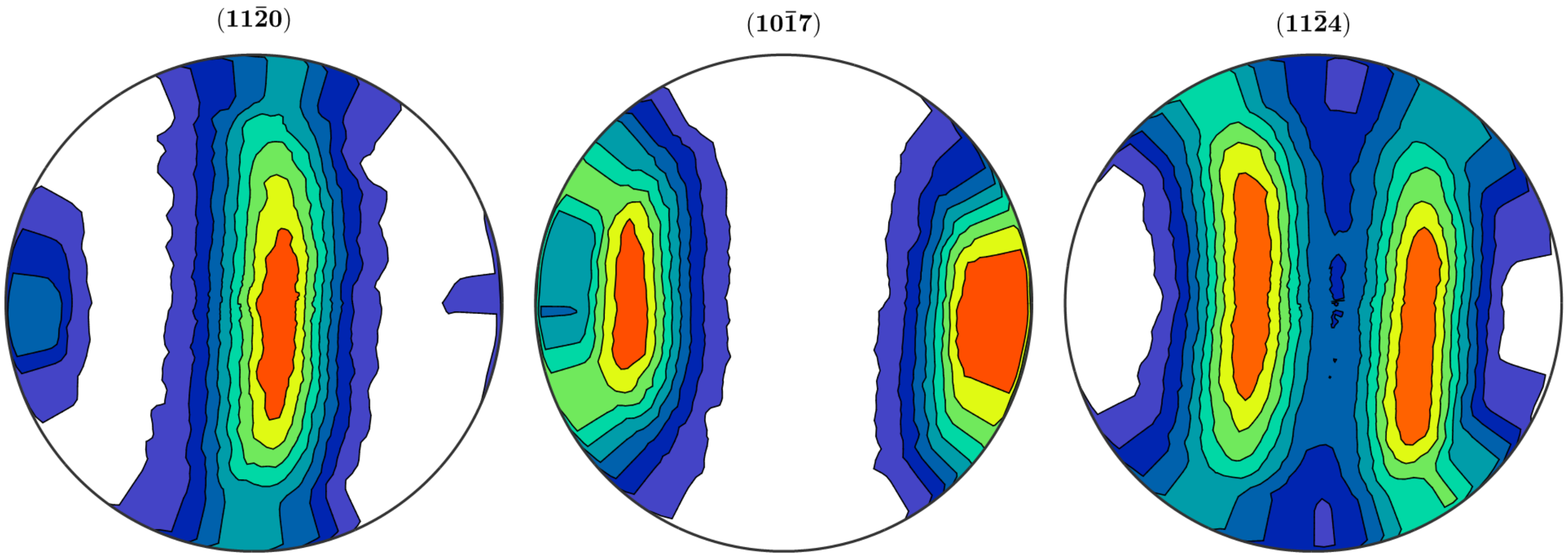
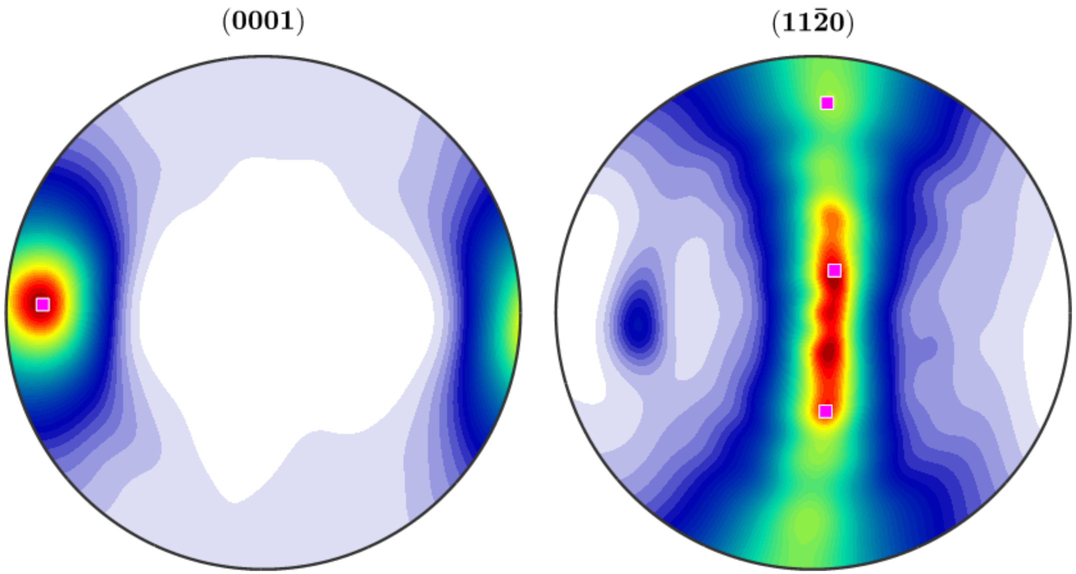
Polfigures plotted with MTEX (halfwidth = 5°)

