BAM

Bundesanstalt für Materialforschung und -prüfung

Sicherheit in Technik und Chemie

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About the reliability of EBSD measurements: Data enhancement

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Outline

1. Introduction
2. Why we are using pattern matching?
3. Some selected examples

First off: *All maps shown are original data without any data manipulation like so-called "data cleaning", smoothing, filtering etc.*

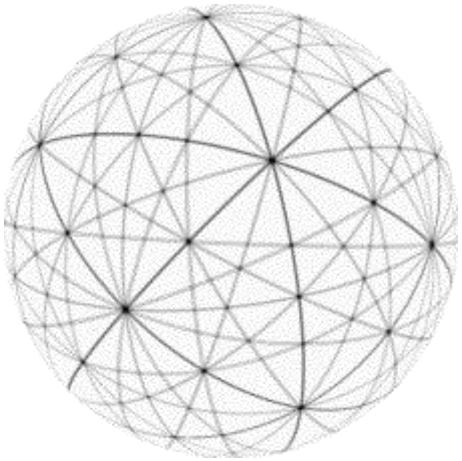
All maps have been derived from patterns with a size of 160x115 pixels only! ⇨ *1 pixel ≈ 0.5×0.5°*

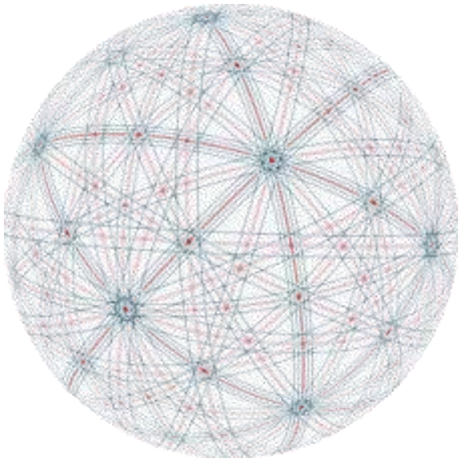
Traditional EBSD

- For phase and orientation determination **a pure geometric approach** based on the angles between a few crystal lattice planes only is used (neither band width, nor intensity).
- Alternatively, a slower algorithm can be used considering the same number of geometrically given **reflection widths** (but still no intensity).
- No crystallography at all during band detection in experimental patterns!

On the other hand:

*For decades dynamical theory of electron **diffraction** enables an **intensity simulation** which **gives access to position, width** & **intensity of "bands"**.*





Some (present!) limitations

Poor band detection and ignorance of intensity have consequences:

- **rudimentary** crystallographic **phase recognition**
  (many phases generate similar "reflections" from the same lattice planes, i.e. "bands" are unexploited)
- **no** real **phase identification**; ⇨ only verification
  *(present systems are **little sensitive to** lattice parameter **ratios only**, but not to lattice parameters itself)*
- **no** automated recognition or **processing of pseudosymmetry**
  (e.g. SiC, Fe_3O_4/FeO, FeS_2, perovskite, superconductors,…)
- Dilemma: **all phases** without inversion center are assumed to **deliver a centrosymmetric signal** (Friedel's rule).
- **systemically reduced** orientation **precision**
  *(a) caused by a mainly **speed-driven "band" detection***
  (b) caused by quite non-crystallographic indexing approaches

...although all tools are available!
Benefits of a good pattern simulation

A physics-based **simulation** has certain **advantages**:

1. An educated **guess about** the **formation** of BKD patterns.
2. Enables a **prior validation of** unknown **phases** regarding the
   - used **indexing approach** (*e.g. number of reflectors, band number searched, phase-specific features*),
   - **discriminability** of phases,
   - possible **pseudosymmetries**, or
   - lattice (parameter) **deviations** of different phase descriptions.
3. An **optimization of** the **measurement setup**, e.g. in combination with a characteristic noise distribution evaluation of the minimally required signal-to-noise ratio.
4. Offers a **maximum** achievable **calibration precision**.
5. Pattern matching still delivers **solutions where traditional EBSD fails**.

Why pattern matching?

Pattern processing, band detection, indexing, phase discrimination **etc. are corporate secrets**!

- We do not have **a** real **chance to evaluate** extracted **orientation or phase data**.
- We (often) do **no**t get any **trustworthy error information**.
- Only if we "know" the exact result, e.g. by other techniques, **we** can **try to explain** or speculate about suspicious deviations, **but** even then **we do not** really **know the reason(s)**.
- Solution: We simply **bypass** this **entire processing**, use the patterns **and** apply/**develop** own and hopefully **better approaches**.

  One of these approaches is **pattern matching**, a simple correlation between two images.

Pattern matching procedure
(for orientation refinement)

Start with a (roughly) **determined** crystal **orientation**
by Hough-related band detection and standard Laue group indexing, or brute force matching

Generation of **simulated patterns in real-time** (incl. all pseudosymmetric solutions)
using a template, model, or master) pattern

Pattern matching
between simulation and experiment

slight **orientation variation**

When pattern matching (PM) is useful?

Practically always then **when conventional indexing**

1) …**does not work** properly.
   - <u>theoretical:</u> Unsuitable reflector selection
     (*intensity approximation by the applied theory is too erroneous for the applied ranking*).
   - <u>experimental:</u> Insufficient signal quality for a reliable reflector detection
     (dictionary approach can help).

2) …**cannot work** properly.
   - Non-supported symmetry
     (Friedel's rule, unknown phase, quasicrystals…)

3) …**is insufficient** if orientation precision needs to be higher,
   e.g. for a reasonable estimation of the dislocation density.

Applications of PM in the past

Keyword: Pseudosymmetry

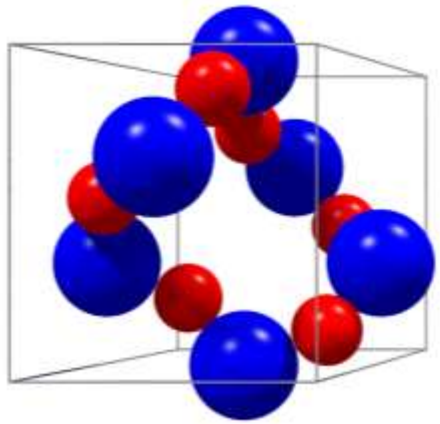
1. Crystal **structure-related** phenomena
    - Discrimination of **left- and right-handed varieties** (*of different (quartz) or identical (tartaric acid) space-group types*)

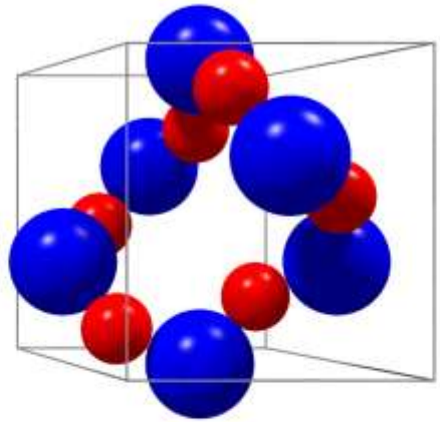
Quartz

Here slightly higher resolved patterns!

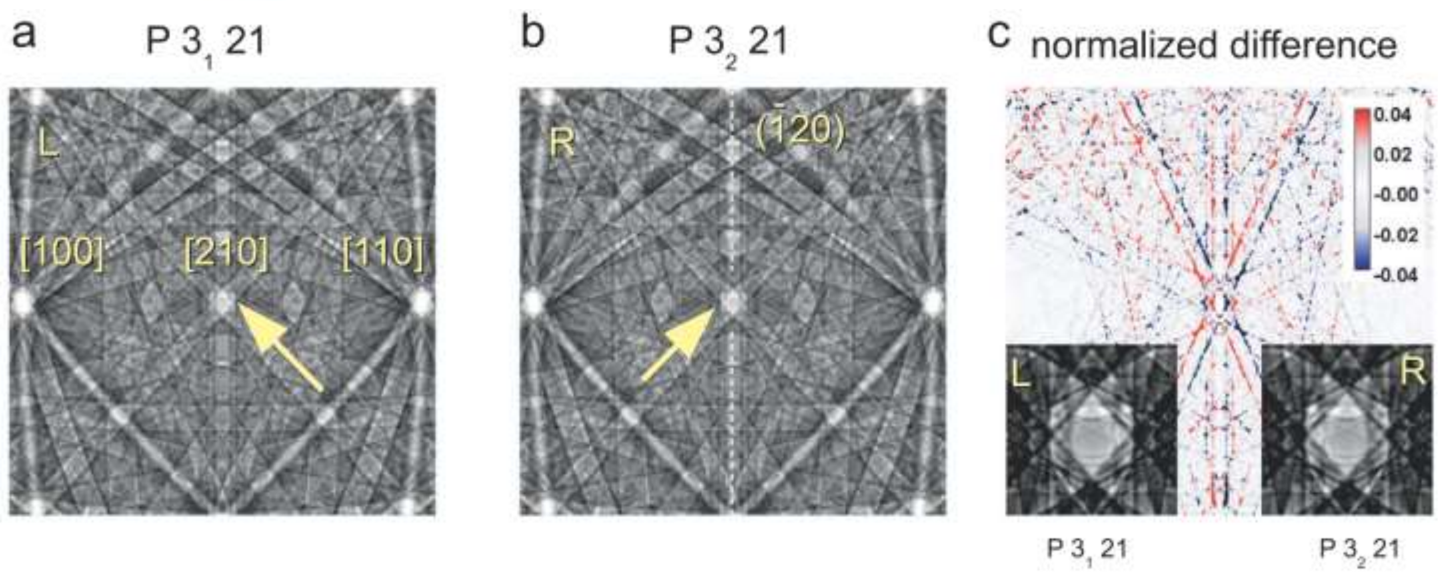
Winkelmann, A. & Nolze, G.
Chirality determination of quartz crystals using Electron Backscatter Diffraction. *Ultramicroscopy*, **2015**, *149*, 58-63

$P\,3_1 21\ (152)$





$P\,3_2 21\ (154)$



Applications of PM in the past

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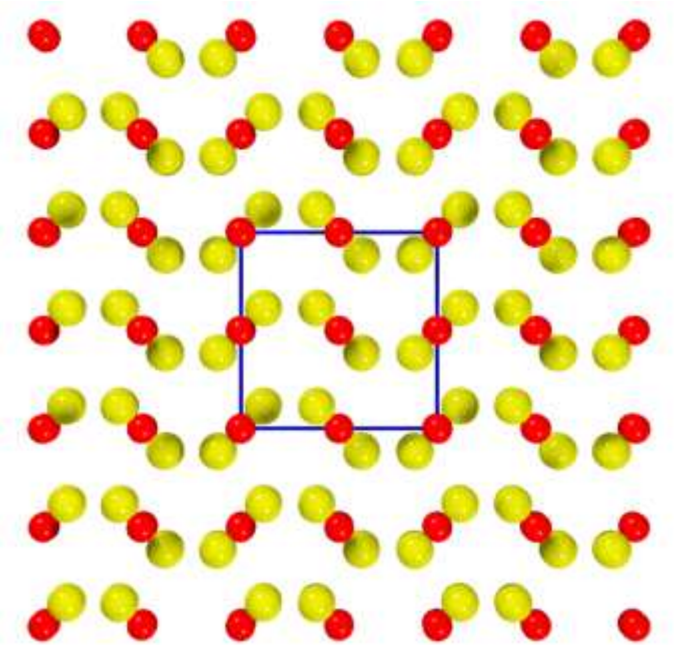
1. Crystal **structure-related** phenomena
   - Discrimination of **left- and right-handed varieties** (*of different (quartz) or identical (tartaric acid) space-group types*)
   - **Prevention of misindexing** in a higher-symmetric lattice (*centrosymmetric pyrite – FeS_2 – cubic but no 4-fold axis*)

