BAM
Bundesanstalt für Materialforschung und -prüfung

Sicherheit in Technik und Chemie

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CALM down: Identifying unknown phases

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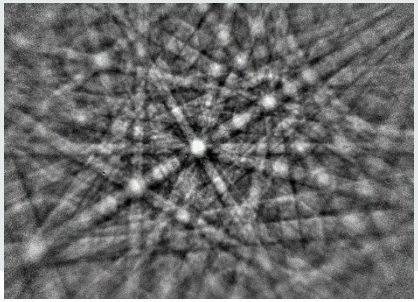
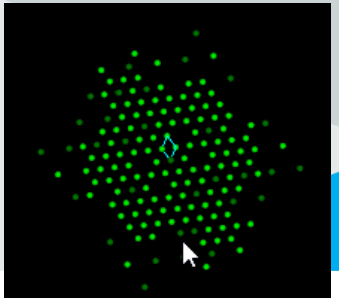
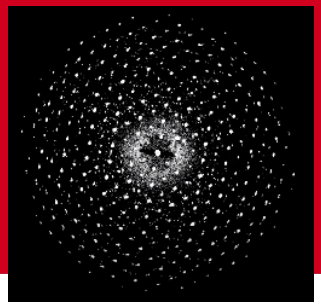
unknown High-Entropy phase





Electron **D**iffraction **T**omography
of Tylenol (acetaminophen)
exposure per frame: 5 ms
total: 3s
R. dos Reis,
Northwestern University (US)



Lattice parameters

Why do we need them? How accurate they have to be?

```
% crystal symmetry
CS_all = {...
  'notIndexed',...
  crystalSymmetry('m-3m', [2.866 2.866 2.866], 'mineral', 'Kamacite', 'color', 'light blue'),...
  crystalSymmetry('m-3m', [3.656 3.656 3.656], 'mineral', 'Taenite', 'color', 'light green'),...
  crystalSymmetry('1 1 2/m', [10.4192 6.0186 4.7768], [90,90,90.952]*degree, 'mineral', 'Sarcopside', 'color', 'magenta'),...
  crystalSymmetry('-4', [9.04 9.04 4.462], 'mineral', 'Schreibersite', 'color', 'yellow')};
CS=CS_all;
```

MTEX mainly works with angles to display pole figures of any kind.

Generally valid computations

	[uvw] (*lattice*)	**[hkl]*** (*reciprocal lattice*)
Angle	$\cos\varrho = \frac{\boldsymbol{R}_1 \cdot \boldsymbol{R}_2}{\lvert\boldsymbol{R}_1\rvert \cdot \lvert\boldsymbol{R}_2\rvert}$	$\cos\varphi = \frac{\boldsymbol{R}_1^* \cdot \boldsymbol{R}_2^*}{\lvert\boldsymbol{R}_1^*\rvert \cdot \lvert\boldsymbol{R}_2^*\rvert}$
Length	$\lvert\boldsymbol{R}_i\rvert = \sqrt{\boldsymbol{R}_i \cdot \boldsymbol{R}_i}$	$\lvert\boldsymbol{R}_i^*\rvert = \sqrt{\boldsymbol{R}_i^* \cdot \boldsymbol{R}_i^*}$
Dot product	$\boldsymbol{R}_i \cdot \boldsymbol{R}_j = [u\,v\,w]_i \cdot \mathbf{G} \cdot \begin{bmatrix} u \\ v \\ w \end{bmatrix}_j$	$\boldsymbol{R}_i * \cdot \boldsymbol{R}_j^* = [h\,k\,l]_i^* \cdot \mathbf{G}^* \cdot \begin{bmatrix} h \\ k \\ l \end{bmatrix}_j^*$ $[h\,k\,l]^* \perp (h\,k\,l)$