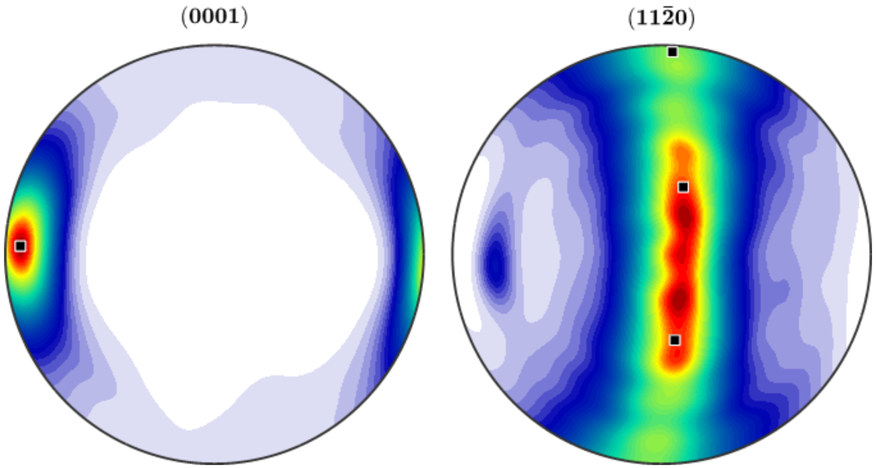


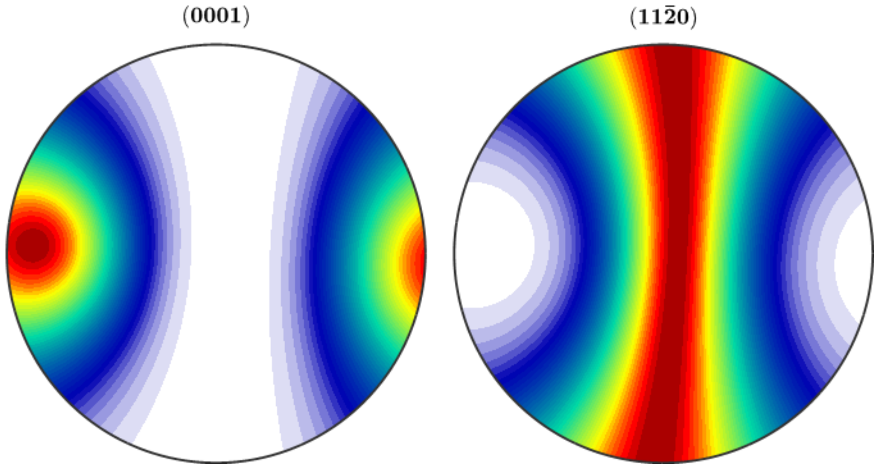
Calculation of ODF
Plot of polfigures



4. Evaluation with MTEX

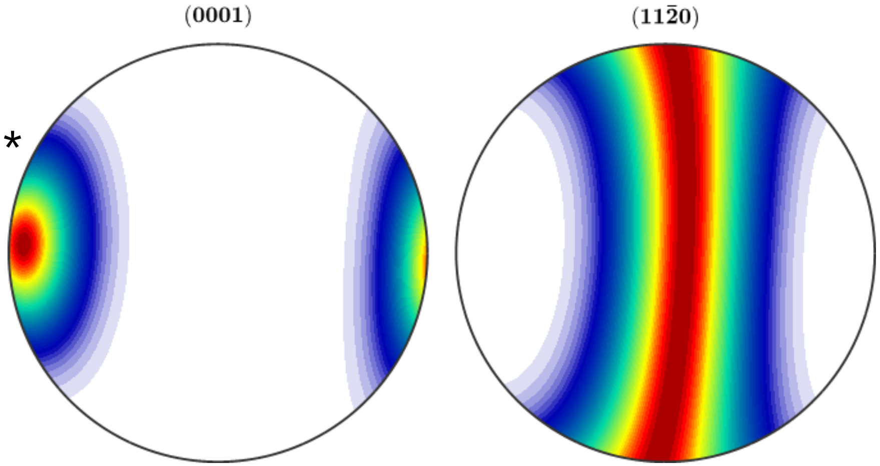
polefigure of ODF





Multex Fit
fibre: 72 % , halfwidth: 30 %

MTEX Fit
ODF_mea = x*ODF_Fibe + (1-x)*ODF_Uni*
fibre:69 %, halfwidth: 17 %
uniform: 31%



*matlab script at the end

4. Evaluation with MTEX

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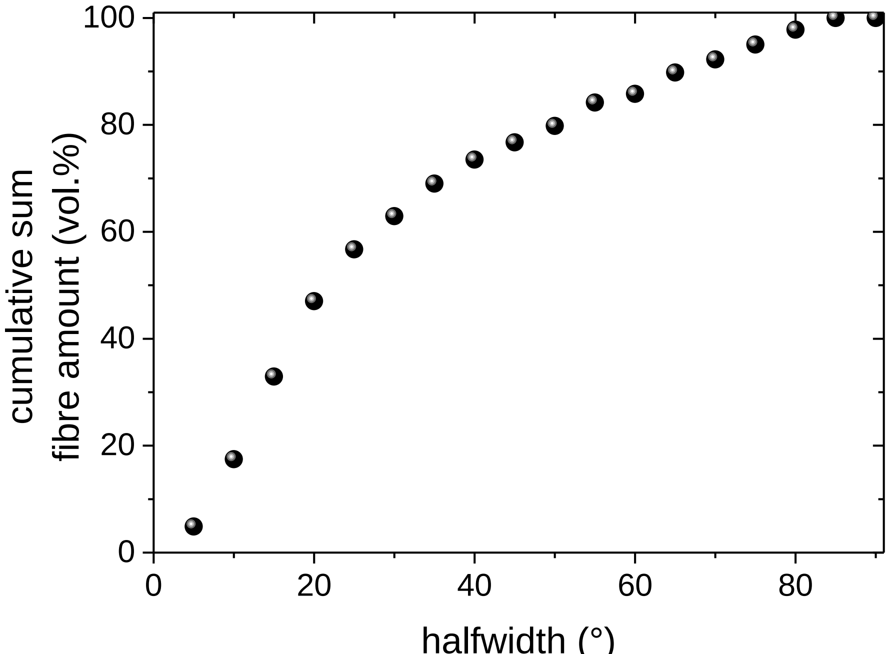
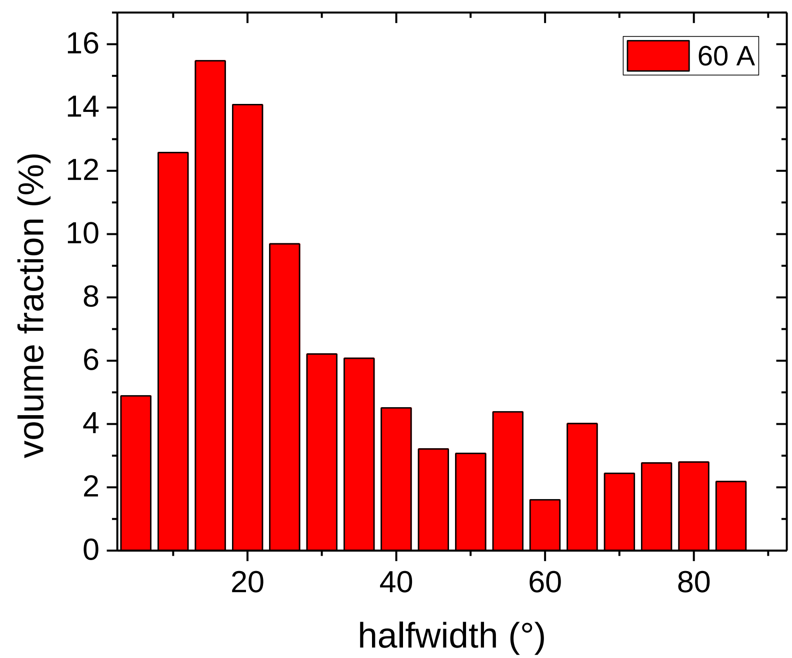
```
Anteilfibre = [];
i = 1;
for theta = 5 : 5 : 90
    theta_array(i) = theta;
    Anteilfibre(i) = fibreVolume(odf_measured, Miller(0,0,1,cs), o_min_vector,…
theta*degree) * 100;
    i = i+1;
end
```





