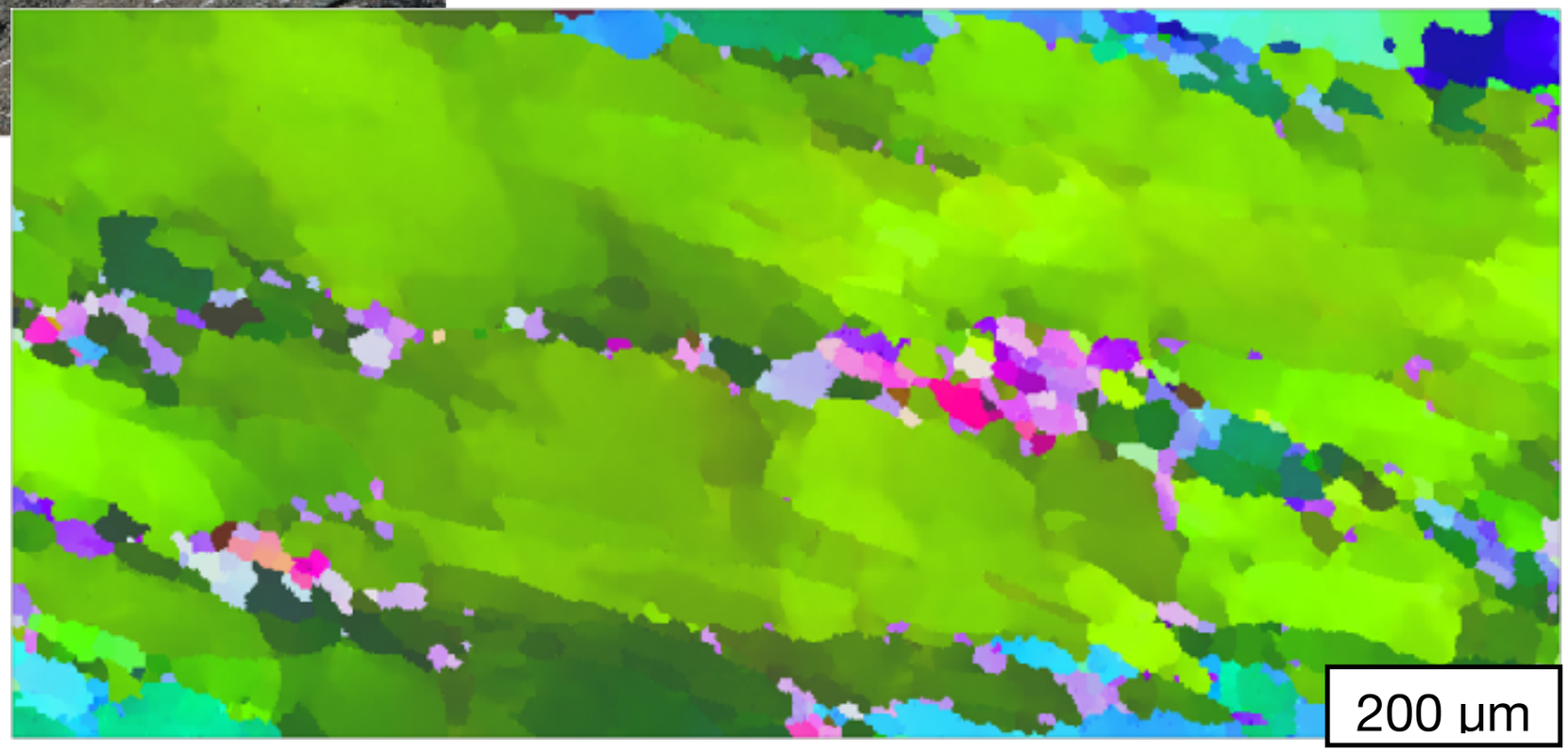
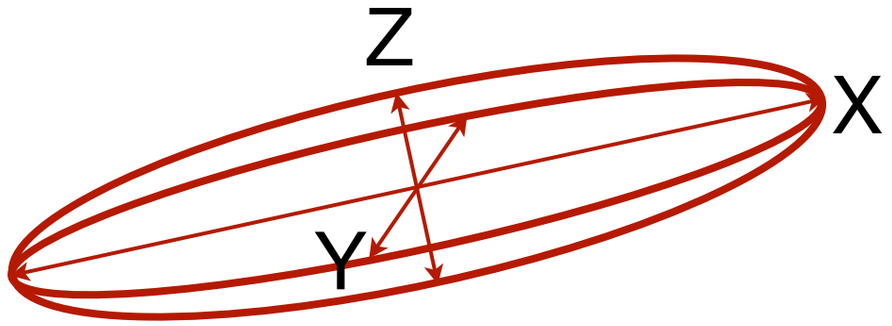
EBSD on geologic samples - from mountain to crystal orientation