Impact of low-symmetric Laue groups
Pyrite

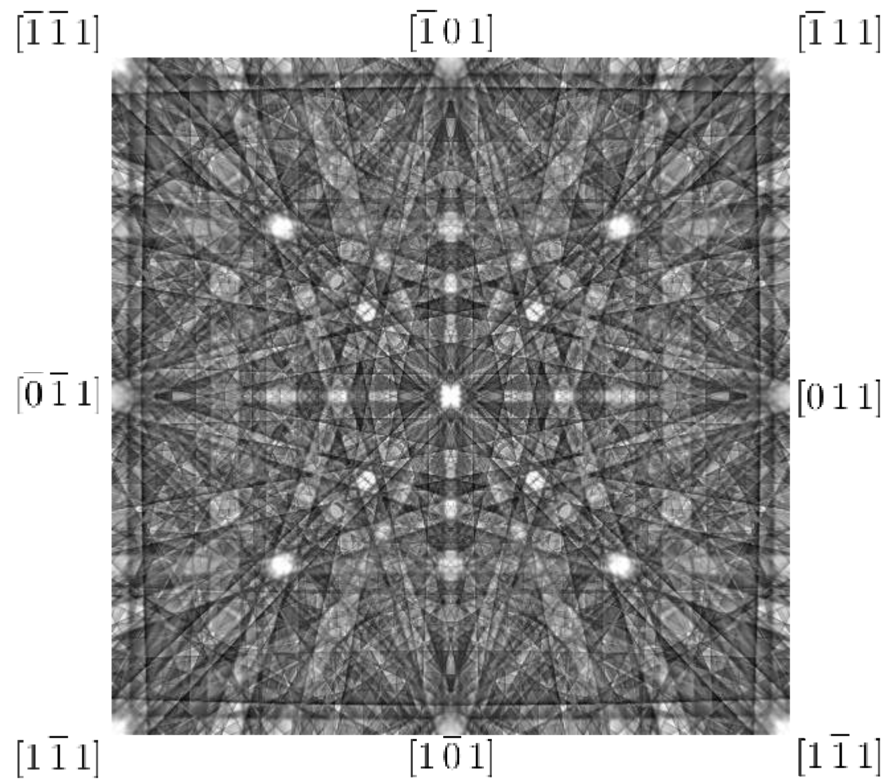
G. Nolze, A. Winkelmann, A. P. Boyle: *Pattern matching approach to pseudosymmetry problems in electron backscatter diffraction*. Ultramicroscopy, **2016**, *160*, 146-154



$P\,2_1/a\,\bar{3}$

Fe	$4a$	$0, 0, 0$
S	$4c$	x, x, x

- Pseudosymmetry means:
  A pattern **looks higher-symmetric than it is**!



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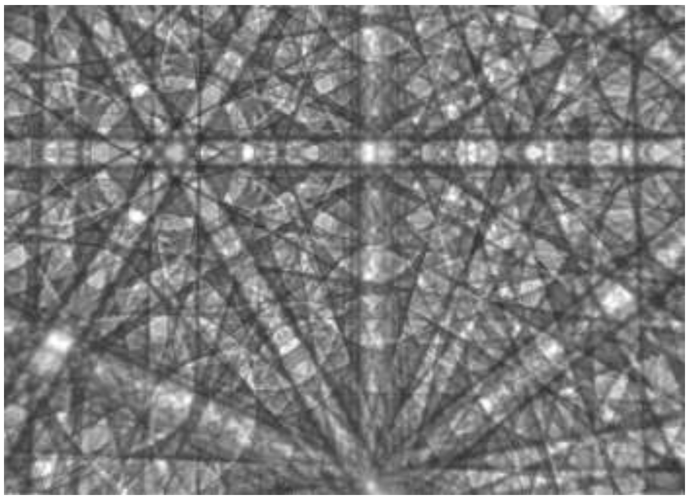
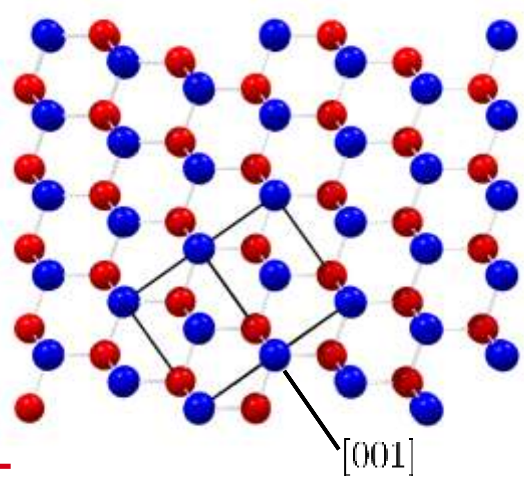
1. Crystal **structure-related** phenomena

- Discrimination of **left- and right-handed varieties**
  (*of different (quartz) or identical (tartaric acid) space-group types*)
- **Prevention of misindexing** in a higher-symmetric lattice
  (*centrosymmetric pyrite – FeS_2 – cubic but no 4-fold axis*)
- **Differentiation between polar directions** or planes
  (*in **non**-centrosymmetric phases, e.g. semiconductor compounds*)

Non-centrosymmetry in GaP

Winkelmann, A. & Nolze, G.; *Point-group sensitive orientation mapping of non-centrosymmetric crystals* Appl. Phys. Lett., **2015**, *106*, 072101





- A **90° rotation around [001]** does not change the unit cell, but **only varies** the **P** atoms **positions** (red colored).
- According to the switch of P-atoms the **pattern partially changes** as well.
- Some bands
  - **switch** the **intensity**,
  - **seem to move**, and
  - **do not vary** at all.
- We have **polar & non-polar** planes in the same phase, **at the same time**…

Applications of PM in the past

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   - Discrimination of **left- and right-handed varieties** (*of different (quartz) or identical (tartaric acid) space-group types*)
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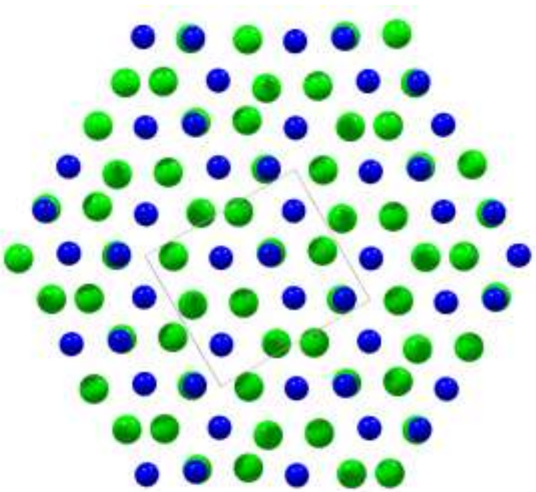
   - **Differentiation between polar directions** or planes (*in non-centrosymmetric phases, e.g. semiconductor compounds*)

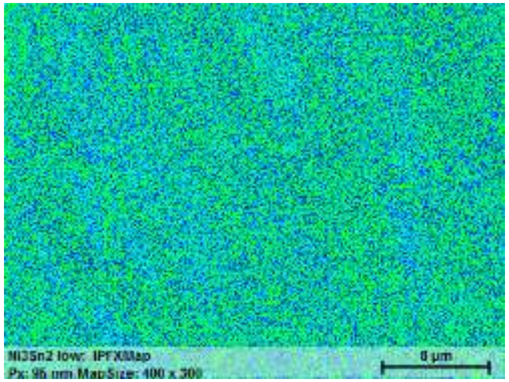
2. Crystal **lattice-related** phenomena
   - **Prevention of misindexing** caused by lattice transformations *(e.g. hexagonal ⇨ orthorhombic (Ni_3Sn_2), or any other sublattice description with similar structure)*

Ni_3Sn_2
hexagonal ⇨ orthorhombic

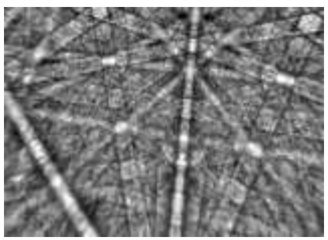
A. Leineweber, TU Freiberg



FSE image (EBSD patterns)



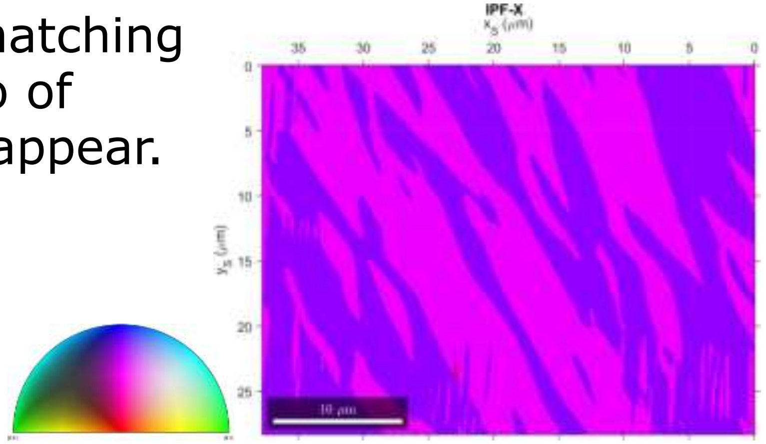
IPF map of the RT phase (orthorhombic, mmm).



The crystal structure is **pseudosymmetric**.

After 120° rotation around [010] both **images** are **not identical** but very similar.

After pattern matching in this map two of three variants appear.



Applications of PM in the past

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1. Crystal **structure-related** phenomena
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2. Crystal **lattice-related** phenomena
   - **Prevention of misindexing** caused by lattice transformations
     (e.g. hexagonal ⇨ orthorhombic (Ni_3Sn_2), or any other sublattice description with similar structure)
   - **Orientation precision**
     (relevant for all phases!)

How to reduce the orientation error?