Metric tensor

$$\mathbf{G} = \begin{pmatrix} a_o^2 & a_o \cdot b_o \cos\gamma & a_o \cdot c_o \cos\beta \\ g_{12} & b_o^2 & b_o \cdot c_o \cos\alpha \\ g_{13} & g_{23} & c_o^2 \end{pmatrix} = b_o^2 \cdot \begin{pmatrix} \left(\frac{a_o}{b_o}\right)^2 & \left(\frac{a_o}{b_o}\right)\cos\gamma & \left(\frac{a_o}{b_o}\right) \cdot \left(\frac{c_o}{b_o}\right)\cos\beta \\ g_{12} & 1 & \left(\frac{c_o}{b_o}\right)\cos\alpha \\ g_{13} & g_{23} & \left(\frac{c_o}{b_o}\right)^2 \end{pmatrix} = b_o^2 \cdot \mathbf{G}_r$$

From this follows:

For angle computations we only **need** the lattice parameter ratios

$$\frac{a_o}{b_o}, \frac{c_o}{b_o} \quad \textbf{and} \quad \alpha, \beta, \gamma \ .$$

These data are **available** from a single EBSD pattern **with similar accuracy to orientations**.

How is that possible?

High correlation

between bands, intersections and widths

An **EBSD pattern** can be seen as **visual representation of two coupled**, highly **overdetermined systems of equations**.

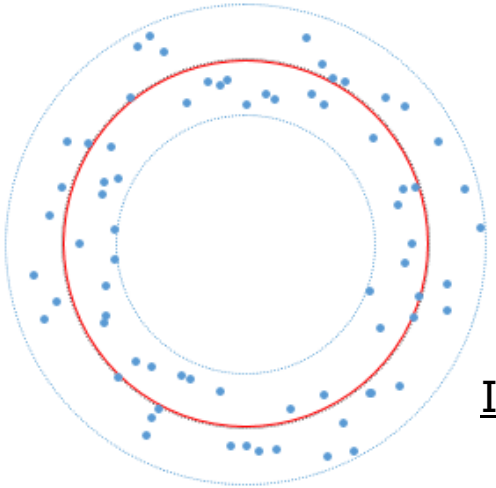
- Only **12 numbers** are unknown:
  - In maximum (!) six lattice parameters: a, b, c, α, β, γ
  - the projection center: (PC_x, PC_y, PC_z) (assumed to be known!)
  - the orientation: (φ_1,Φ,φ_2)
- A single EBSD pattern delivers **50-200 bands** described by distance, slope, and width, effectively providing **150-600** numerical **values for** the determination of only **9 unknowns**.
- The high correlation results from
  1. crystal and reciprocal lattice which are **translation lattices**:

$$[u\,v\,w]_k = [u\,v\,w]_i + [u\,v\,w]_j \qquad [h\,k\,l]^*_k = [h\,k\,l]^*_i + [h\,k\,l]^*_j$$

  2. simple **relationships** between crystal and reciprocal lattice:

$$[u\,v\,w]_r = [h\,k\,l]^*_s \times [h\,k\,l]^*_t \qquad [h\,k\,l]^*_n = [u\,v\,w]_p \times [u\,v\,w]_q$$

Inherent constrains
Principle



Question: How well is a circle described by a point cloud when we know it is a circle?

- ***The black circle*** *between the blue ones (deviation range) is the one* ***we are looking for****.*
- *The* ***blue dots*** *are* ***randomly shifted*** *from the black circle.*
- *The* ***red circle*** *is drawn using the average* ***diameter derived from the blue dots****.*

Insight: **If** the **shape** of an object **is known**, **detection is easier and** much **more accurate**.

(cf. car license plate recognition from a few pixels only)