4. Evaluation with MTEX

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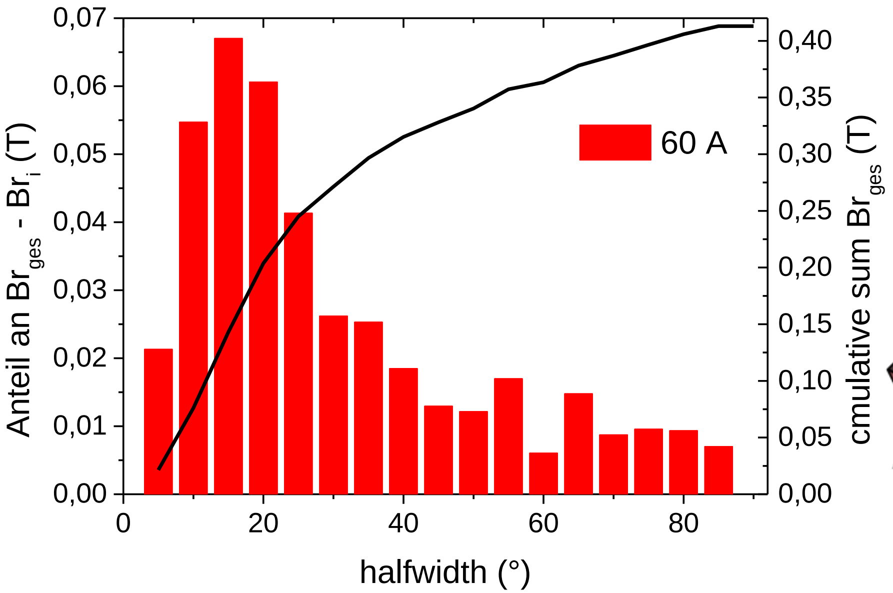
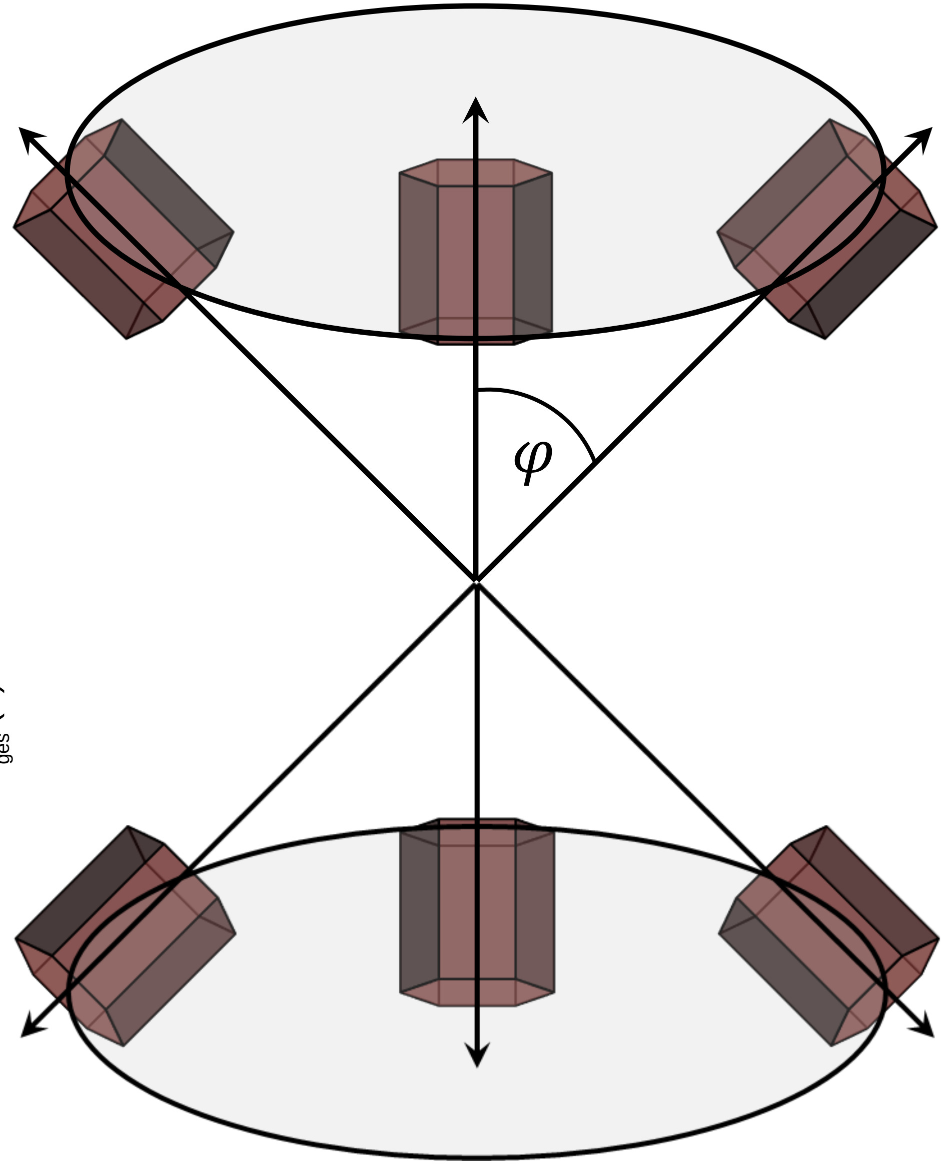
Br (theo.) = 0.437 T
in c direction