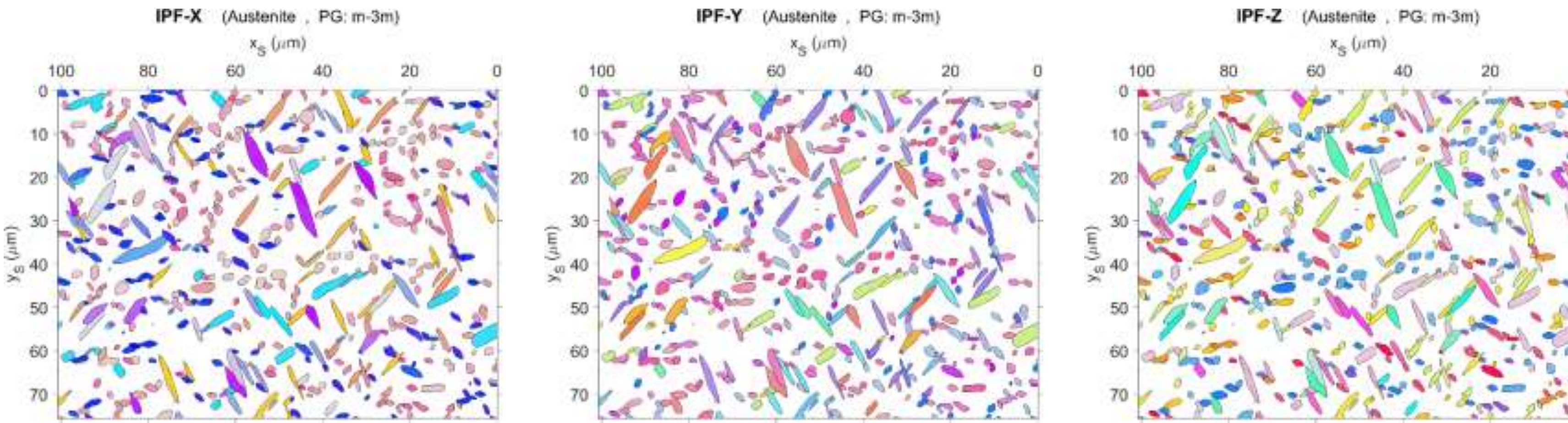
- **For** any **minimization of the** (unknown) orientation **error**, or a discrimination of pseudosymmetric orientations or phases some apparently **obvious solutions** exist:

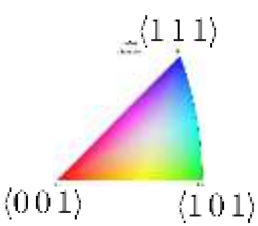
  1. use of the **highest** possible **pattern resolution** and the longest distance between sample and detector
  2. most **precise** knowledge about the **projection center position** *(pattern center & detector-sample distance)*
  3. the **best** acceptable **signal-to-noise ratio**
  4. most **precise band detection** *(e.g. optimum Hough resolution)*
  5. a **robust indexing** *(whatever that means…)*
  6. …

- At least points 1.-3. are **discussed** several times **for HR-EBSD**.
- **For small patterns** used **at** standard **orientation mapping**, however, the influence of most of these options is **quite vague**.

Duplex steel (γ-precipitates in α-matrix)
Orientation description (raw data)

Nolze, G. et al. *Improving the precision of orientation measurements from technical materials via EBSD pattern matching*. Acta Mater., **2018**, *159*, 408-415

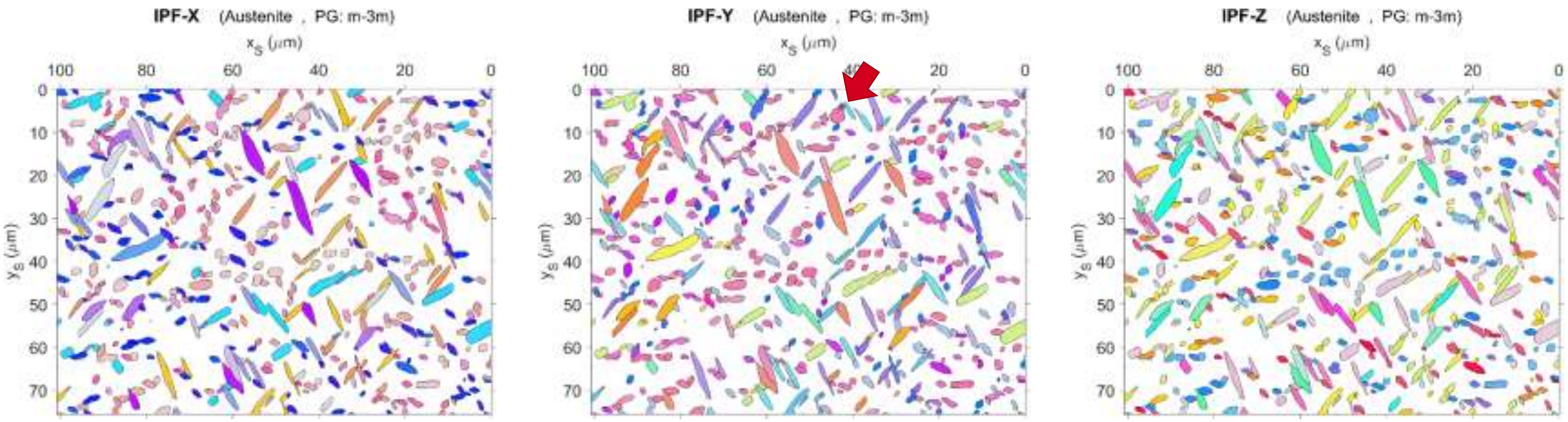


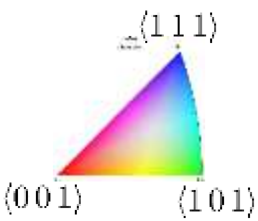


- **For orientation description** a typical **standard EBSD** is absolutely **sufficient**.

Duplex steel (γ-precipitates in α-matrix)
Orientation description (after PM)

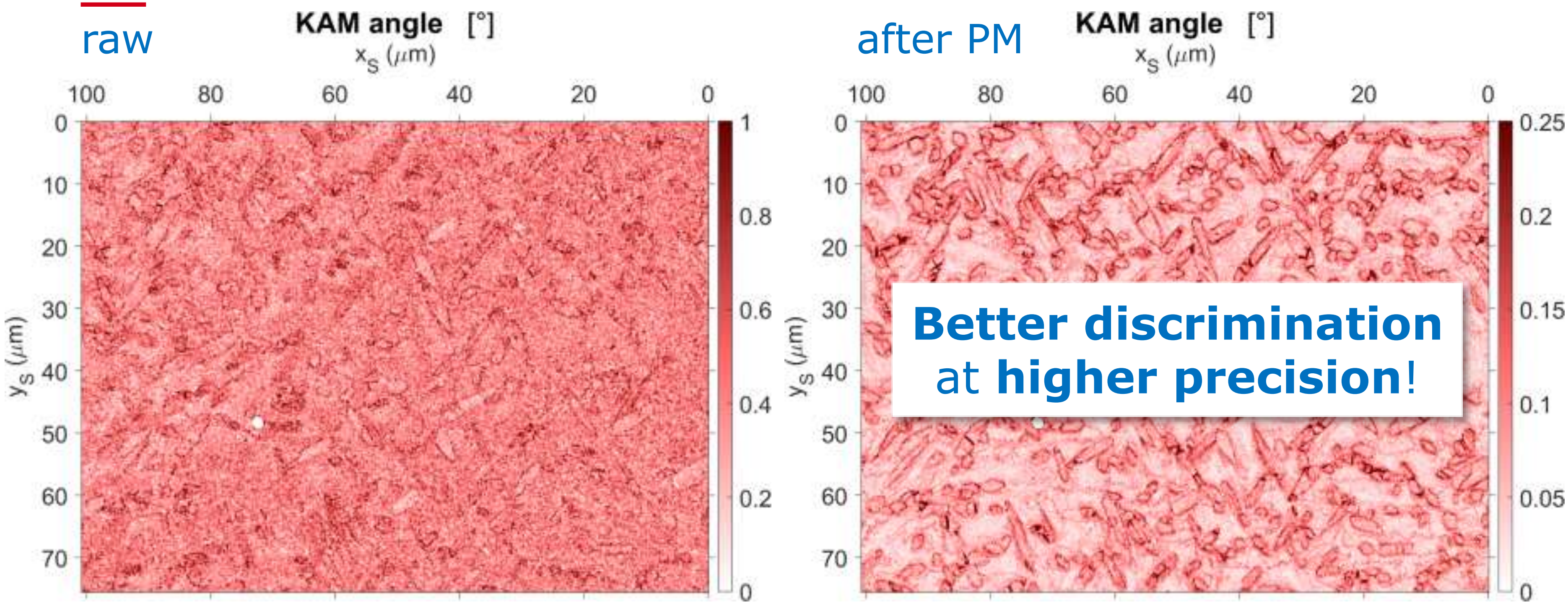
Nolze, G. et al. *Improving the precision of orientation measurements from technical materials via EBSD pattern matching*. Acta Mater., **2018**, *159*, 408-415





- **For orientation description** a typical **standard EBSD** is absolutely **sufficient**.
- **Limitations** in accuracy and precision are **only visible in direct comparison**, if at all.
- A **higher** orientation **precision** is **only** necessary **when misorientations** are **imaged**.

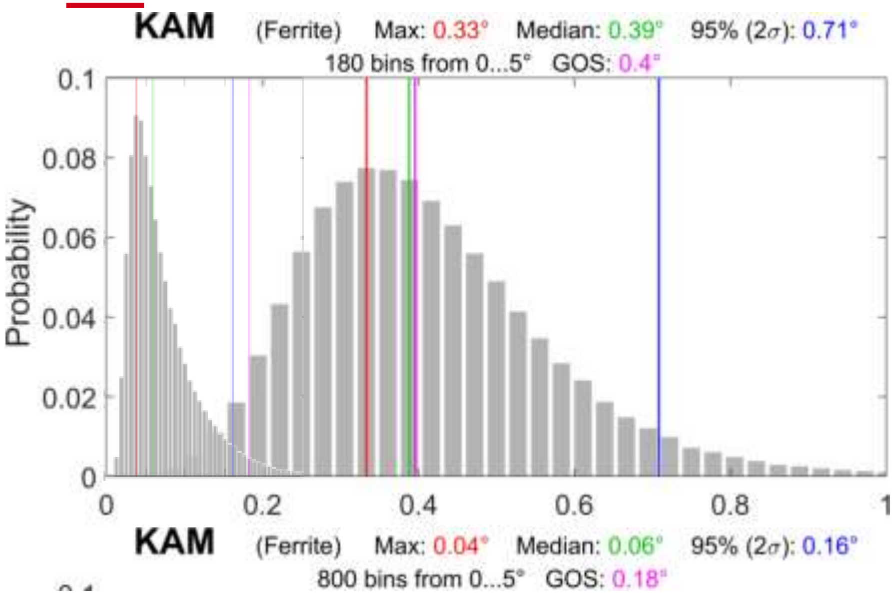
Duplex steel (γ-precipitates in α-matrix)
KAM angle

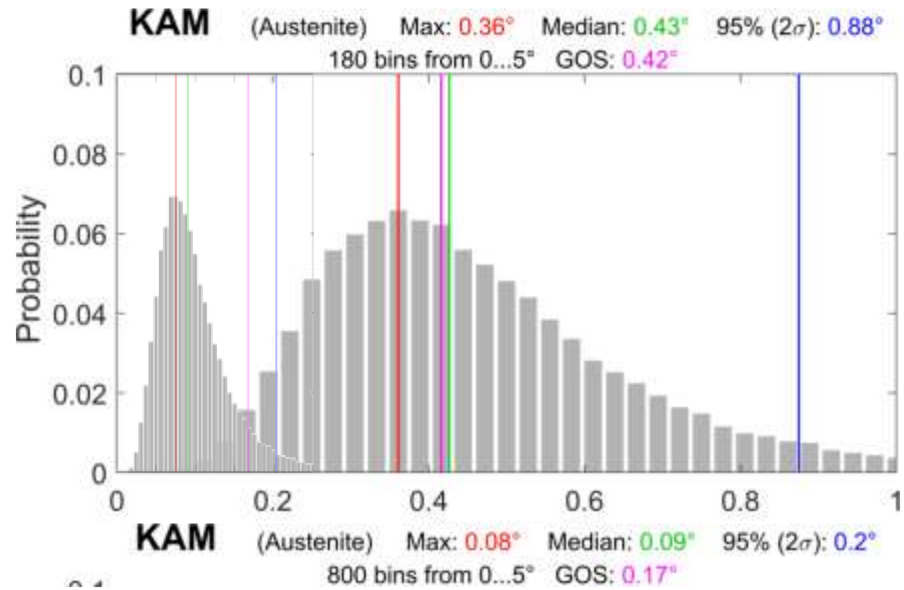


- **KAM** (kernel average misorientation) describes the **misorientation** between **local to adjacent measurement positions**.
- KAM **images** local **orientation gradients** and is therefore useful for **small lattice rotations** (e.g. subgrain boundaries etc.).