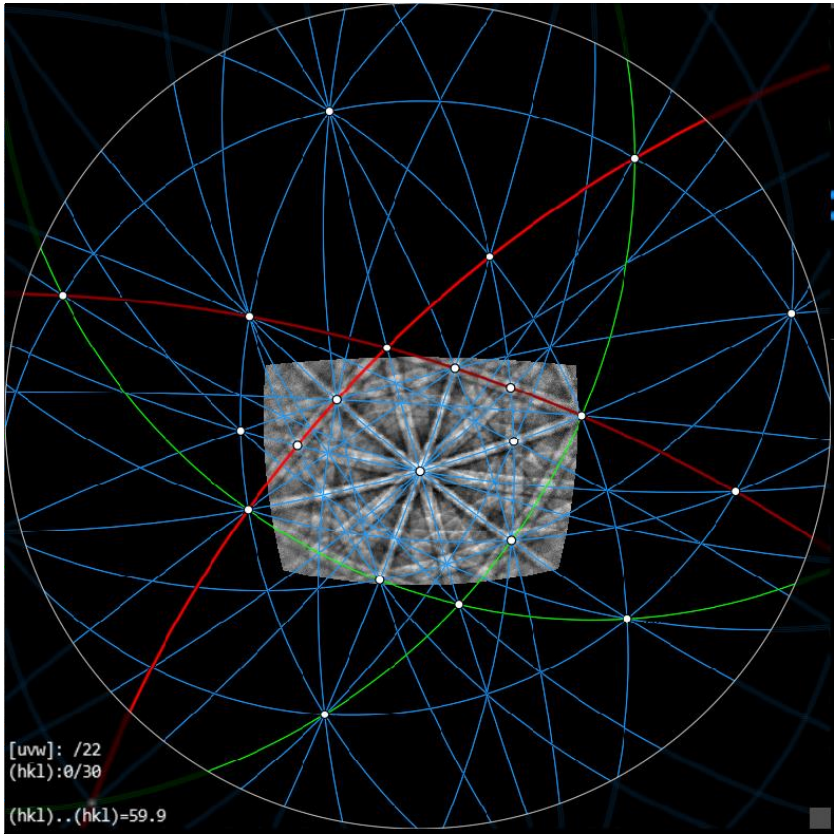
For EBSD patterns follows, mainly from projective geometry:

- In a pattern **lattice plane traces are straight lines**.
- Intersections of traces describe lattice directions: **two (hkl) ⇨ [uvw]**.
- But also **two [uvw] ⇨ (hkl)**.
- The indices **h,k,l** as well as **u,v,w** are **small integers**.

Pattern analysis in CALM

Crystallographic Analysis of the Lattice Metric

1. The **projection center** (pattern center + DD): 4-direction approach.
2. The **trace positions** of lattice planes: 4-line approach.
3. The **bandwidth** definition / selection.

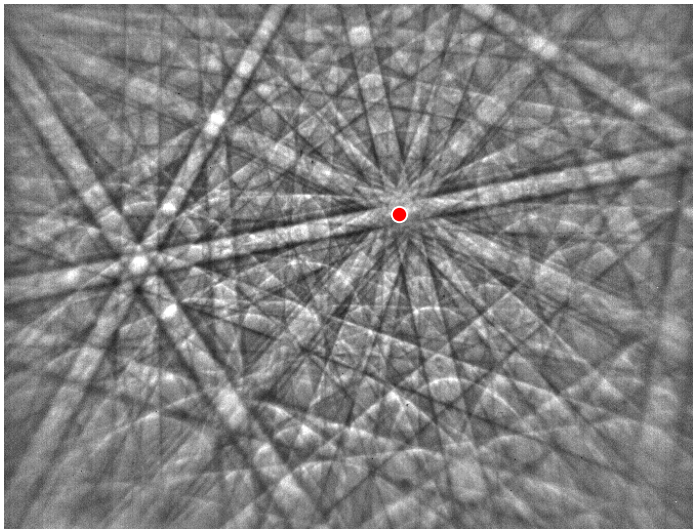


Please note:

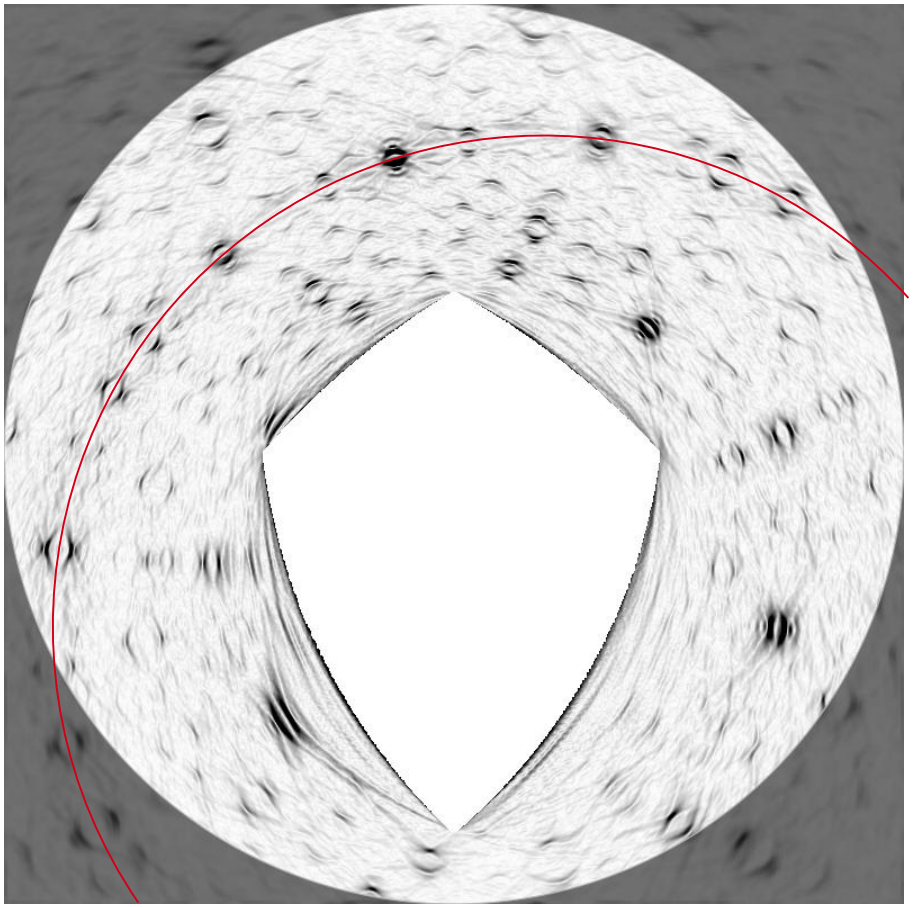
High resolution patterns are beneficial but **not** absolutely **necessary**!

Edge filtered Funk transform

Spherical Radon transform of great circles



GaAs

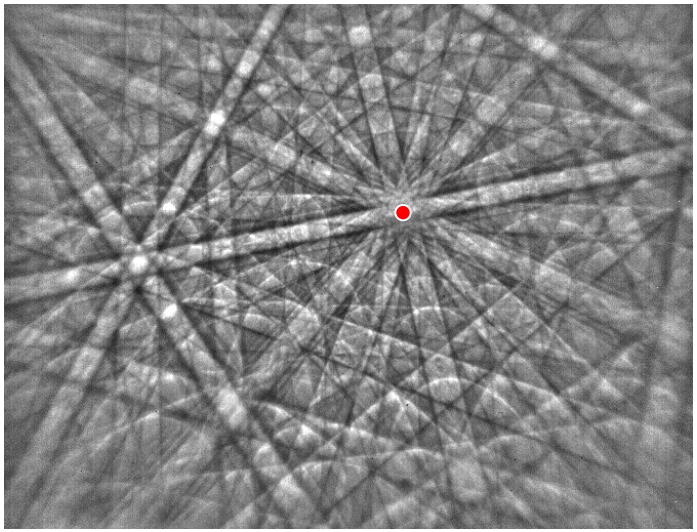


- Stereographic **projection of** the **reciprocal lattice**.
- **(uvw)*** are **great circles**.