$$B_{R_i} = B_{R_{theo}} \cos \varphi_i$$

$$B_{R_{ges}} = \sum_{i}^{N_{90°}} B_{R_{theo}} \cos(\Delta\varphi * i)$$

$$B_{R_{ges}} = 0.413\ T;\ \frac{B_{R_{ges}}}{B_{R_{theo}}} 100 = 95\ \%$$





5. Summary – 60 A sample

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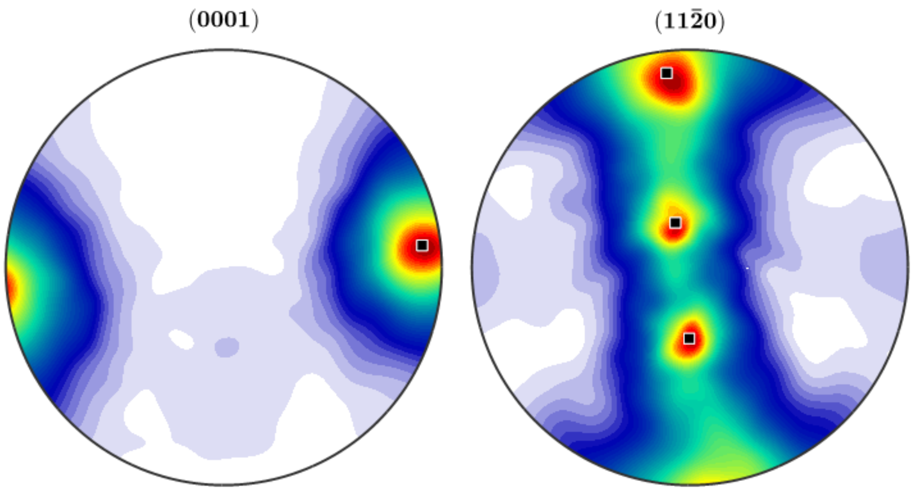
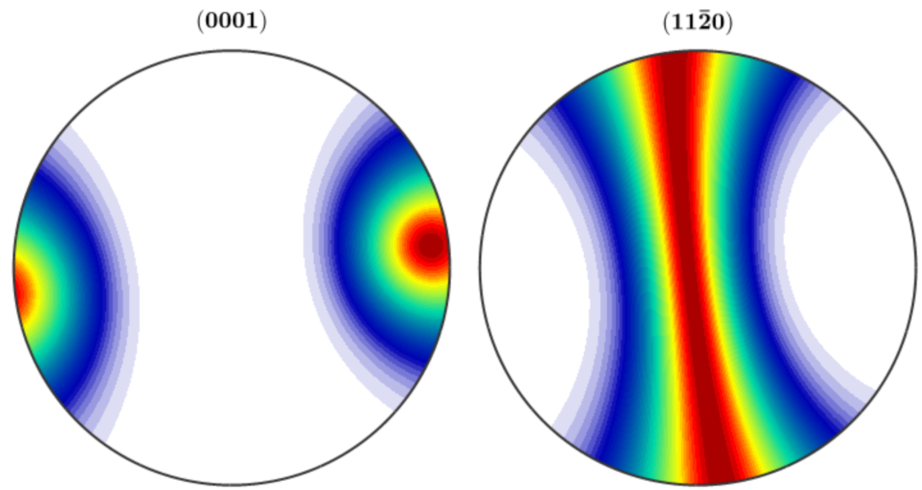
EBSD polefigure of ODF