Duplex steel

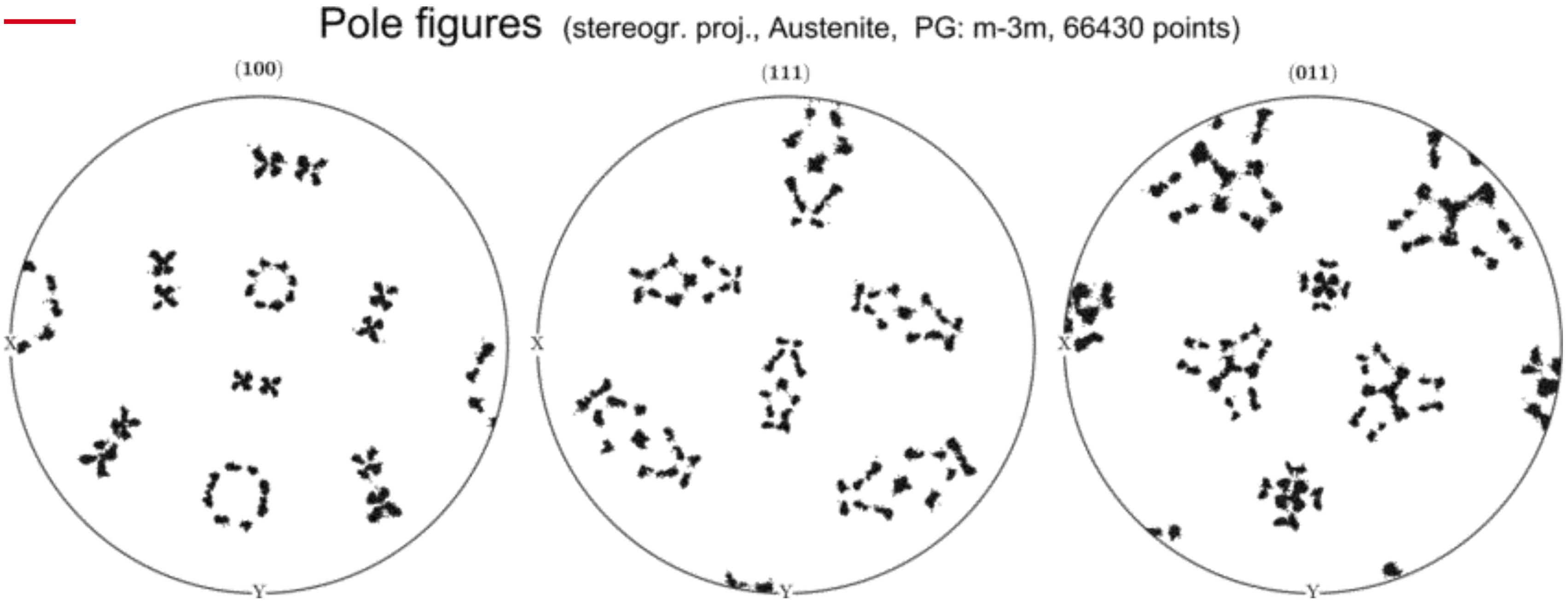
160x115 pixel only !





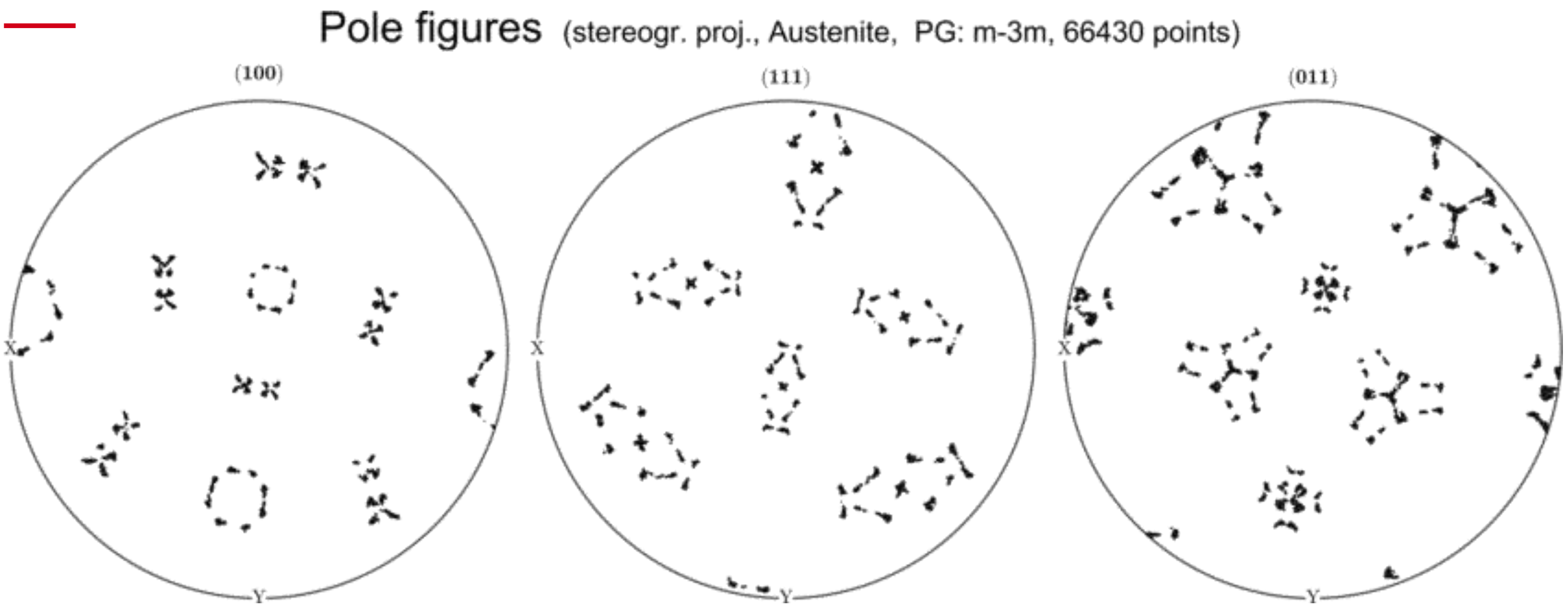
- KAM angle histograms display a **clear improvement** for both phases **during pattern matching**.
- **All characteristic** measures like maximum (8x), median (6x), or 2σ range (4x) **are considerably improved**.
- …although the grain orientation spread (GOS) has been halved only.

Pole figures (raw data)



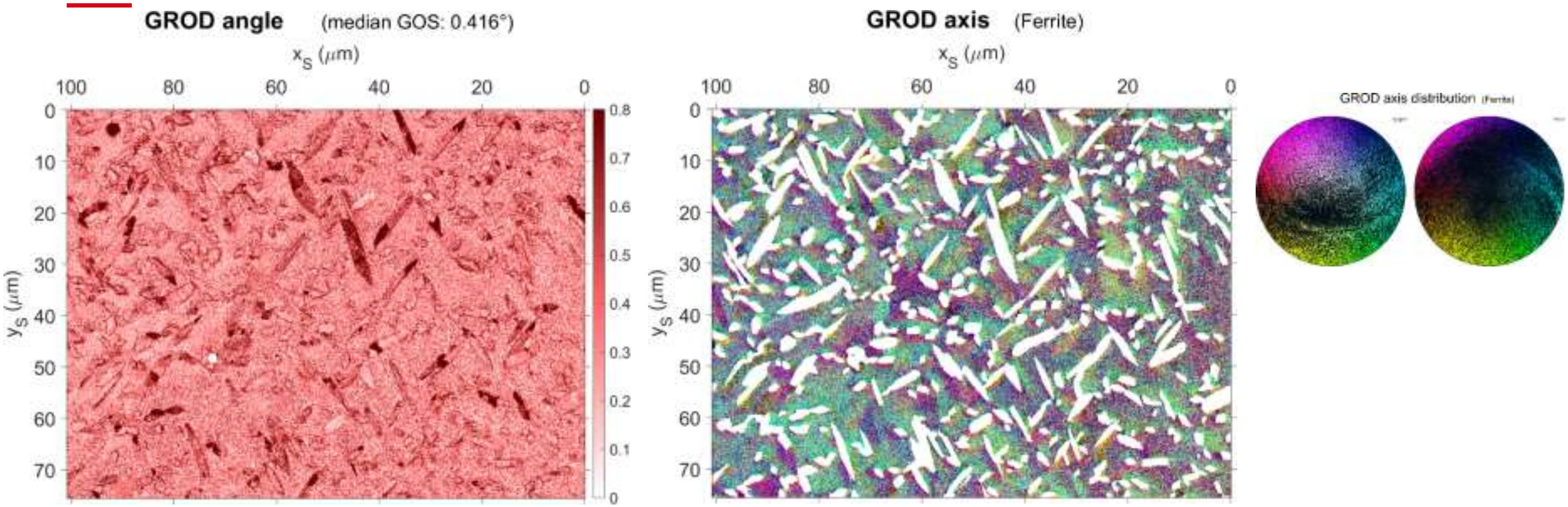
- The **grain orientation spread** (GOS) **effects** the pole distribution even in **pole figures**.

Pole figures (refined data)



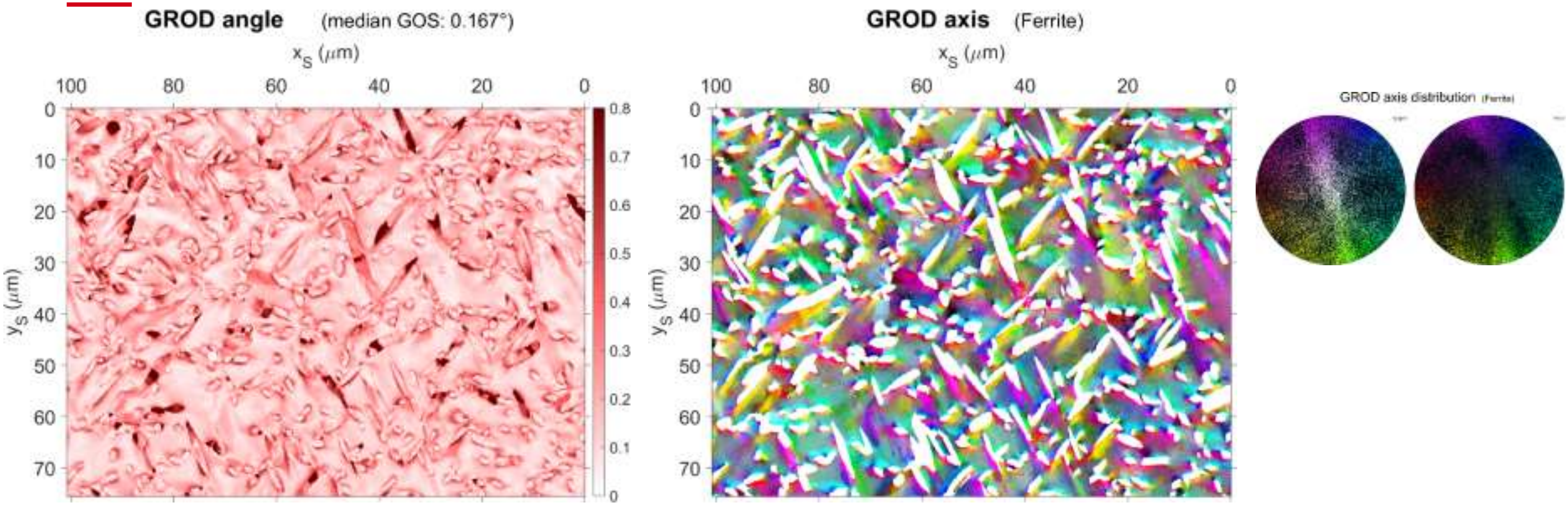
- The **grain orientation spread** (GOS) **effects** the pole distribution even in **pole figures**.
- **Spread** is visibly **reduced** which improves any quantification.
- <u>Please note:</u> **Orientations** after PM are **slightly misaligned** (very likely caused by a poor PC or a misused detector tilt).