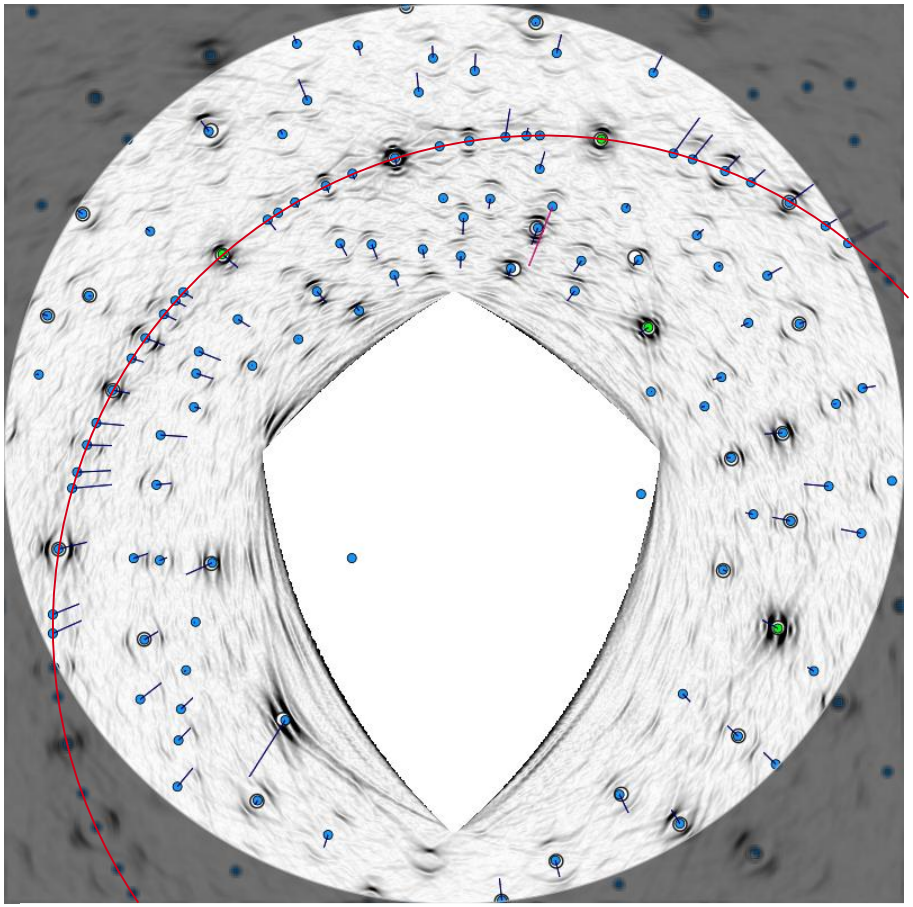
$$[u\,v\,w] \perp (u\,v\,w)^*$$

Edge filtered Funk transform

Spherical Radon transform of great circles



GaAs



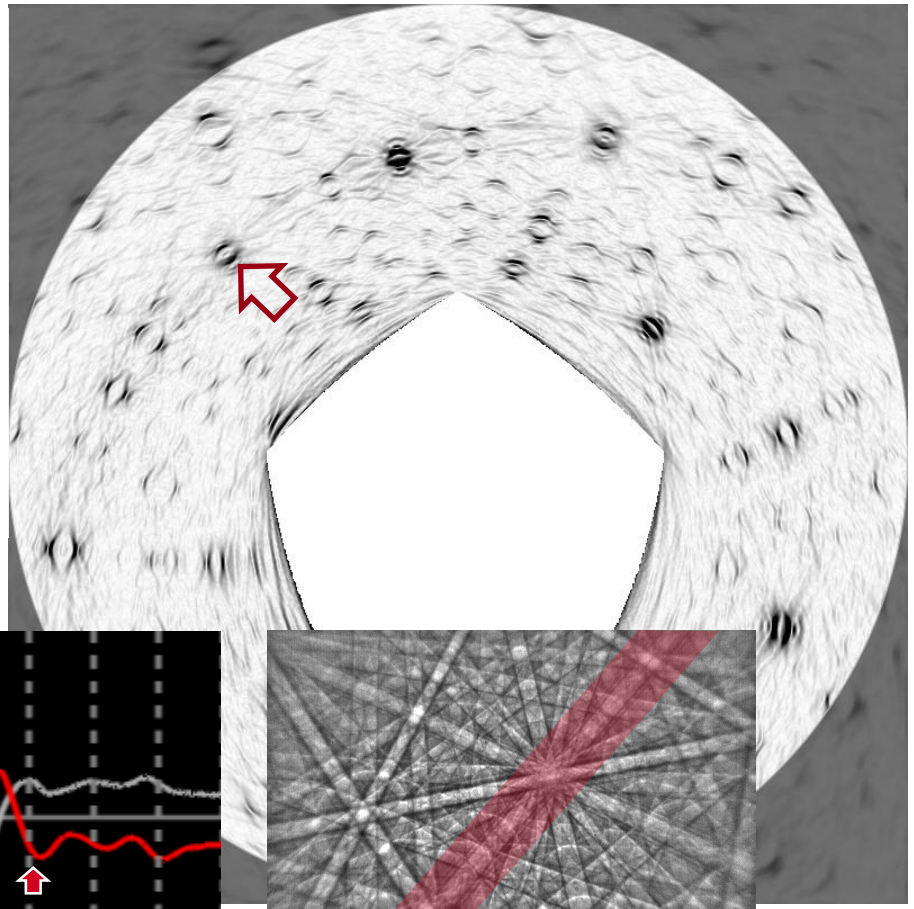
- Stereographic **projection of** the **reciprocal lattice**.
- **(uvw)*** are **great circles**.
- **[hkl]*** are the (blue) centered **points** in ring-shaped features.
- The **bars** are **tiny misalignments** of the traces.