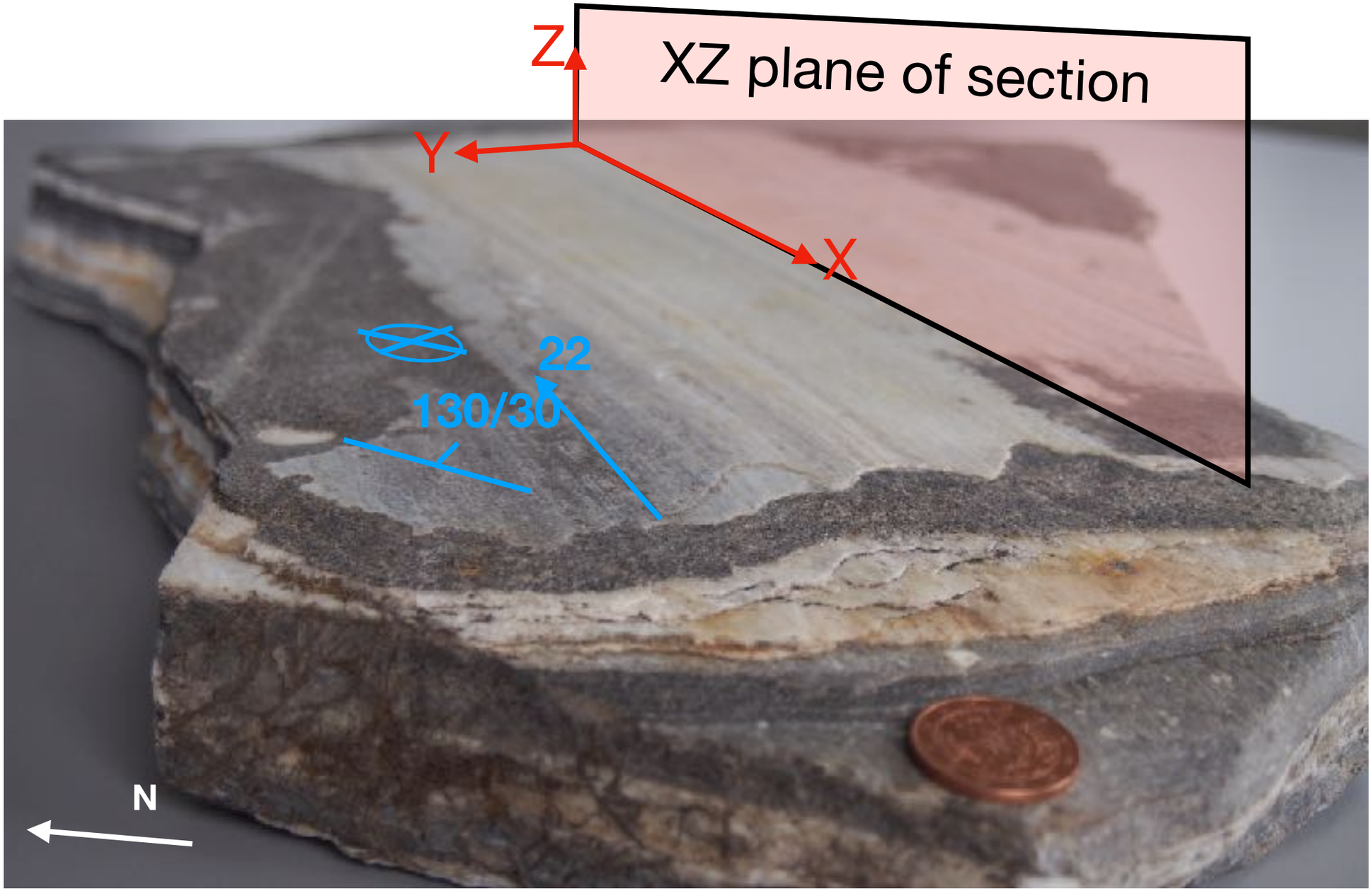
Piz Platta



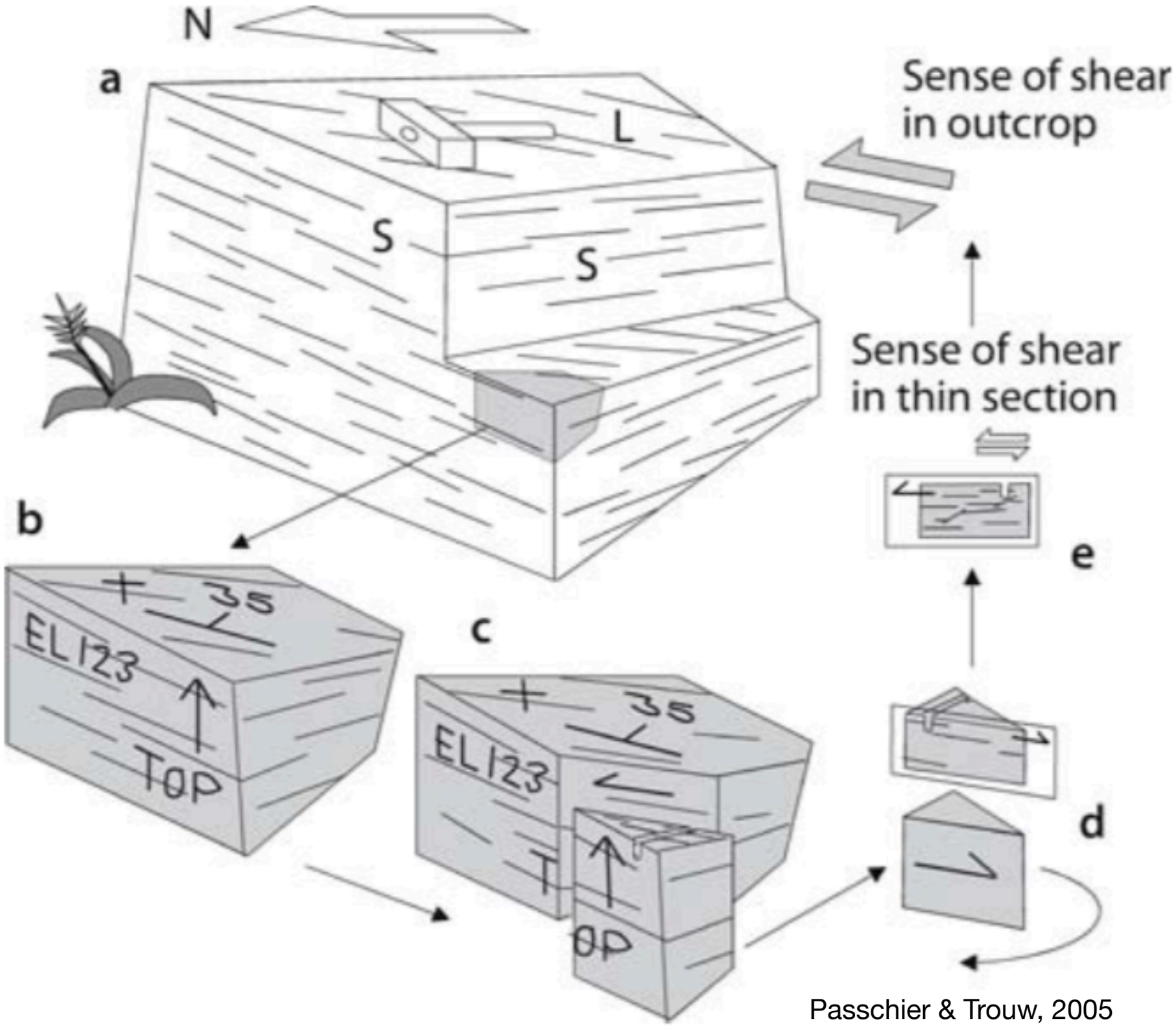
Specimen coordinates

in case of deformed rocks:
foliation ~ often XY plane of finite strain
(stretching) lineation ~ X of finite strain





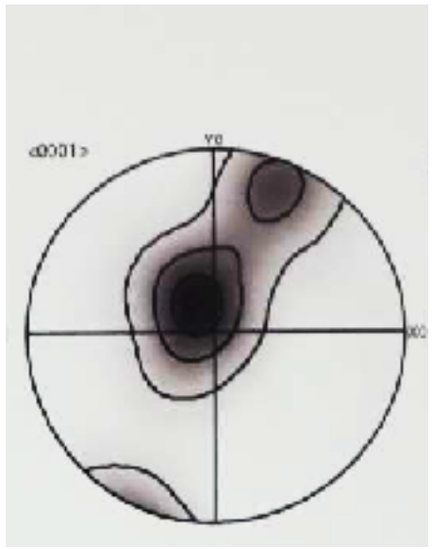
Specimen coordinate system



Passchier & Trouw, 2005

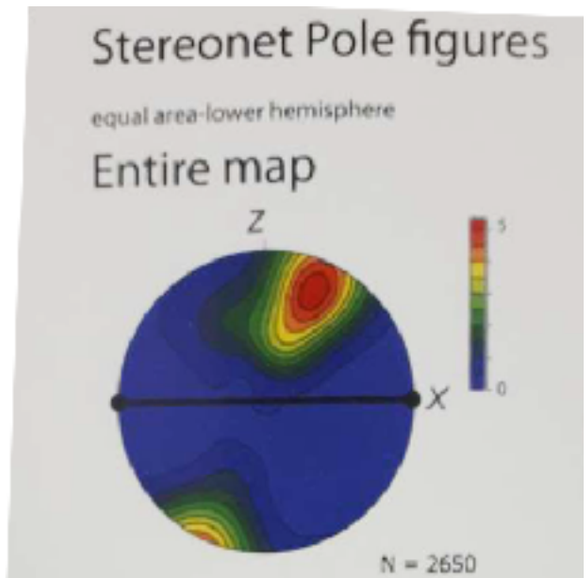
reference frames are (in many cases) important

EBSD (Oxford)



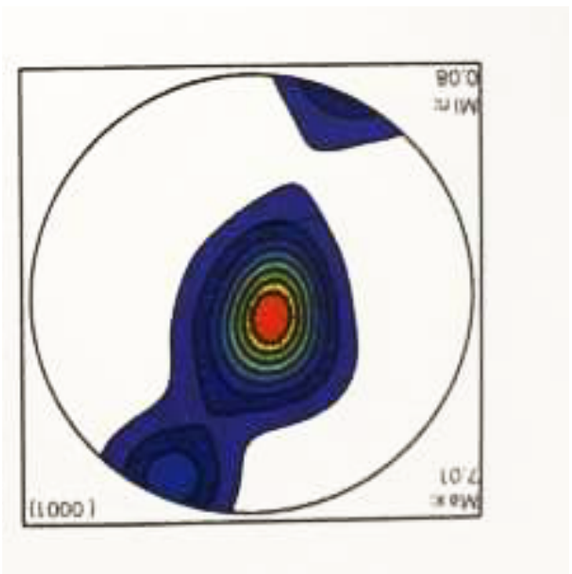
lower

optical (FA)



lower

EBSD (TSL/OIM)



(originally upper)

optical (CIP)

lower

and also the "correct" one

from:

Geophysical Research Abstracts
Vol. 16, EGU2014-3057, 2014
EGU General Assembly 2014
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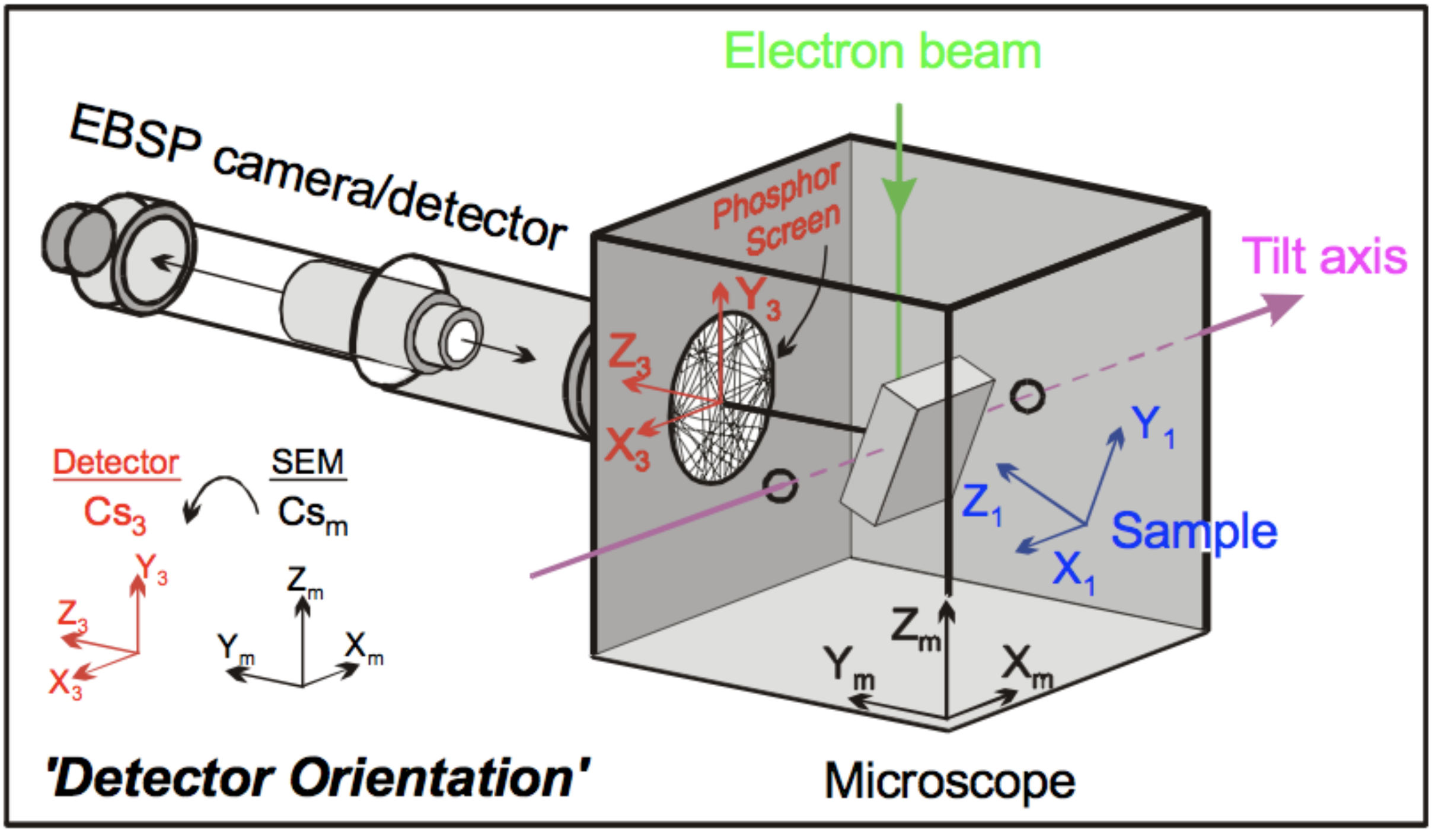
A Community Database of Quartz Microstructures: Can we make measurements that constrain rheology?