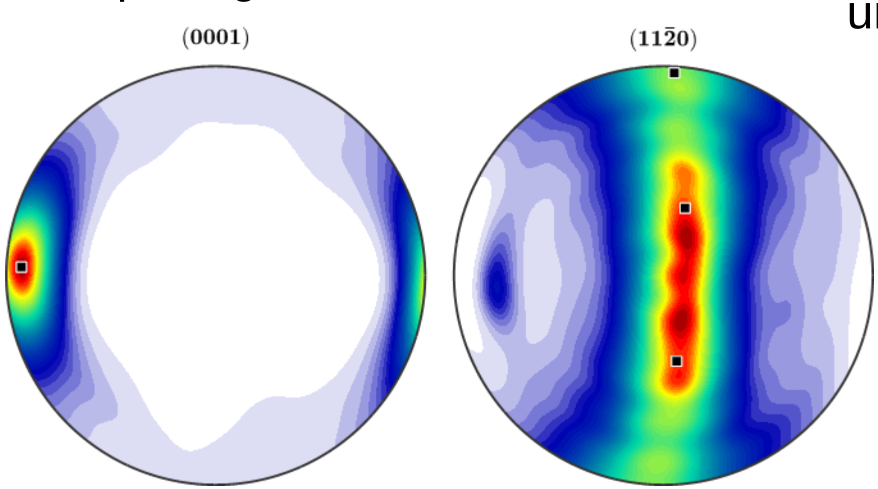


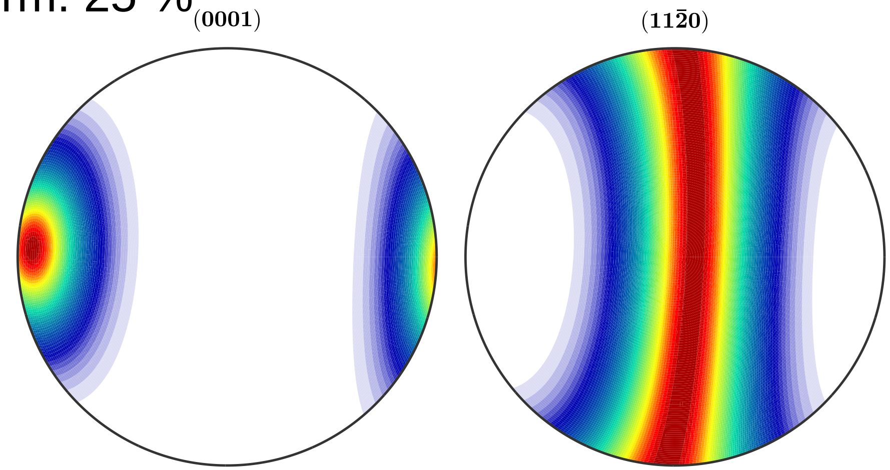
MTEX Fit



fibre: 75 %, halfwidth = 20,5°
uniform: 25 %

XRD polefigure of ODF





fibre:69 %, halfwidth: 17°
uniform: 31%

5. Summary – 60 A sample

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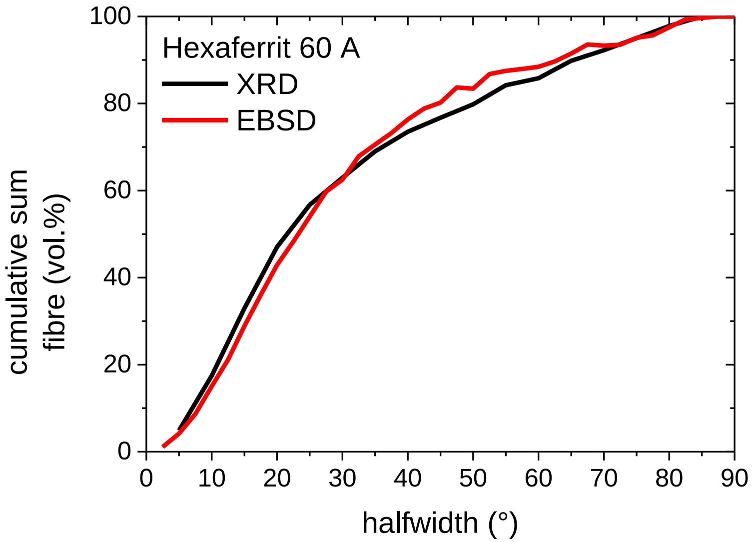