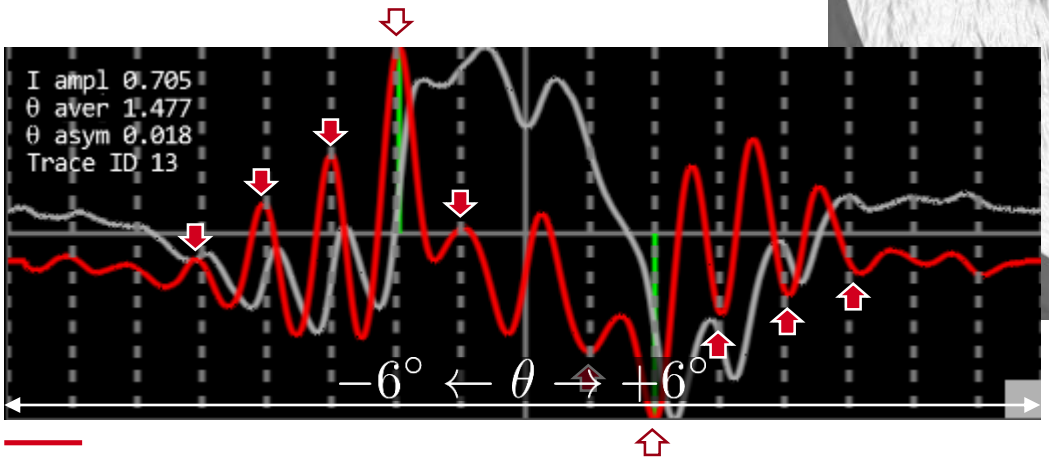
$$[u\,v\,w] \perp (u\,v\,w)^*$$

In the red zone 33 bands can be discovered.

Band profiles

- For each **band** its **profile** (*light gray curve*) can be displayed.
- The **extrema** – left: minima, right: maxima – of the 1st derivative (red curve) **indicate** possible **interference orders**.
- The **bars** at the blue dots **display** θasym, the **asymmetric position** of the extrema (*green lines*) in [°].