Virginia Toy (1), Mark Peternell (2), Luiz Morales (3), and Ruediger Kilian (4)

Coordinate systems in EBSD systems



Absolute orientations:

at around 2014/15: 7 out of 8 tested labs (from one manufacturer) produced 180° rotated orientation, follow-up: https://groups.google.com/d/msg/mtexmail/EpOQo04KsmM/ugvOYTOqBgAJ

Geophysical Research Abstracts
Vol. 18, EGU2016-8221, 2016
EGU General Assembly 2016
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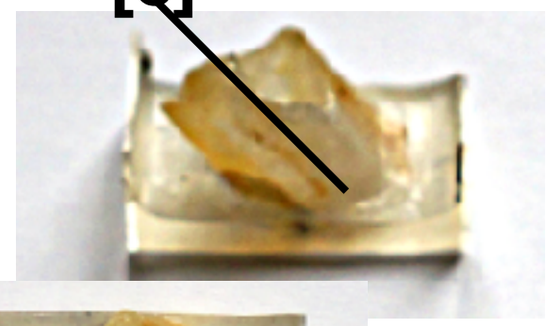
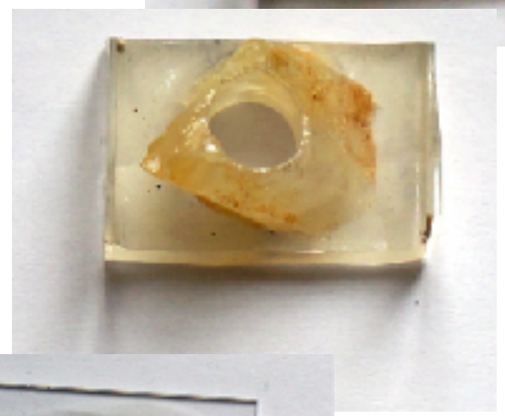
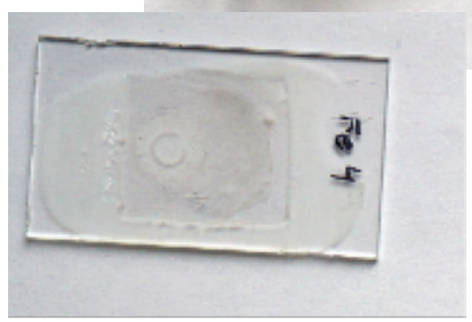
Absolute orientations from EBSD measurements - as easy as it seems?

Rüdiger Kilian (1), Michel Bestmann (2), Renée Heilbronner (1,3)







Materials Characterization 117 (2016) 113–126

Contents lists available at ScienceDirect

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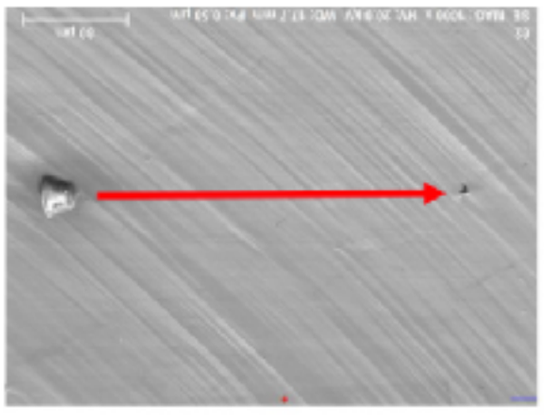
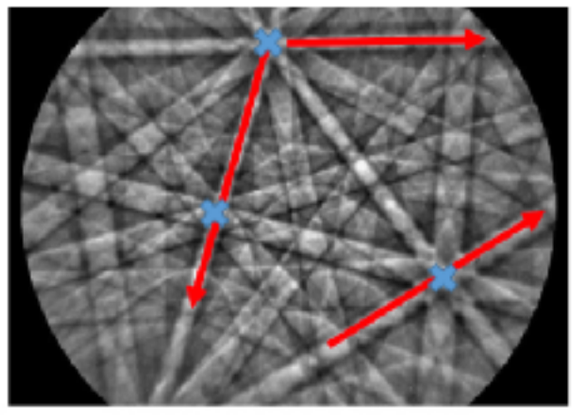
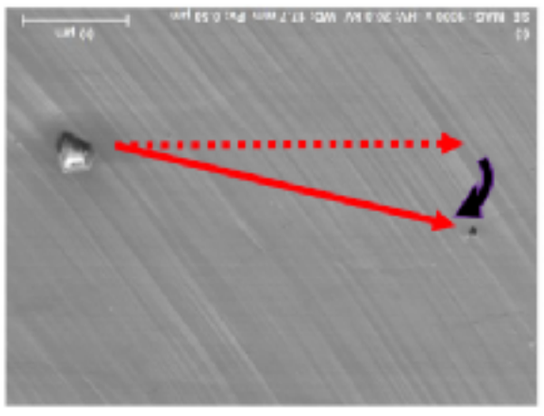
journal homepage: www.elsevier.com/locate/matchar

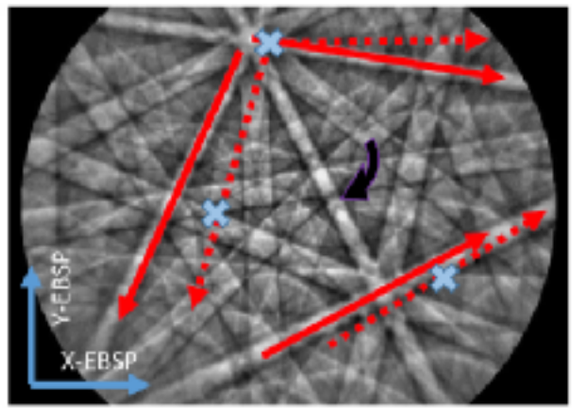
Tutorial: Crystal orientations and EBSD — Or which way is up?