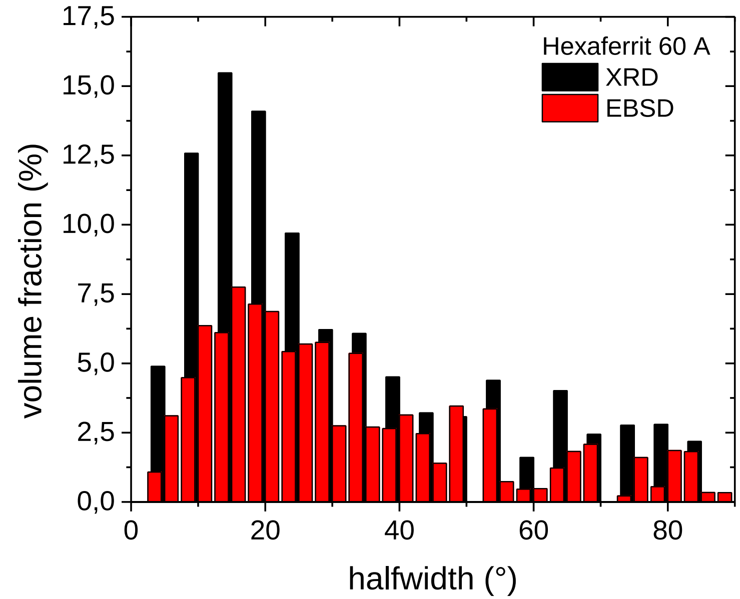

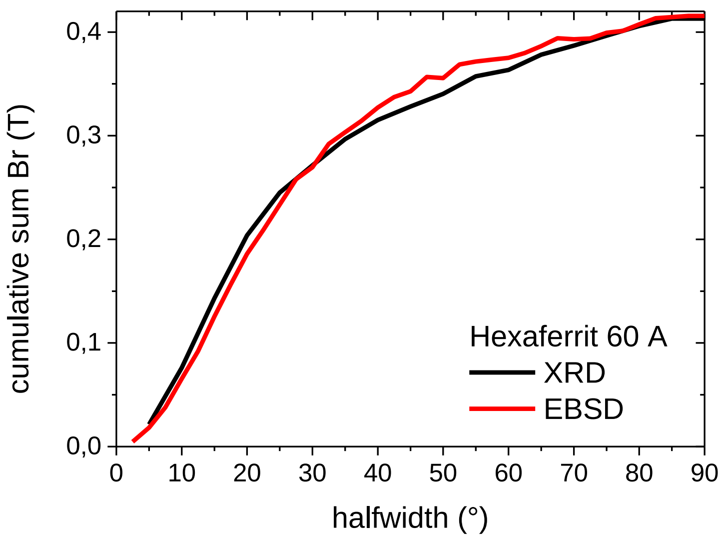

B_r (XRD) = 0.413 T
B_r (EBSD) = 0.416 T

Measured Br:
Br (Robograph) = 0.405 T

TRIDELTA Hartferrite GmbH

6. outlook

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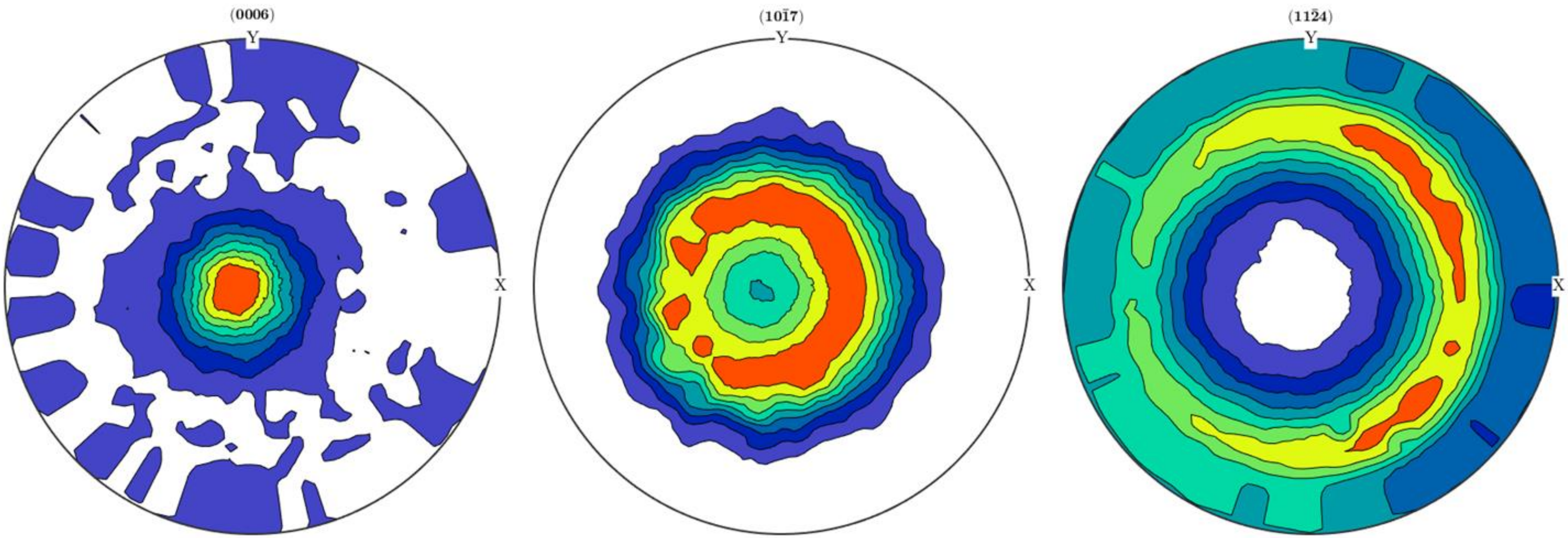
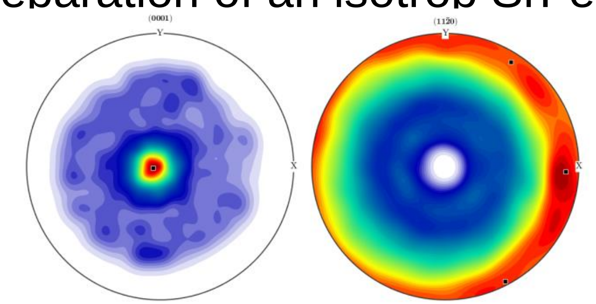
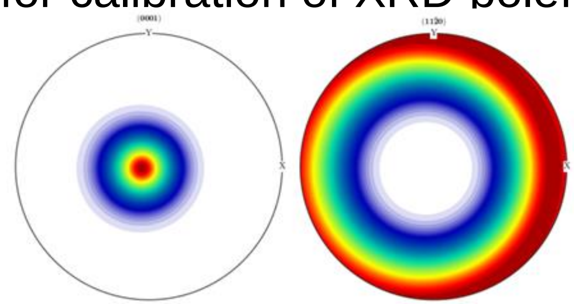
XRD Pole figures of 70 A sample



- measuring of a sample with c in z direction with XRD and EBSD