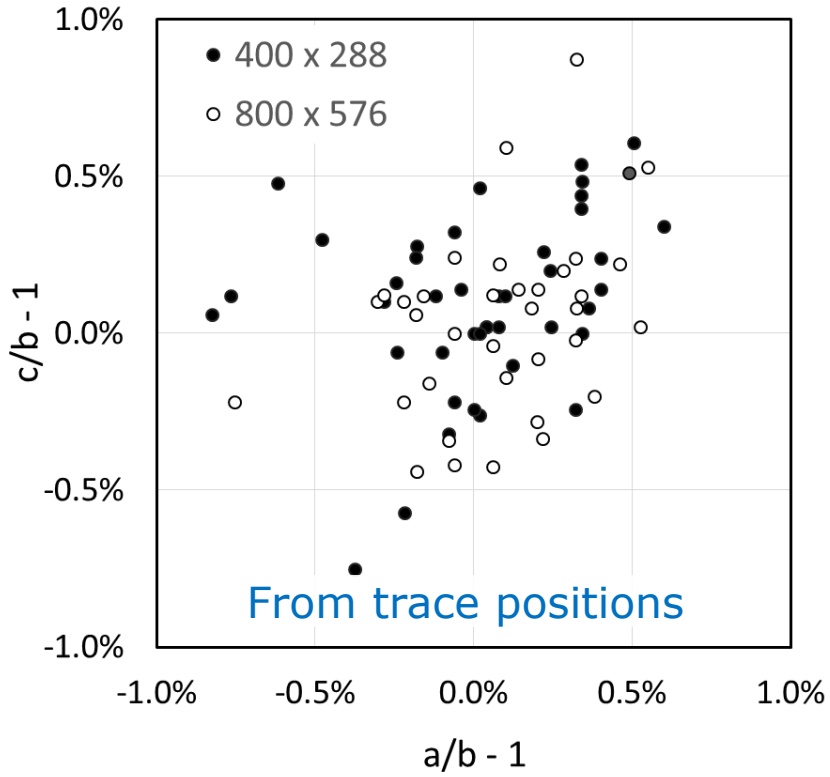




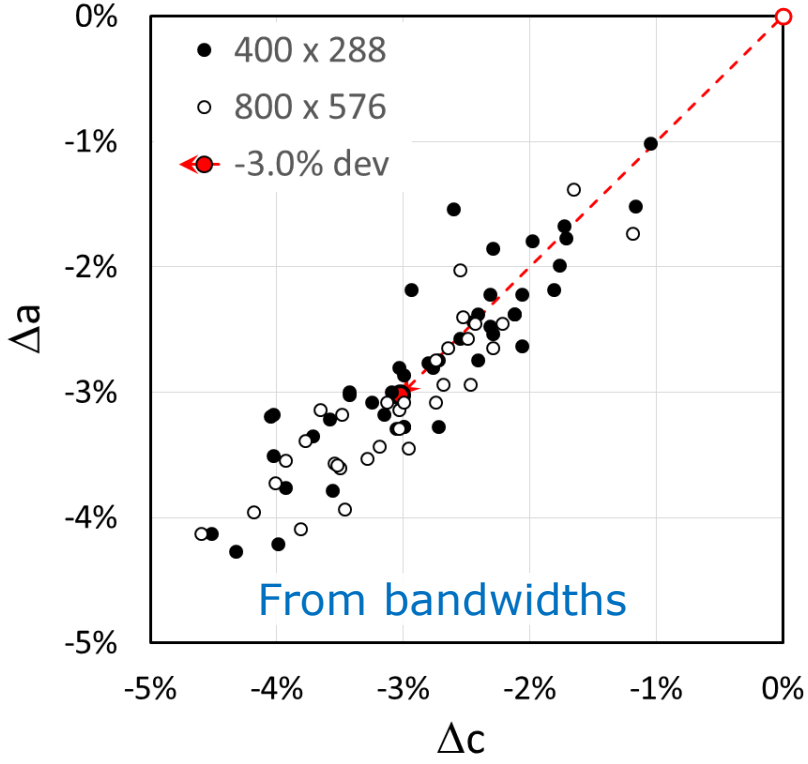
Lattice parameters and ratio

Corundum (rhombohedral)

72 experimental patterns of different orientation and resolution show:



The lattice parameter **ratios** have a deviation **<0.6%**.



The lattice parameters also **scatter only ±1.5%** around a value which is, however, shifted by **-3.0%**.

Possible applications

Main focus

- Lattice parameter and Bravais lattice extraction
- Symmetry approximation
- Lattice parameter ratio mapping

But secondarily other alternative applications

- (Pseudo)symmetry evaluation
- Phase confirmation (superstructures)
- Pattern overlapping
- Projection center confirmation
- Indexing of EBSD patterns
- Charging (local pattern distortions; landing energy)
- Excess deficiency impact
- and of course: TEACHING

CALM: Crystallographic Analysis of the Lattice Metric