T.B. Britton [a,*], J. Jiang [a], Y. Guo [b,1], A. Vilalta-Clemente [b], D. Wallis [c], L.N. Hansen [c], A. Winkelmann [d], A.J. Wilkinson [b]

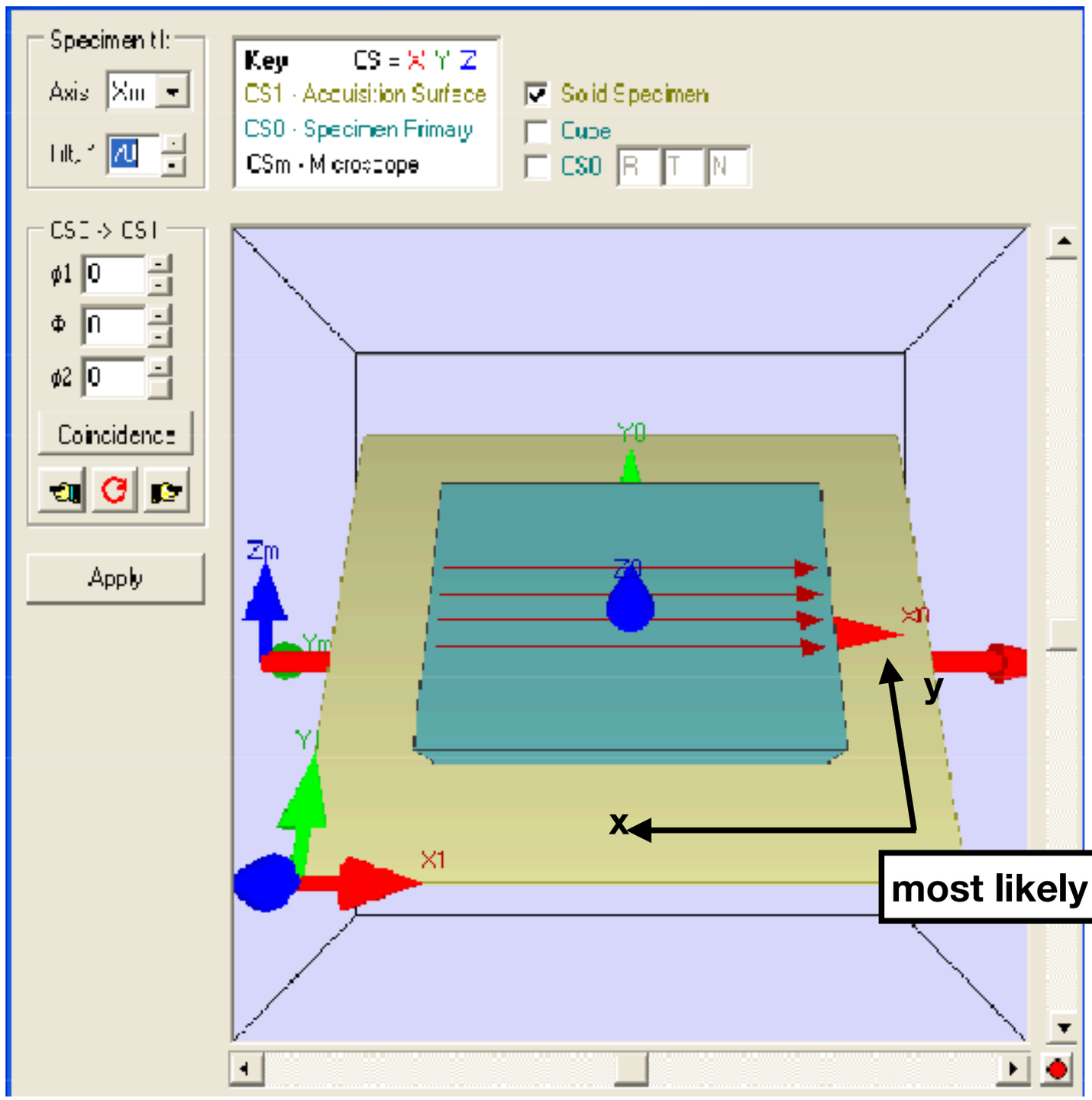








Reference frames: example 1



Channel5 "virtual chamber" camera view

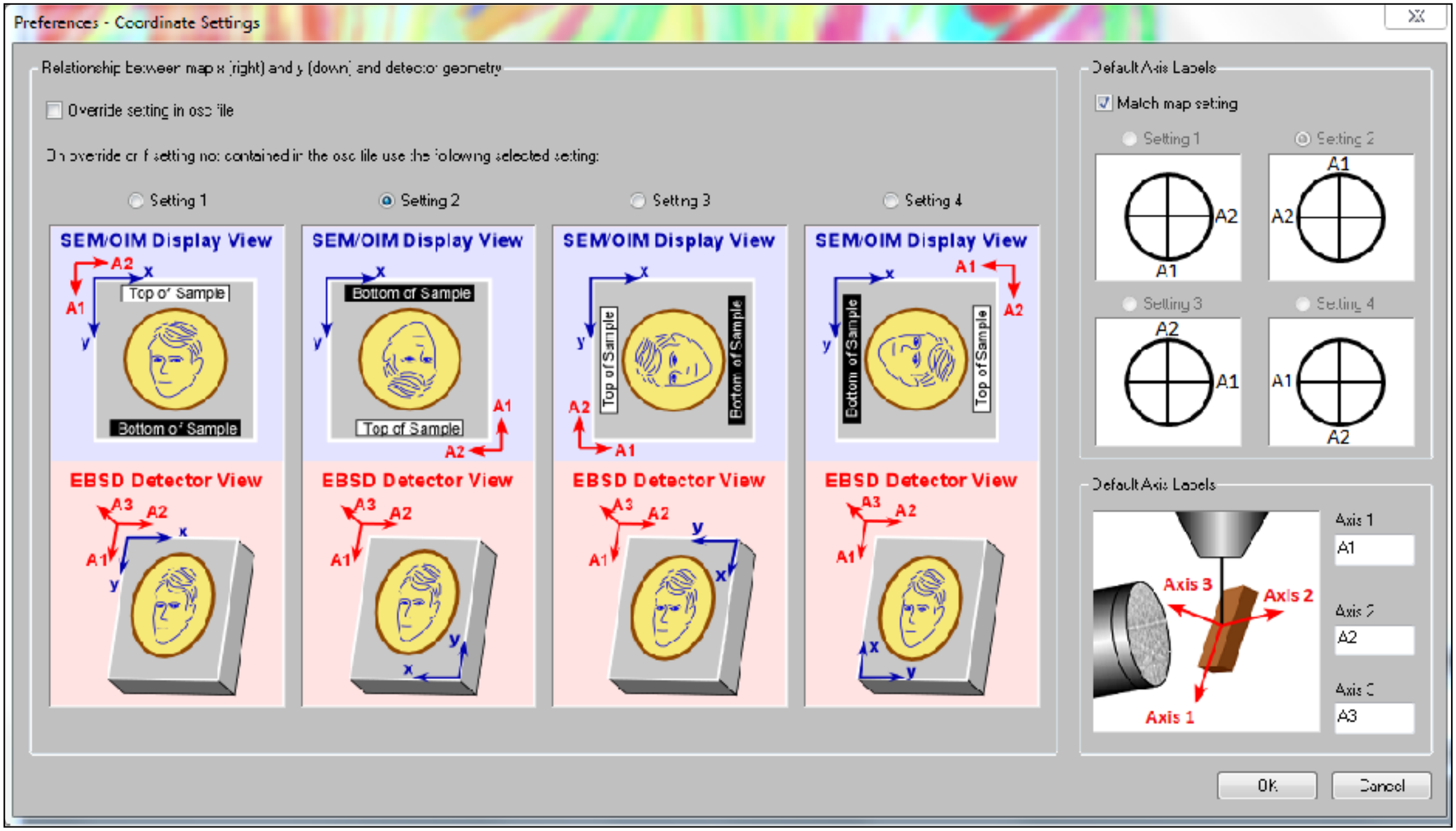
notes for Oxford systems:

- Euler angles are within CS1
- ctf stores angles wrt CS0 (good, if CS1->CS0 relation correct)
- as of at least ~2015, Aztecs different export conventions did nothing on ctf (AcqE1,2,3 =0)
- .crc/.cpr format has relation but binary and subject to change

from .ctf header

```
...
XCells 30
YCells 30
XStep  1
YStep  1
AcqE1  0
AcqE2  0
AcqE3  0
...
```