- use of Co-radiation instead of Cu-radiation
- preparation of an isotrop $SrFe_{12}O_{19}$ sample for calibration of XRD polefigures





6. outlook

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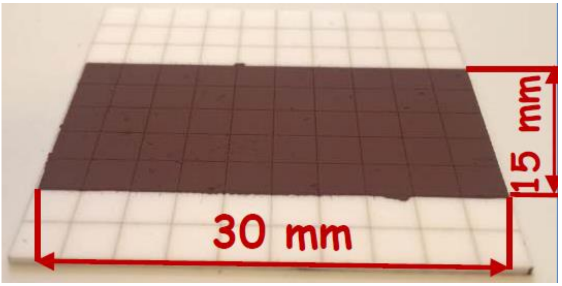
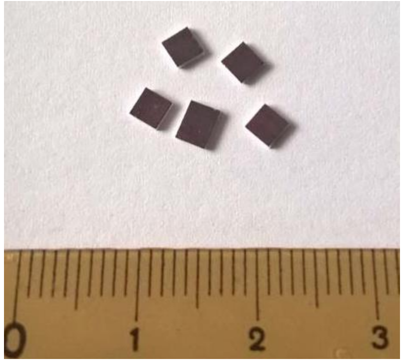
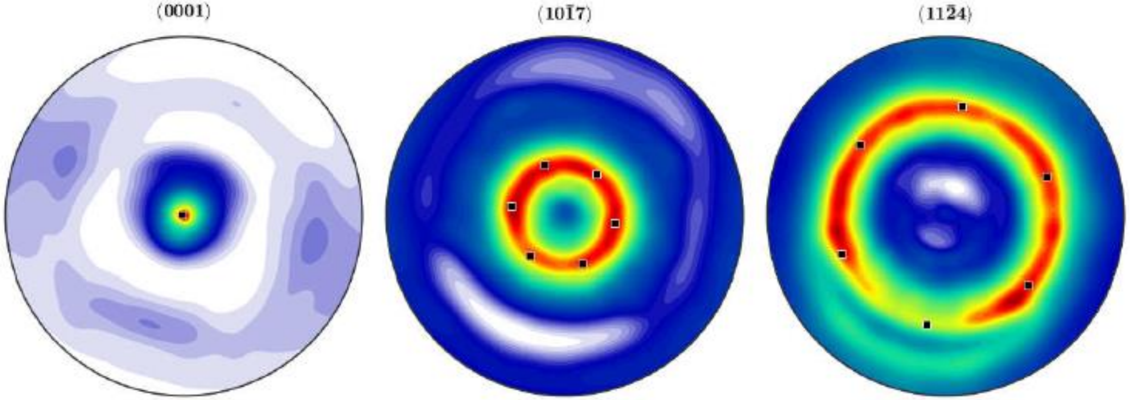
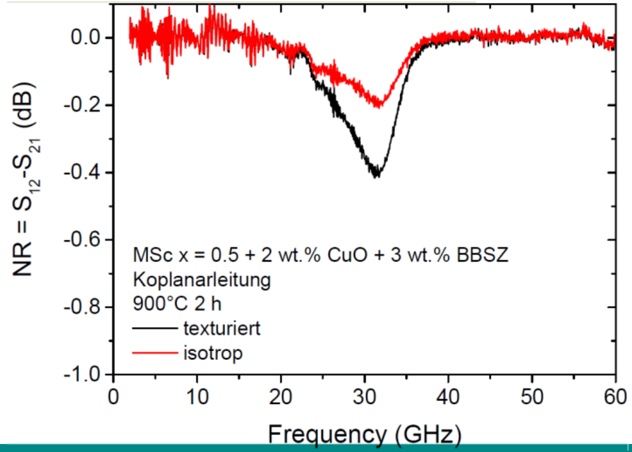
- Characterisation of screen printed thick film hexaferrites for circulators
- Characterisation of KNN piezoelectics