GROD (grain reference orientation deviation) *raw data*



- GROD is the local **misorientation** (**MO**) with respect **to the mean orientation of a** map region previously defined as **grain**.
- A MO can be described by the smallest **MO angle rotated around** the vertical direction of the great circle called **MO axis**.

GROD (grain reference orientation deviation) *refined data*

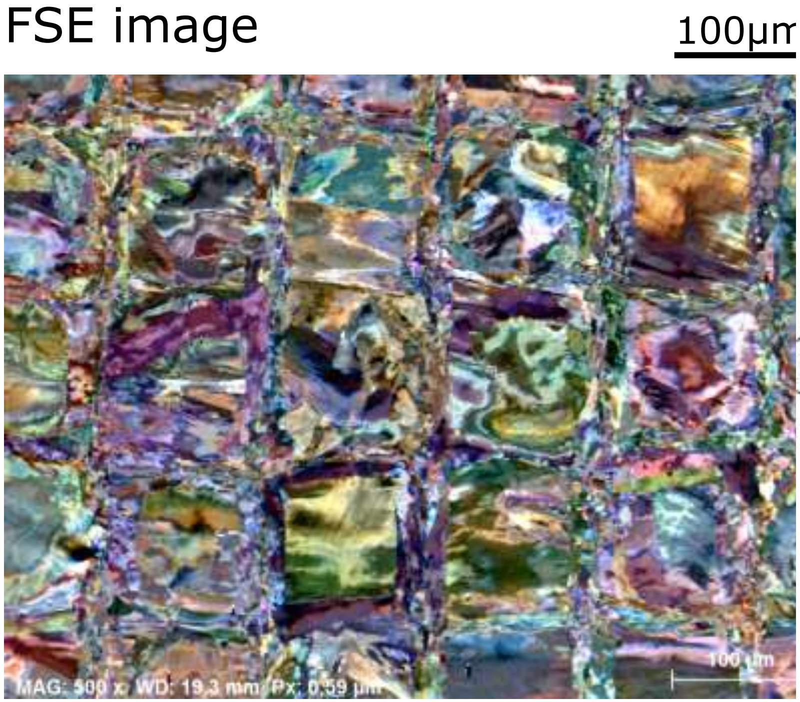


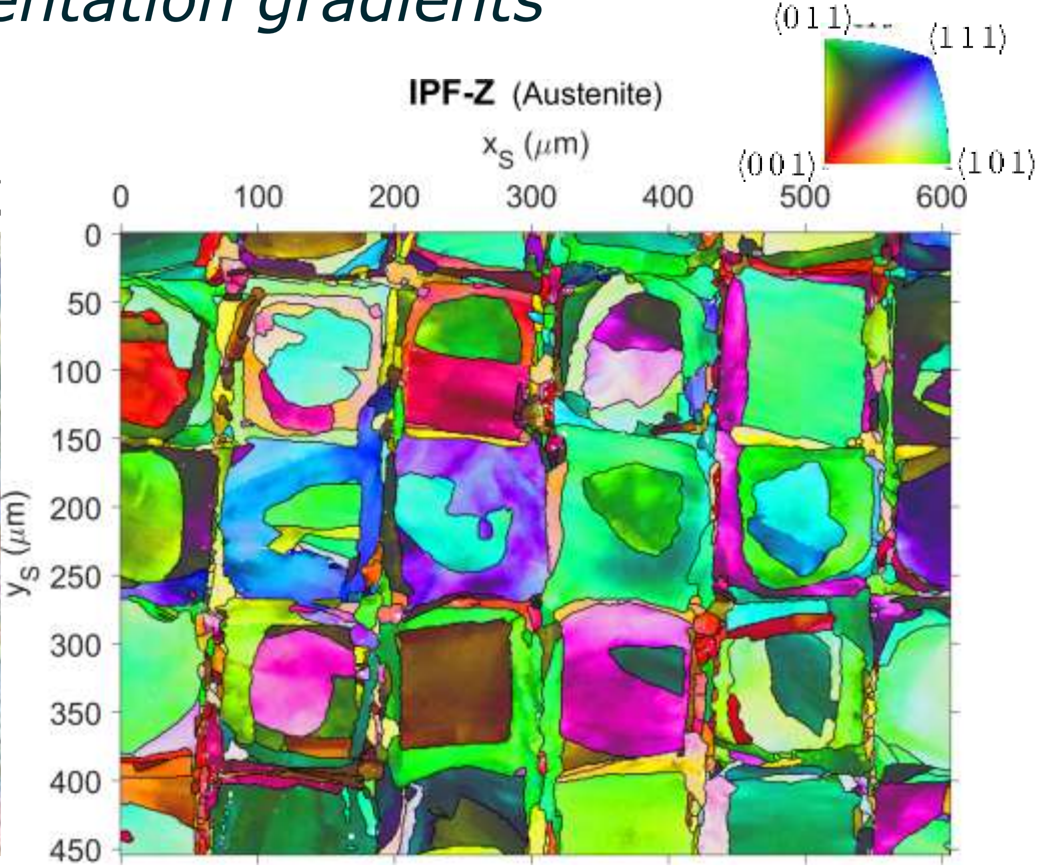
- GROD is the local **misorientation** (**MO**) with respect **to the mean orientation of a** map region previously defined as **grain**.
- A MO can be described by the smallest **MO angle rotated around** the vertical direction of the great circle called **MO axis**.
- Despite small MOs the maps between **raw and refined data** look qualitatively as well as quantitatively **quite different**.

Additive manufactured material

Materials of high misorientation gradients

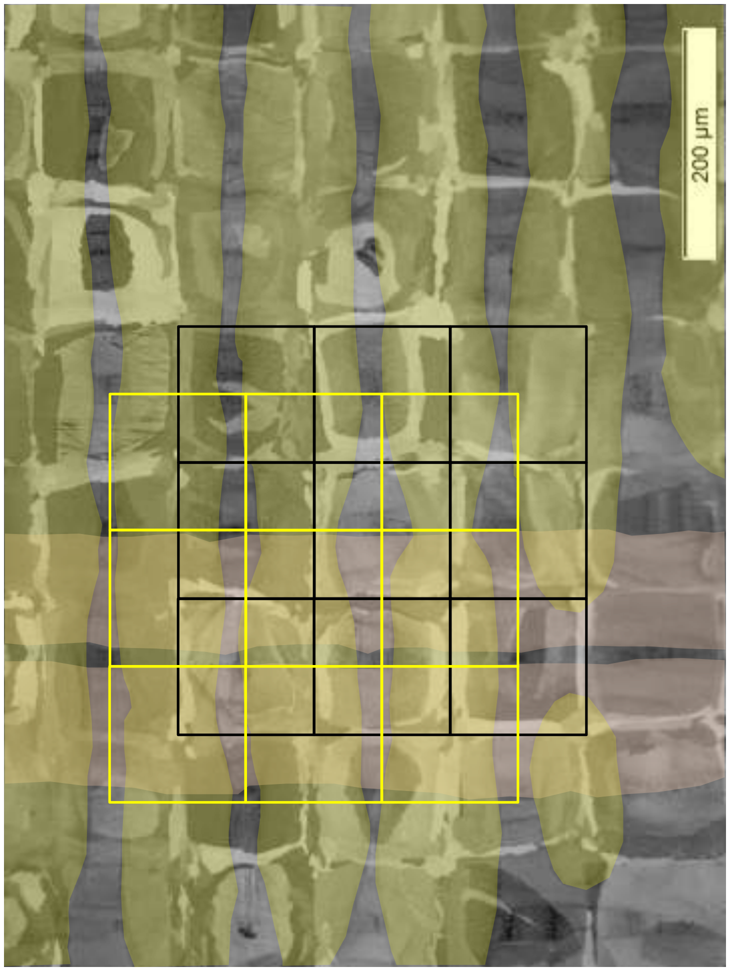
FSE image





- Undeniable: **Complex microstructures** with high potential!
- Caused by manufacturing **overlaid mesoscopic order**.
- **If big misorientations**: High orientation **precision required**?
- Can we see the bead interfaces?