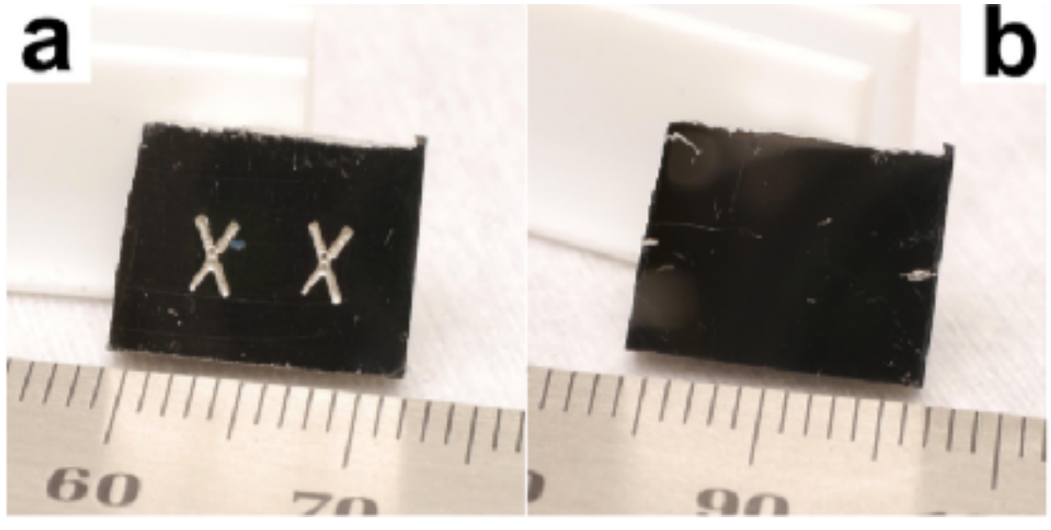
Reference frames: example 2



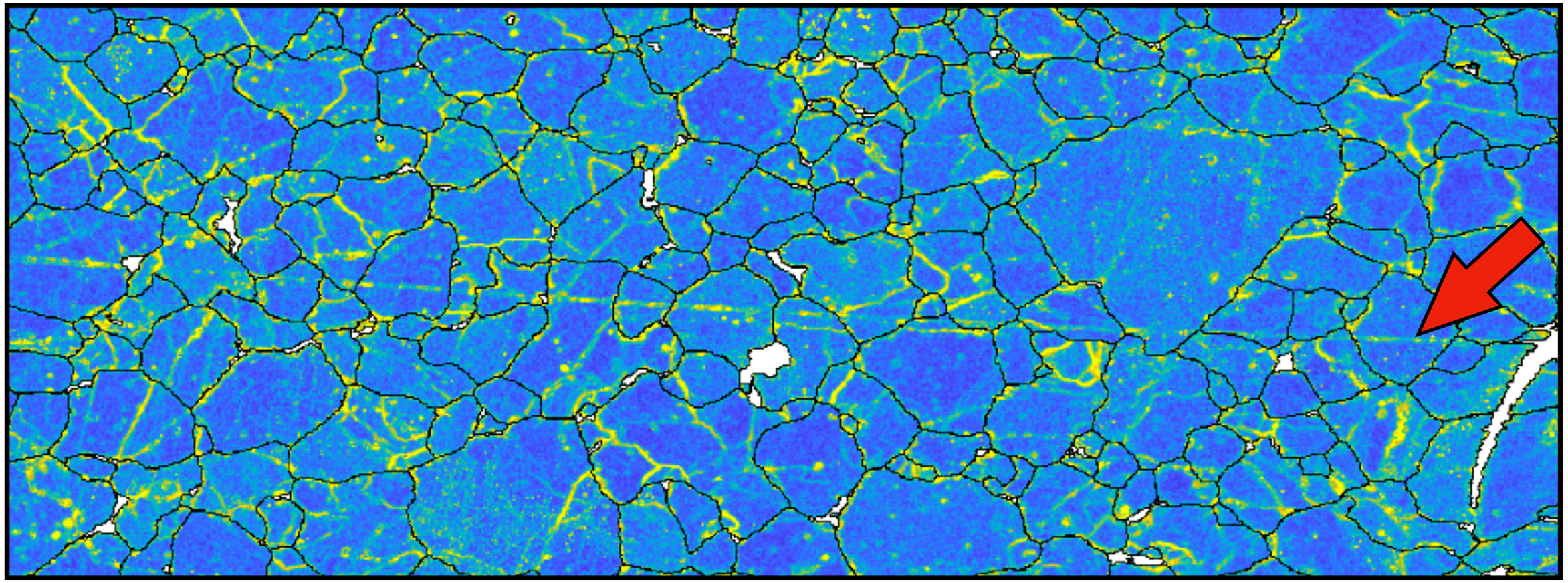
EDAX/OIM

- .ang files do not store the setting!
- .osc is binary and subject to change
- .hdf contains the setting