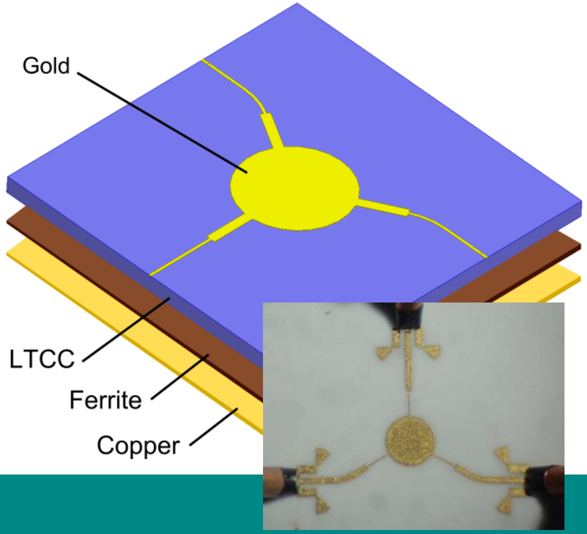








15 mm



Thanks!

Script for MTEX Fit

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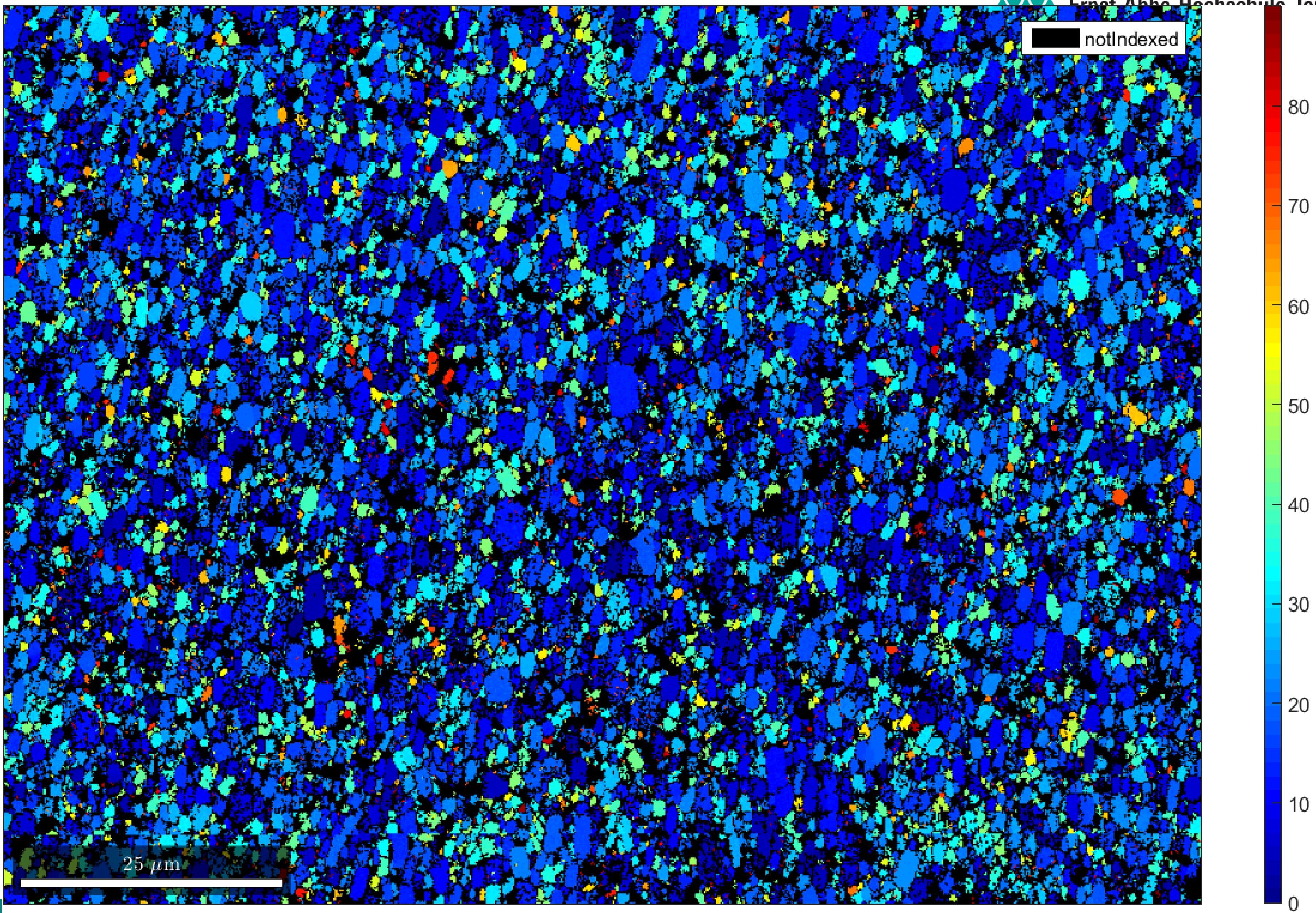
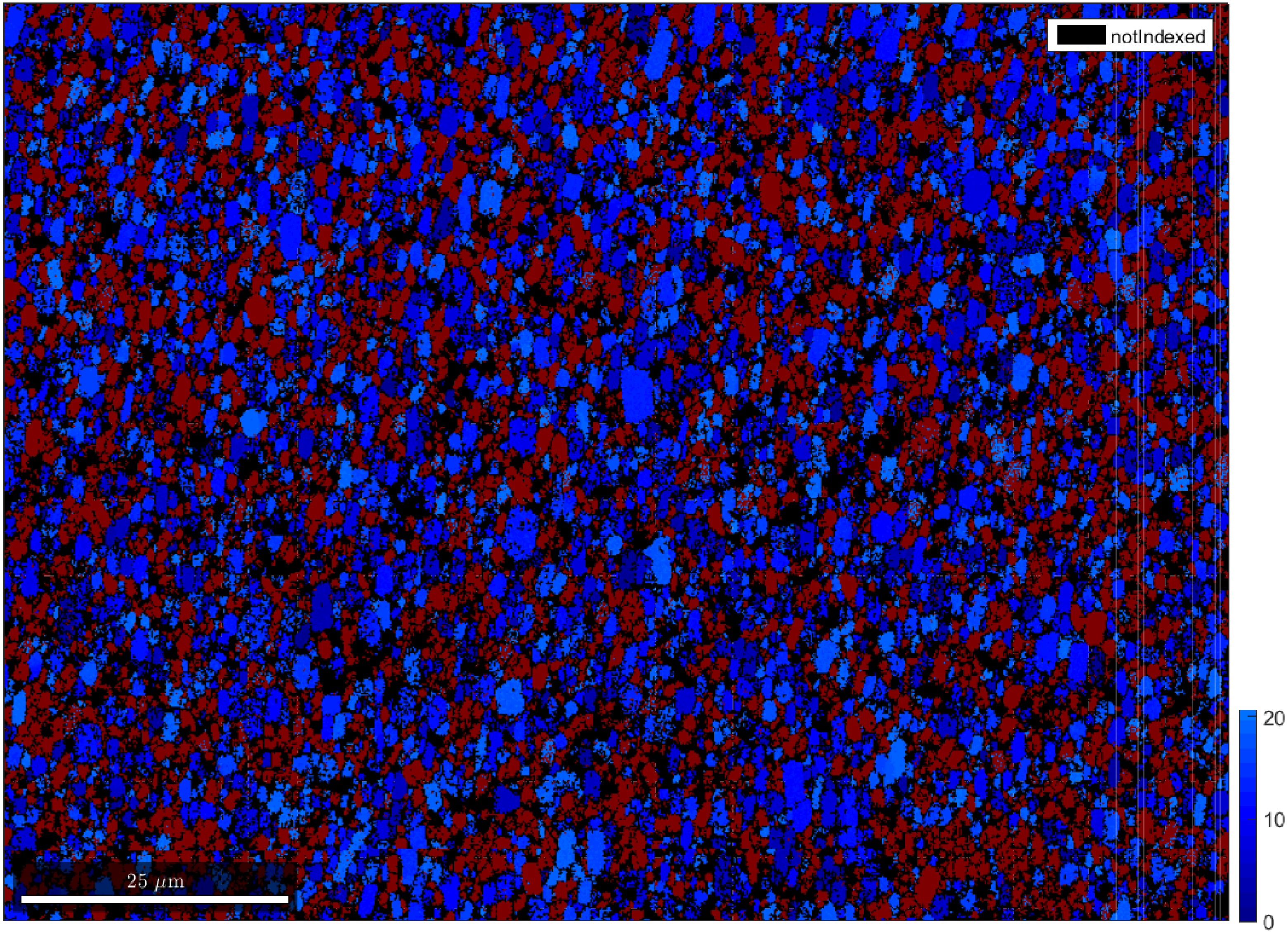
```
%% Fitten Fibre
Miller_c_Achse = Miller (0 ,0 , 1 ,cs);
fibrevector = odf_max_orientation*Miller_c_Achse;
odf_fibre = fibreODF(Miller(0,0,1,cs),fibrevector,'halfwidth',20*degree);

% definition Anfangsvektor, x(1): Amplituden; x(2), x(3), x(4): Eulerwinkel (°), x(5): Halfwidth unimodale ODF
[h1, h2, h3] = Euler(odf_max_orientation,'Bunge');
x0 = [0 h1/degree h2/degree h3/degree 10];
% Definition Nebenbedingungen: 0<= x(1) <= 1; 0<= x(5) <= 45;
A = [-1 0 0 0 0; ...
    1 0 0 0 0; ...
    0 0 0 0 -1; ...
    0 0 0 0 1];
b = [0;1;0;45];
min_func = @(x)calc_ODF_Error(x,odf_measured, ss,cs);
[x,fval,exitflag,output] = fmincon(min_func,x0,A,b,[],[],[],[],[],optimset('Algorithm','interior-point','Display','iter-
detailed'));
o_min = orientation('Euler',x(2)*degree,x(3)*degree,x(4)*degree,cs,ss);
o_min_vector = o_min*Miller_c_Achse;
ampl_min = x(1);
halfwidth_min = x(5);
odf_min = ampl_min*fibreODF(Miller(0,0,1,cs), o_min_vector,'halfwidth',halfwidth_min*degree) + (1-
ampl_min)*uniformODF(cs,ss);
```


notIndexed
25 μm
80
70
60
50
40
30
20
10
0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25 µm
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