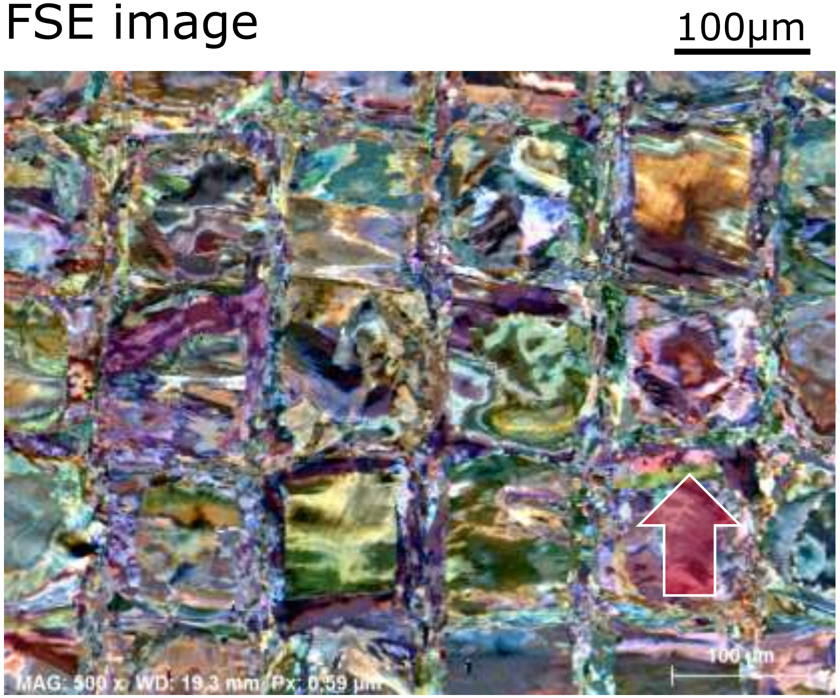
3D laser printing

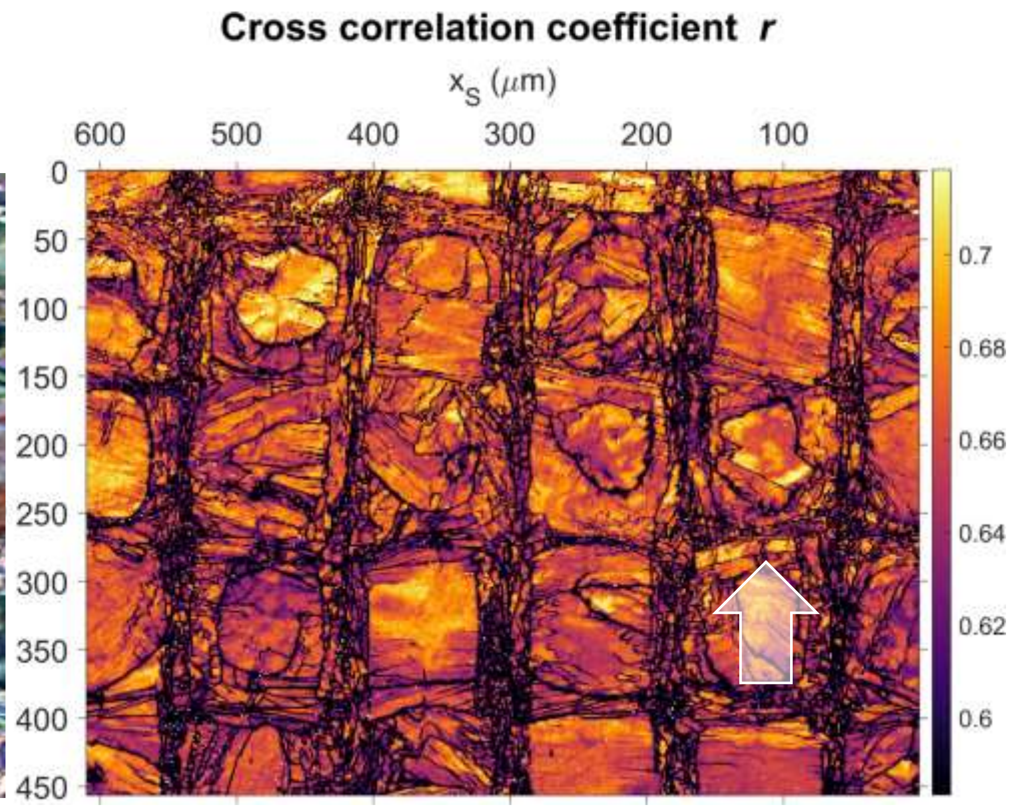


- "Strange" microstructure as result of the printing conditions (pathes).
- **Mainly visible** are here the **crystal** orientations **but also** remaining interfaces of the **weld beads**.
- Two **checkerboard-like** mesoscopic **structures** appears, one from the grain orientations and one from the weld beads **shifted** against each other **by [½, ½]**.
- The **visible**, aligned **grain boundaries mark** the **path of** the **laser beam**.
- **Fundamental questions arise**, e.g., how the **solidification** works, how the **grain size** can be defined, what such a value tells us, etc.?

Additive manufactured material

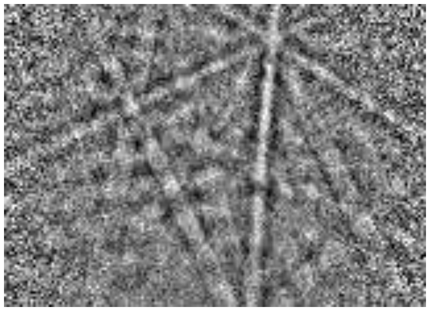
FSE image



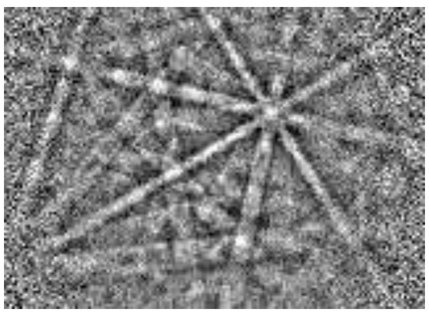


- Weak **indications for misorientations** in the FSE image are suggested **in the centres of the "squares"**.
- **Also** the cross correlation ***r*** **indicates lattice perturbations** along the interface **between two weld beads**.

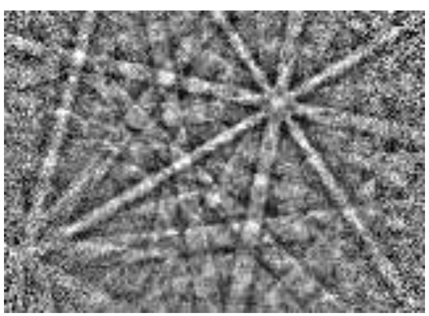
Required diffraction signal quality



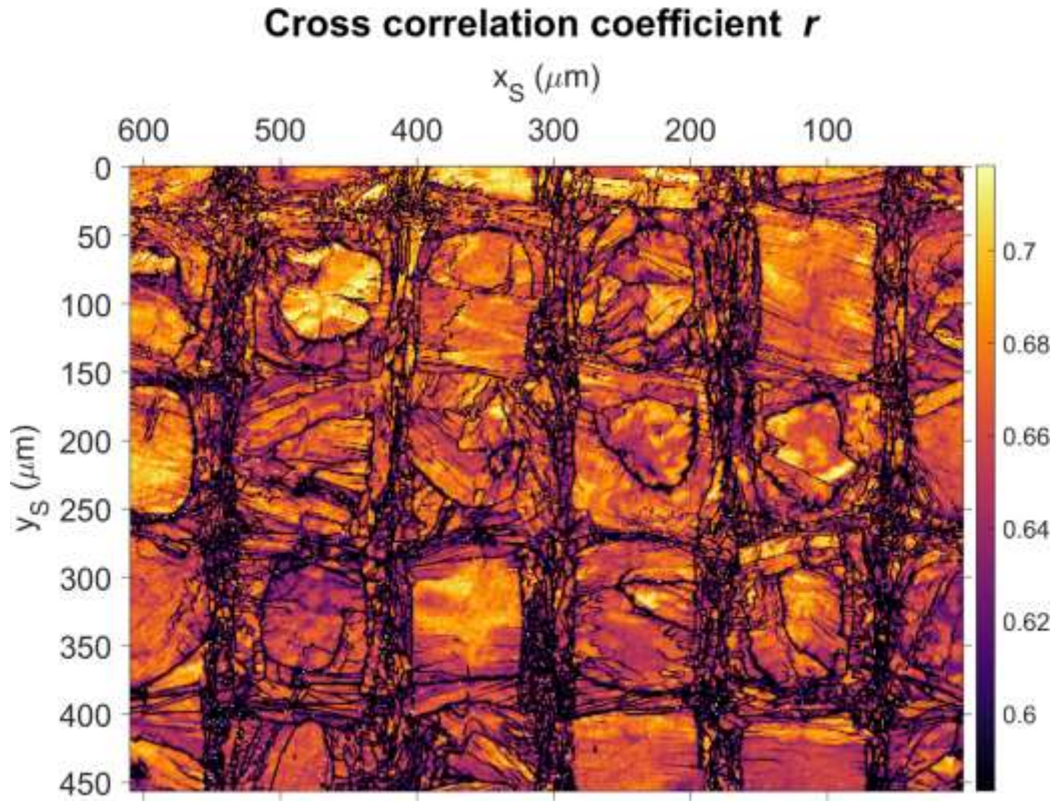
$r = 0.5$



$r = 0.6$



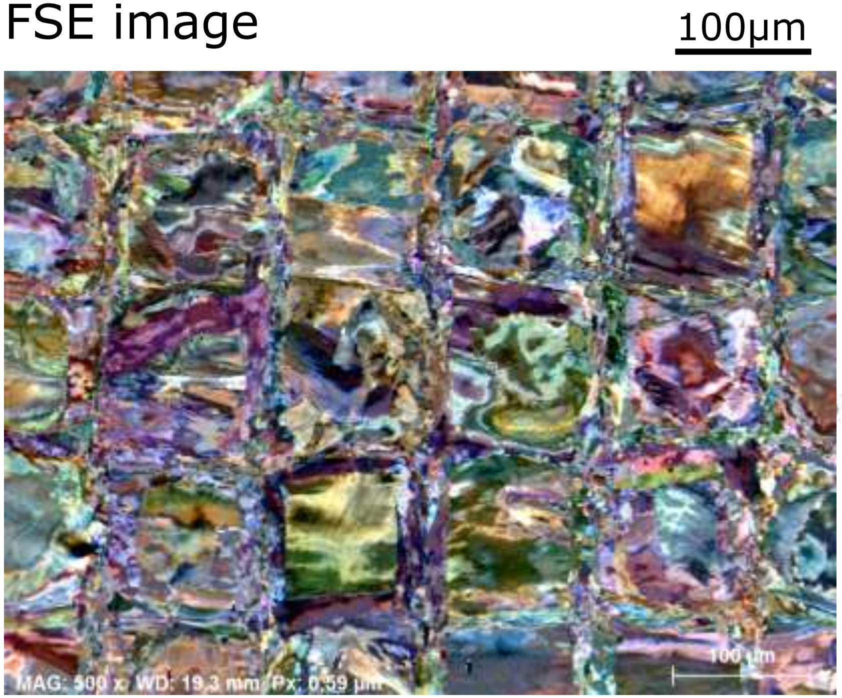
$r = 0.7$

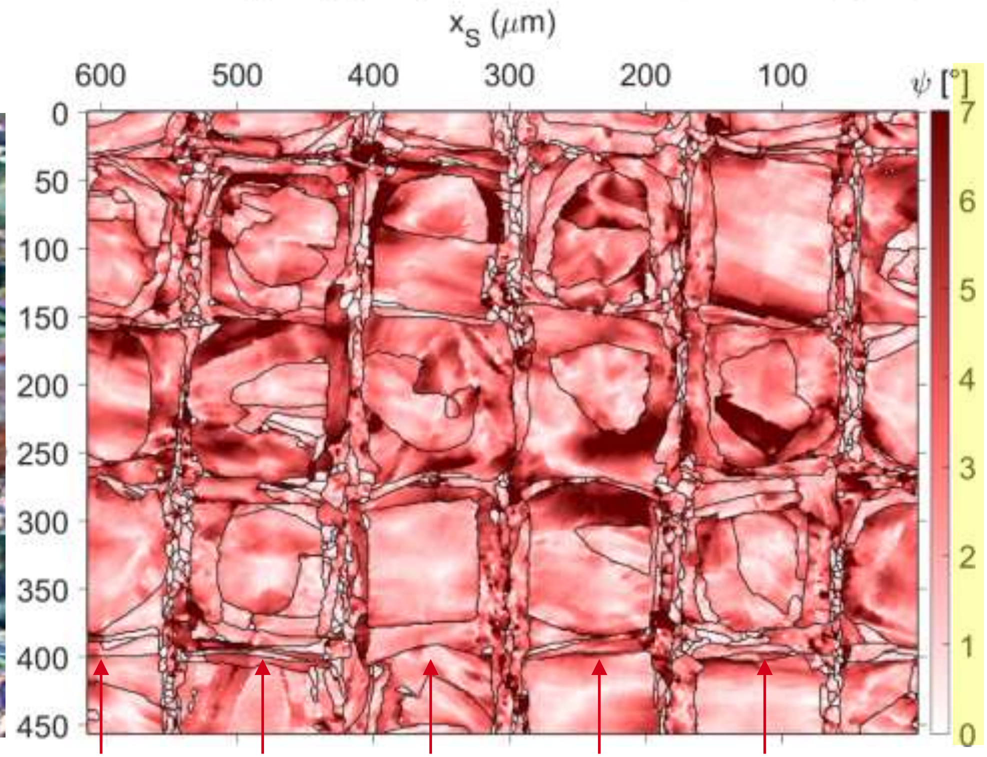


- In order to get a visual impression **which *r* represents which quality** of patterns, for some selected *r* typical patterns are given.