next: sample preparation

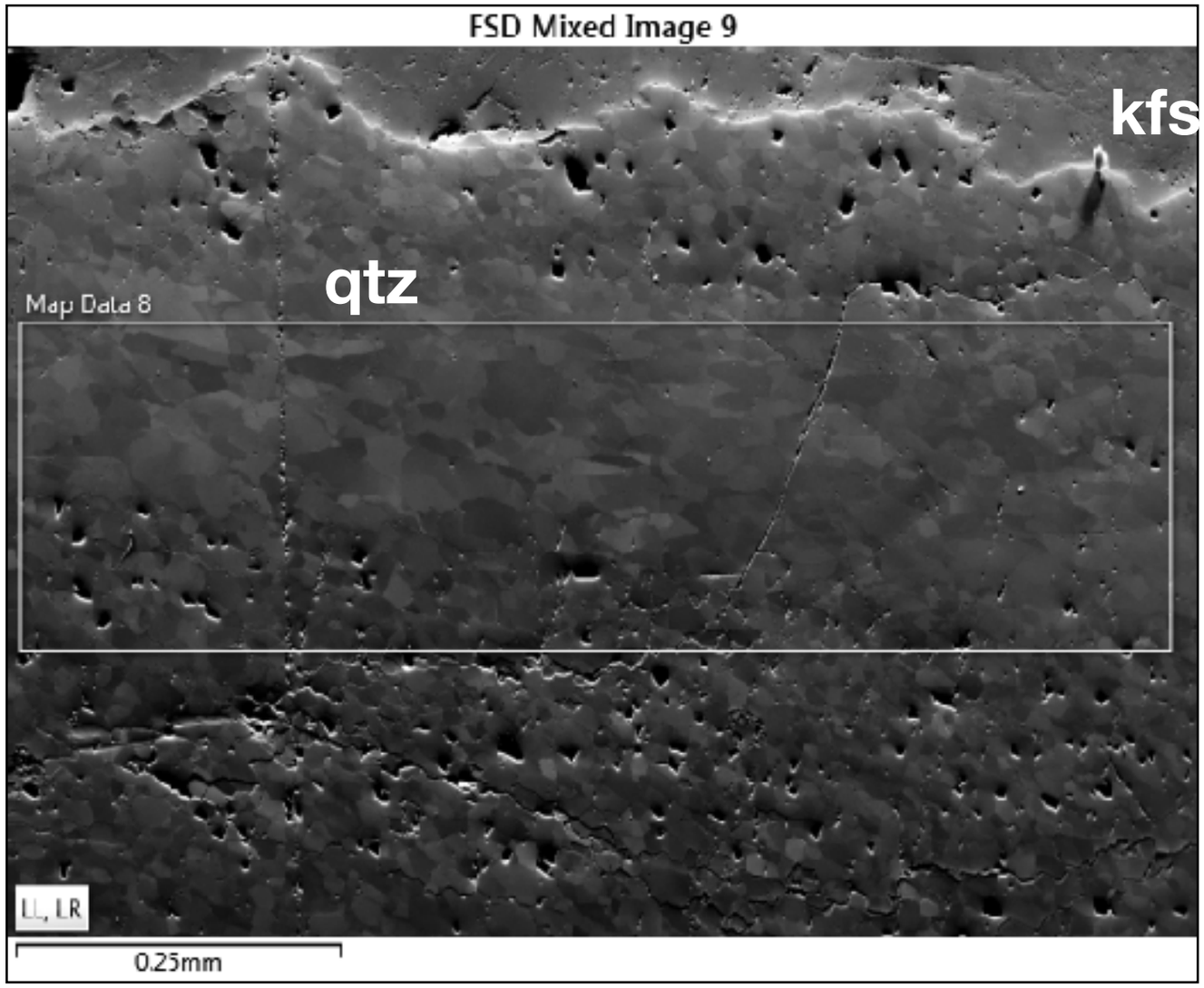


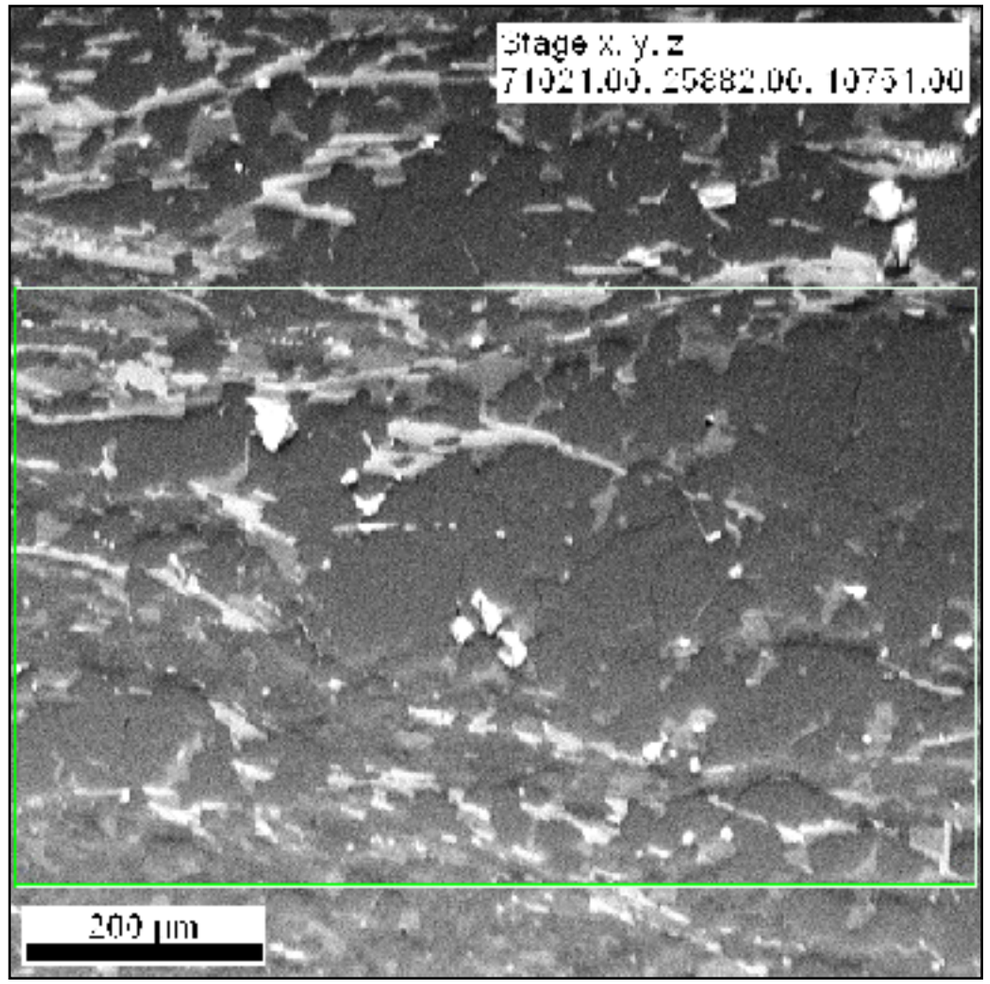
depth of damage: White & Keller, 2015



grinding/polishing defect in polycrystalline quartz /KAM

sample preparation





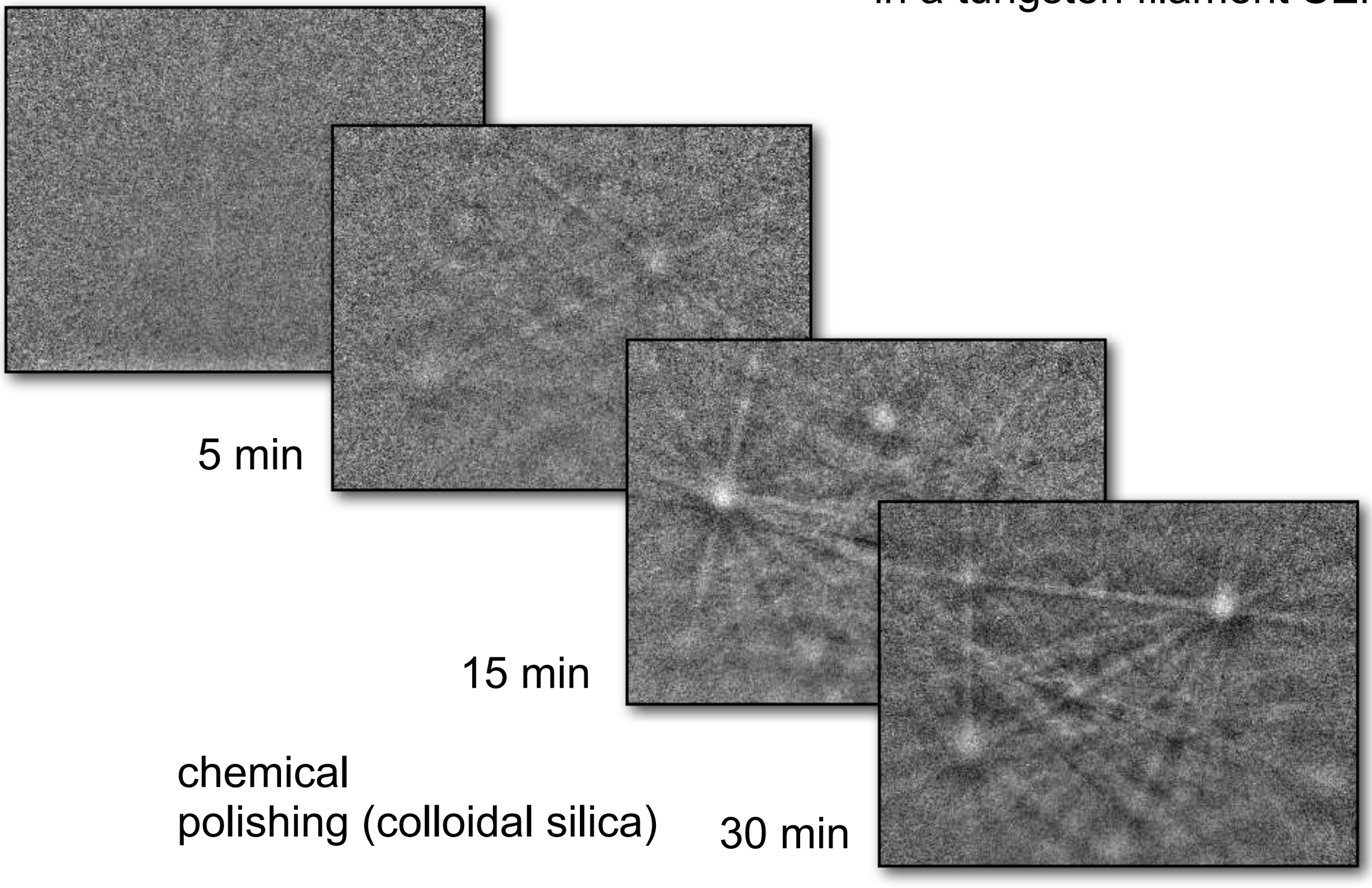
BSE

former standard:

- mechanical polishing 5, 3, 1, 0.25 µm diamond paste
- low force, soft/hard cloth, vibratory pad, depending on material and quality of mechanical polish, 30 min - 10 hrs using alkaline colloidal silica (pH 9-12)
- good results for monophase materials
- sometimes considerable topography for polyphase materials
- long polishing time makes experiments quite difficult

mechanical polish
(0.25µm)