Additive manufactured material
Misorientation angles

FSE image



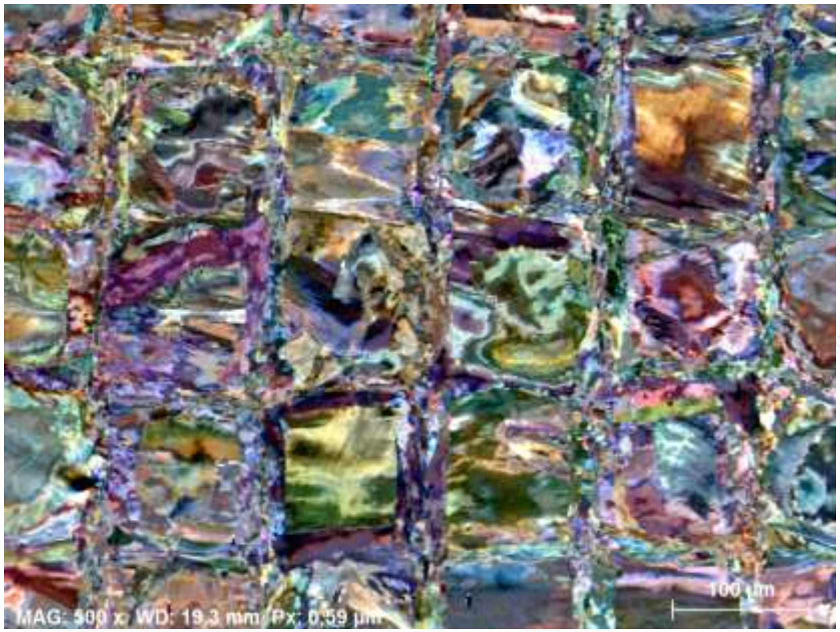


- Perturbations are also **partially visible** in GROD maps.
- Fast cooling during solidification causes **misorientation angles of several degrees** (grain boundaries in black).
- Colours in the FSE images are hard to interpret.

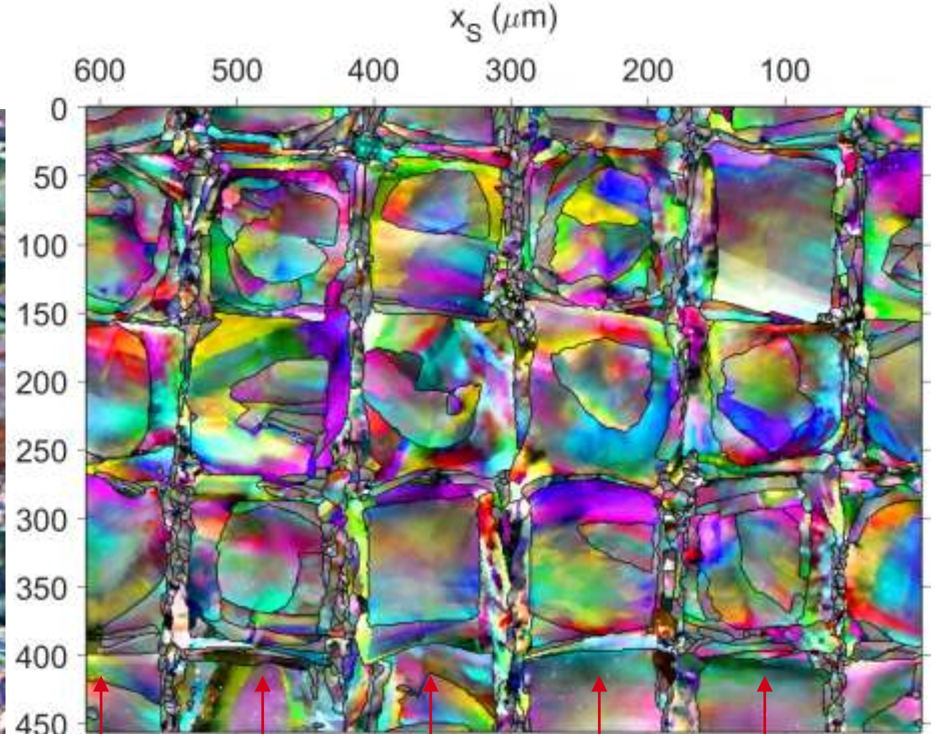
Additive manufactured material
Misorientation axes

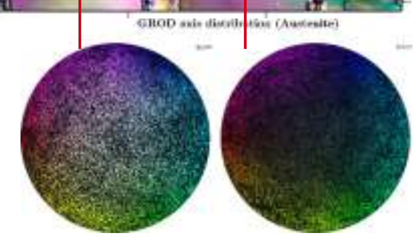
FSE image

100µm



GROD axis (Austenite)



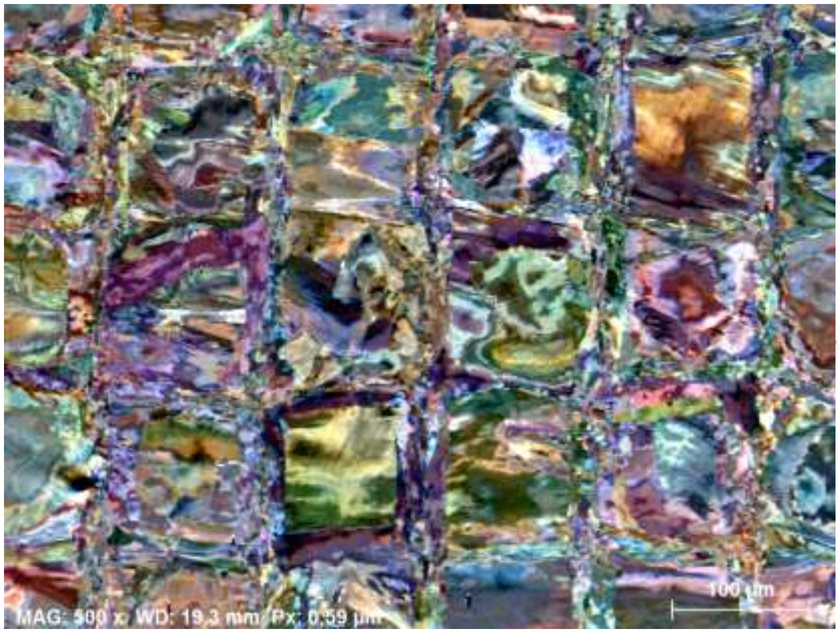


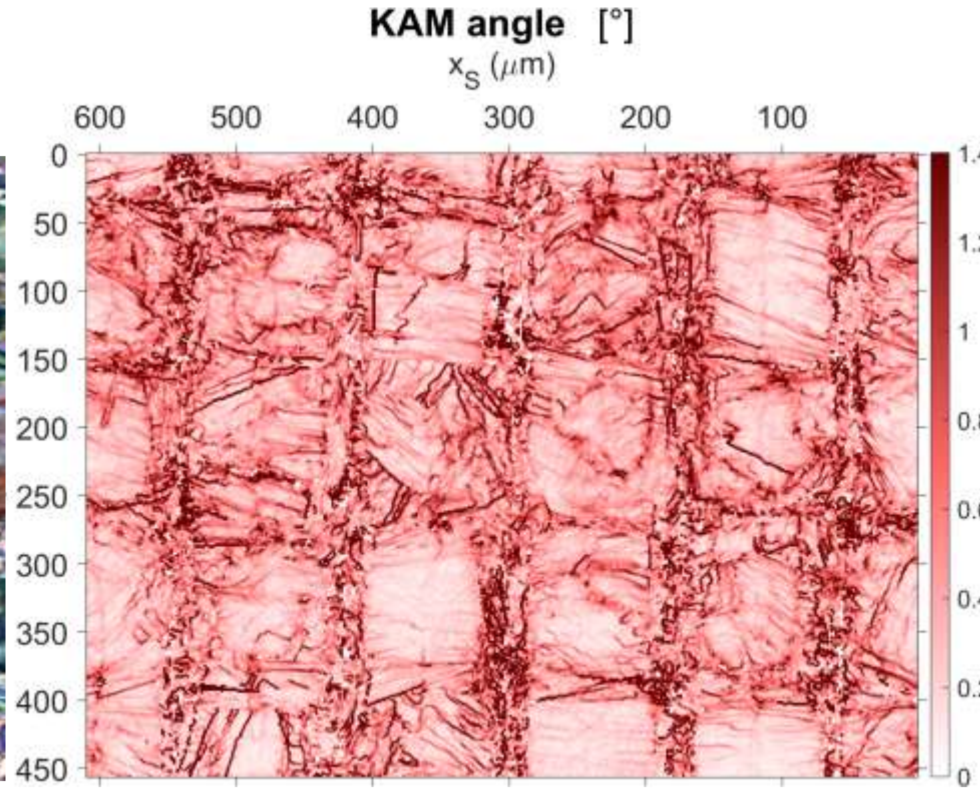
- The resulting **misorientation axes** show a widely **regular distribution** (regarding the sample or processing reference frame).
- However, also they reflect the weld beads interfaces.

Additive manufactured material
Orientation gradient (KAM)

FSE image

100µm

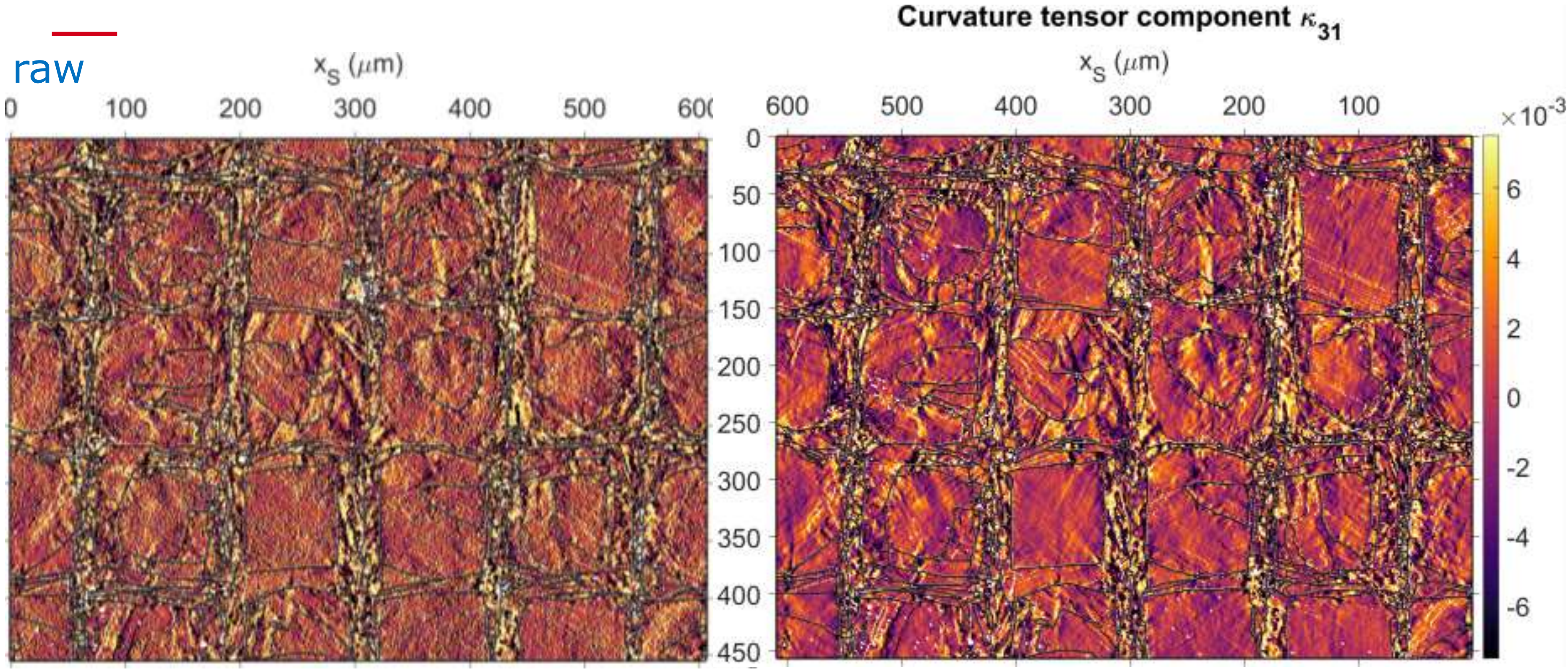




- The KAM angle map displays **numerous subgrain boundaries** preferably perpendicular to the printing path.
- They suggest a **preferred activation of dislocations** causing lattice rotations parallel to the printing direction.

Additive manufactured material

Curvature tensor and GND



- The **curvature tensor** κ (here only single component) **enables** an **estimation of** geometrically necessary dislocation (**GND**) **density**.
- Despite strong deformations the use of **raw orientation data** seems to be **misleading** for an interpretation of GNDs.

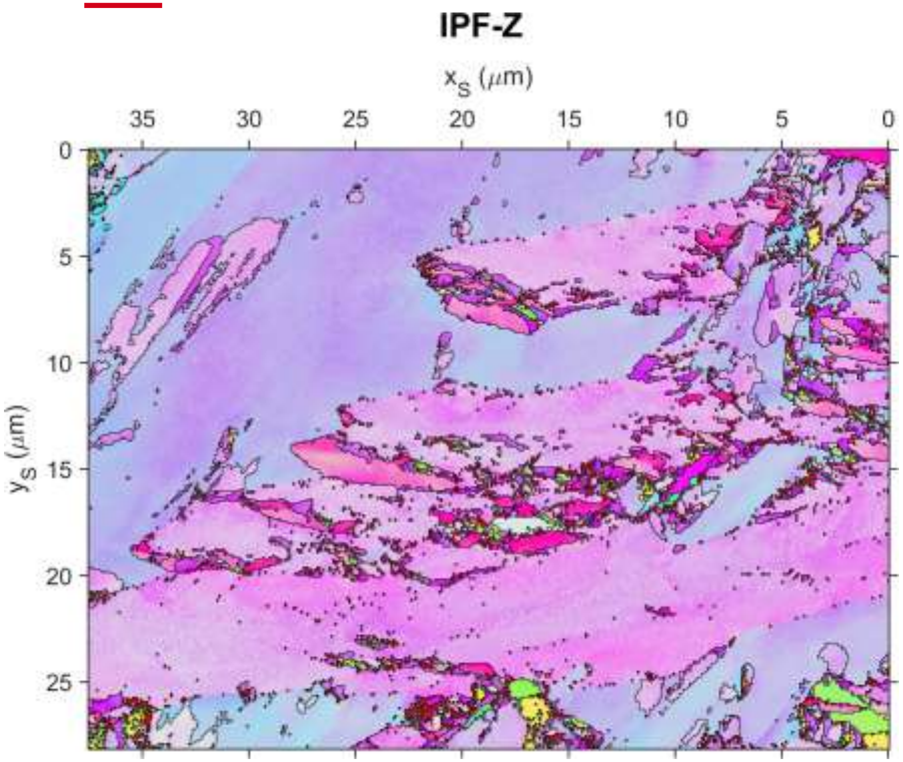
Applications of PM in the past

Keyword: Pseudosymmetry

1. Crystal **structure-related** phenomena
   - Discrimination of **left- and right-handed varieties**
     (*of different (quartz) or identical (tartaric acid) space-group types*)
   - **Prevention of misindexing** in a higher-symmetric lattice
     (*pyrite – FeS_2 – cubic but no 4-fold axis*)
   - **Differentiation between polar directions** or planes
     (*in non-centrosymmetric phases, e.g. semiconductor compounds*)

2. Crystal **lattice-related** phenomena
   - **Prevention of misindexing** caused by lattice transformations
     (e.g. hexagonal ⇨ orthorhombic (Ni_3Sn_2), or any other sublattice description with similar structure)
   - **Orientation precision**
     (*any materials*)
   - **Variable** lattice parameter **ratio**
     (gradient materials caused by diffusion or strain, e.g. martensite)

Variable tetragonality in martensite?



Winkelmann, A. et al. *Mapping of local lattice parameter ratios by projective Kikuchi pattern matching*, Physical Review Materials 2, **2018**, 123803

- One of our latter ideas concerned the **discrimination of c and a** in tetragonal phases **with c/a ≈ 1**.
- How far we can go for **imperfect** phases as **martensite**?

There are **certain problems** to solve, already for a "comparable" presentations because of

- **different** crystal **symmetries** *(IPF colour keys do not match)*,
- continuously **varying lattice parameter ratios** *(infinite phase number vs phase list, how to process pole figures?)*

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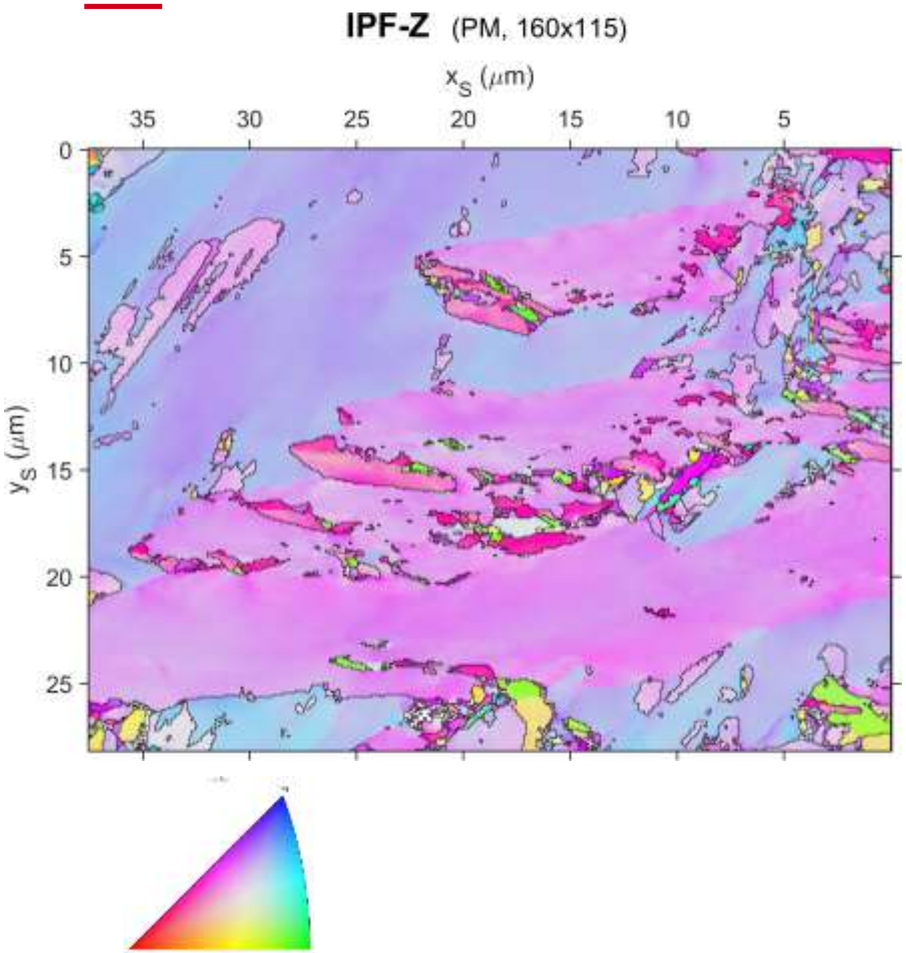

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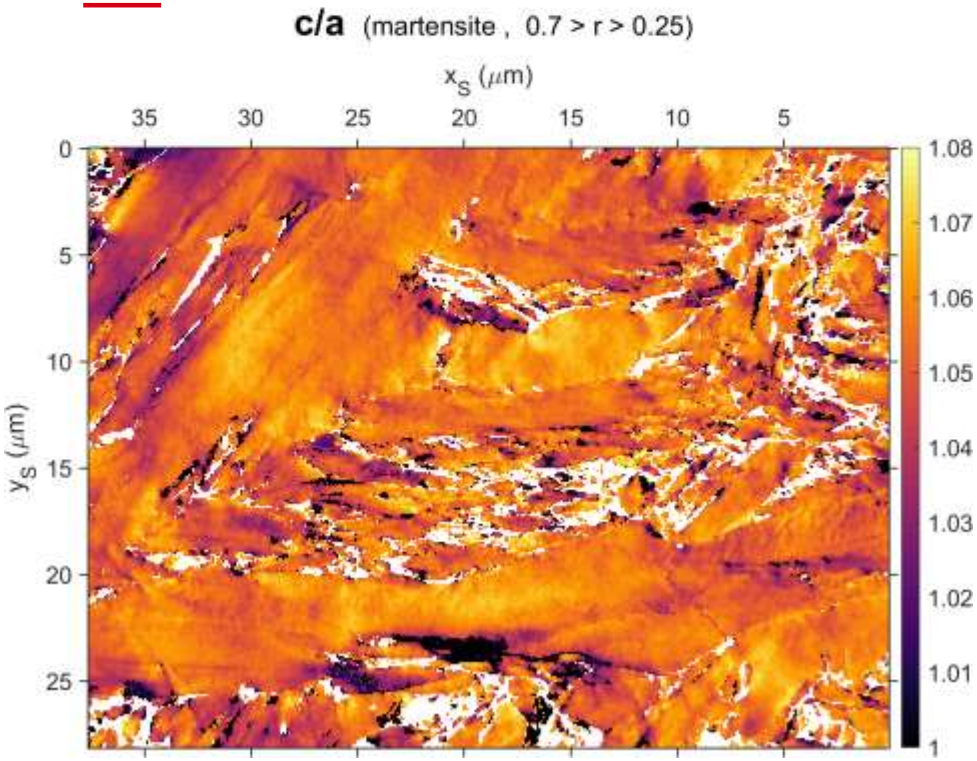

The white areas indicate either martensite with r<.25, or the remaining austenite.

There will be tomorrow in the Young Scientists' Session a talk which gives a few more information about this topic.

Conclusion and Summary

Conventional orientation mapping with **EBSD** is absolutely **sufficient if**

- **only** the **orientation** (texture) of a material **is of interest**,
- the **phases are** well-**known**,
- **misorientations <0.5°** are **irrelevant**.

Pattern **M**atching **is** one **suitable** tool to increase the orientation correctness (accuracy & precision) in order **to**

- **dissolve** any kind of **pseudosymmetry** hidden in crystal structure or lattice
  *(⇨ **big misorientations** caused by small lattice distortions, or by the misuse of Friedel's rule)*,
- **measure small misorientations** below the common orientation-noise level *(⇨ high precision)*
- **discover lattice** parameter **variations** *(gradient materials)*