quartz at 20 kV/15 nA
in a tungsten filament SEM



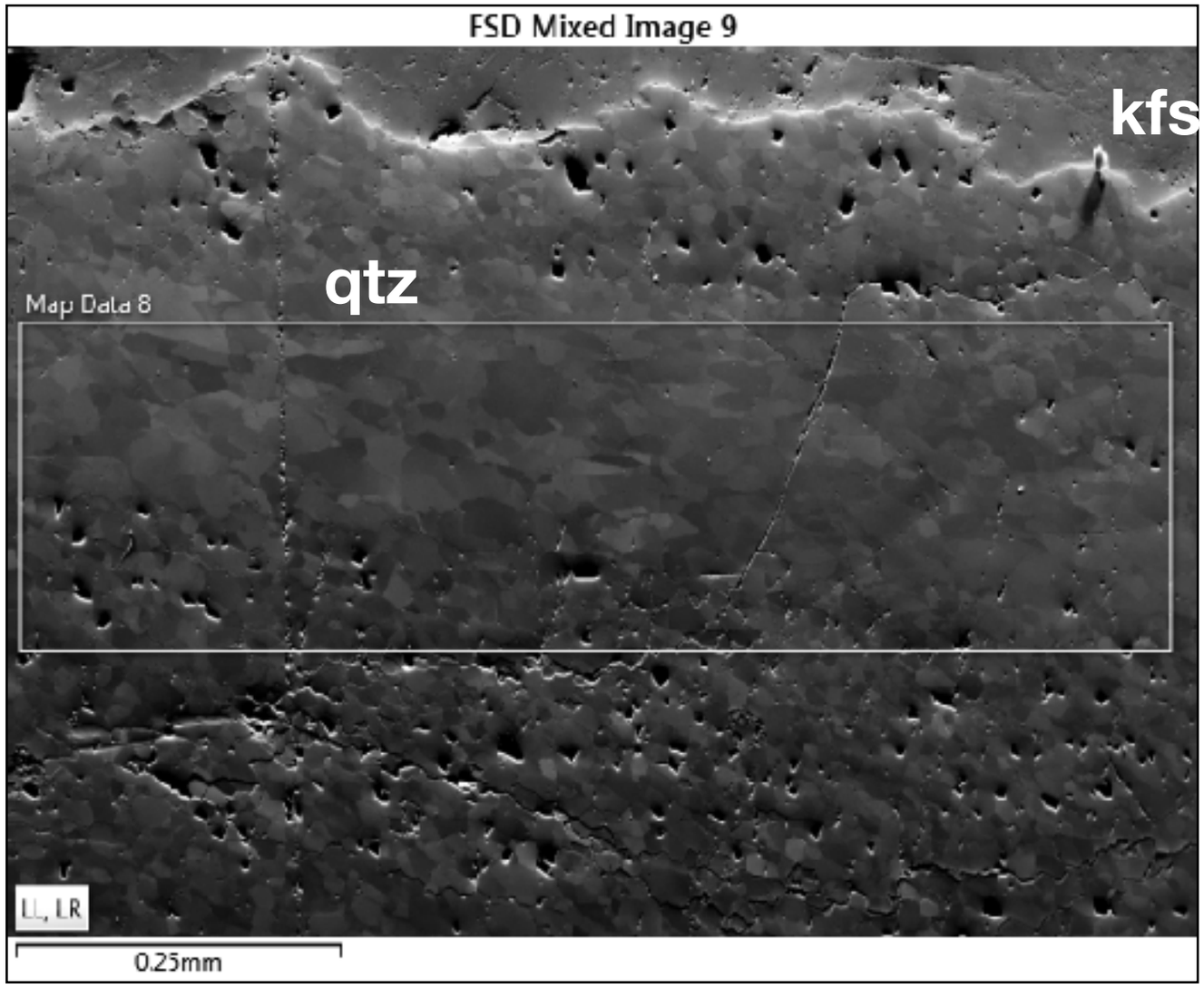
5 min

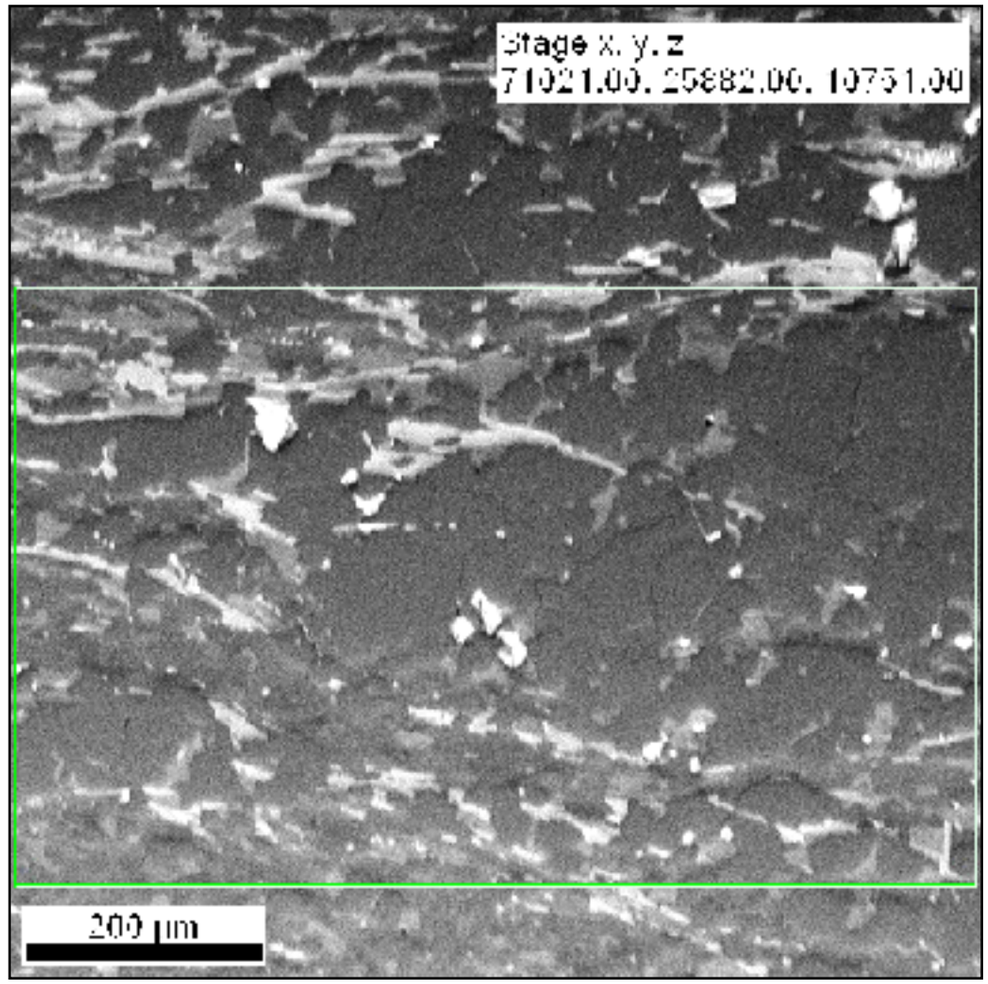
15 min

chemical
polishing (colloidal silica)

30 min

Sample preparation



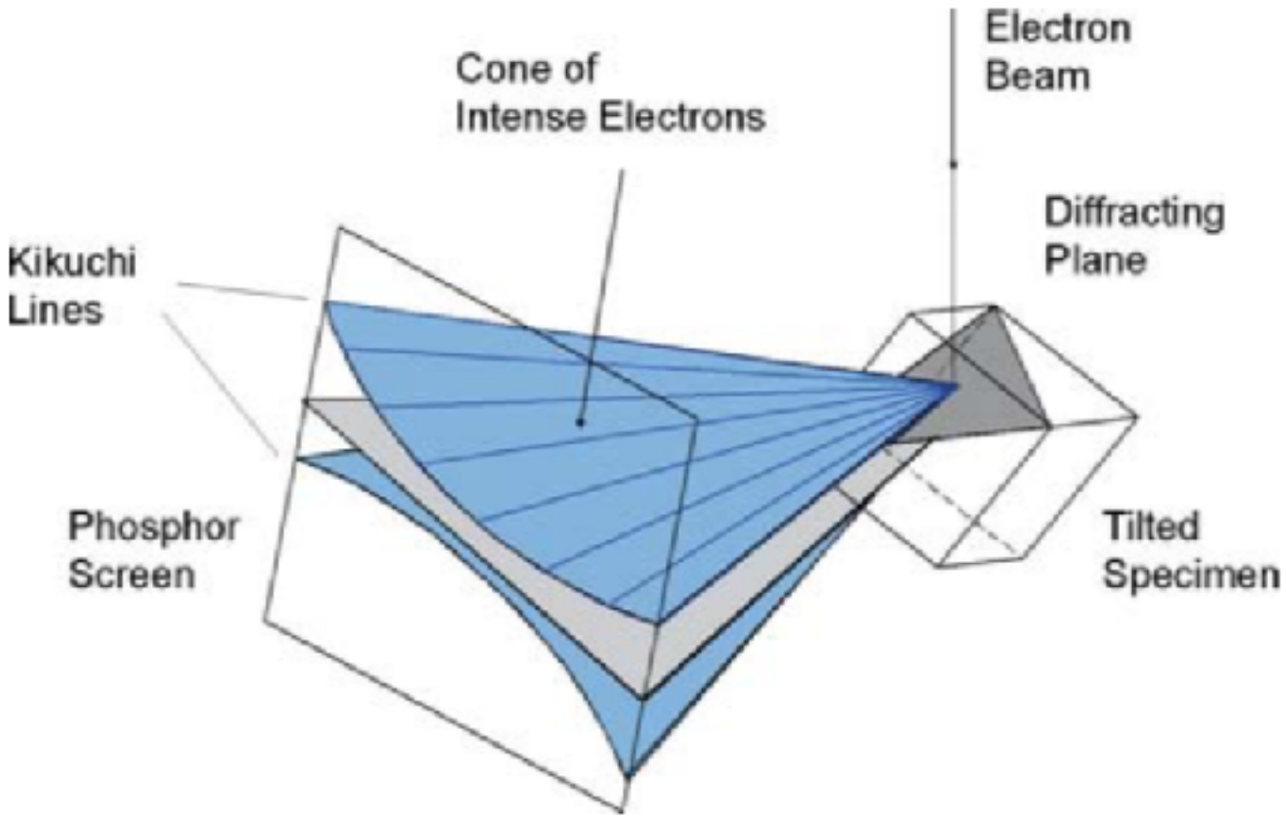


BSE

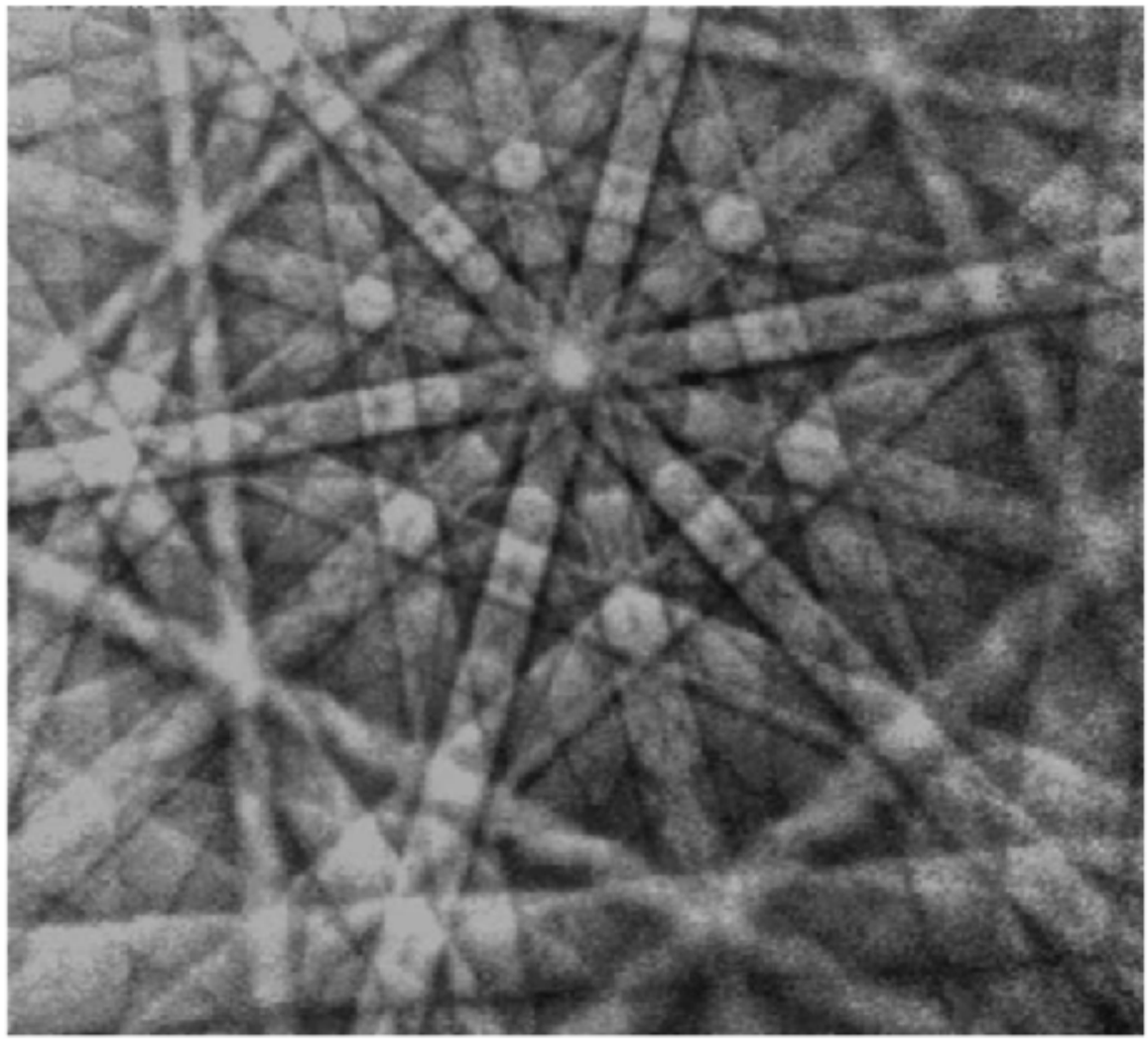
alternative:

- mechanical polishing 5, 3, 1, 0.25 µm diamond paste
- relatively high force, (10-20N/thin section), relatively fast polishing on soft, porous pad, colloidal silica (1-2 minutes)
- slightly better on polyphase materials

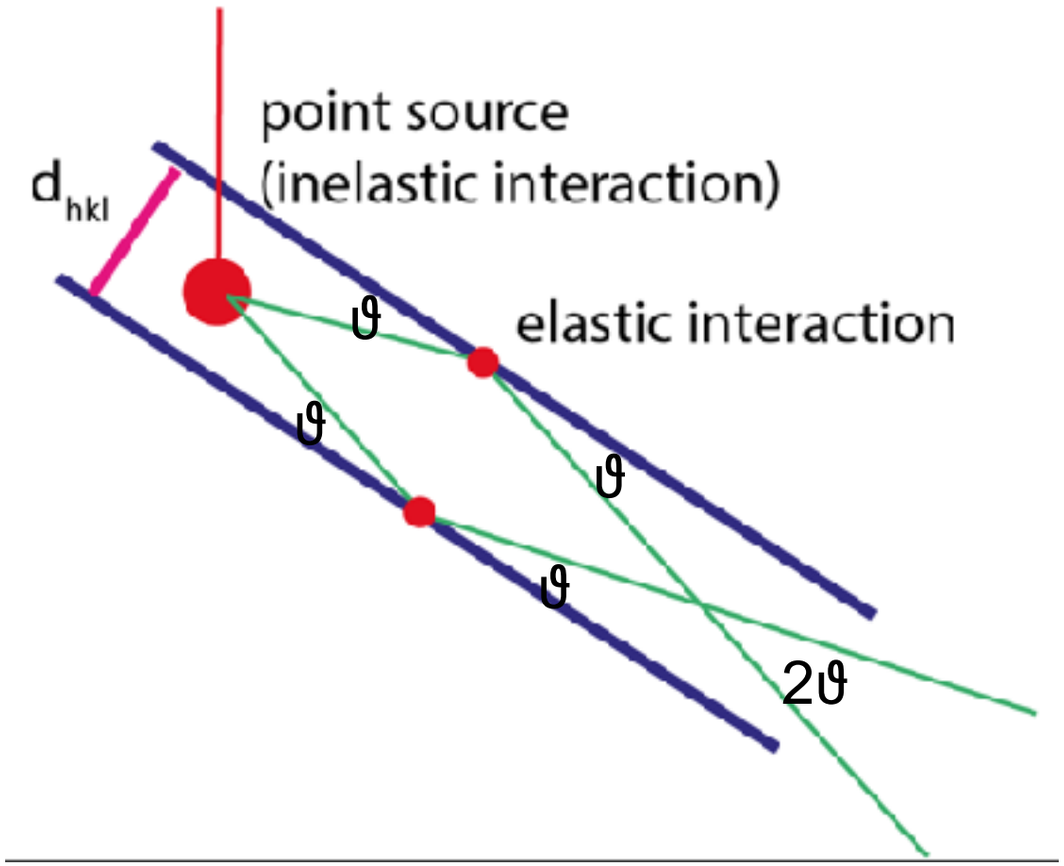
Formation of Kikuchi bands:

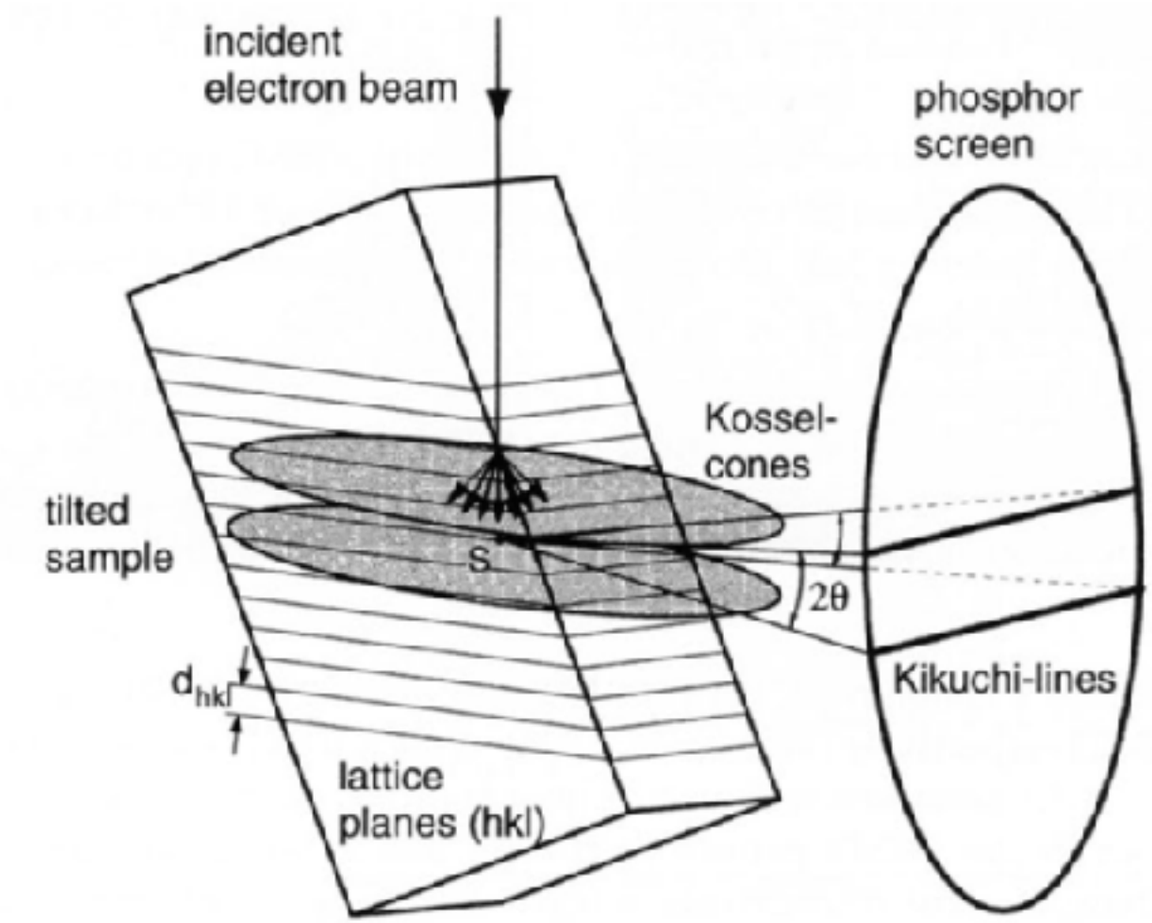


pattern: ~ gnomonic projection of crystal lattice
width of a band = $2*\vartheta_{hkl}$



Formation of Kikuchi bands:



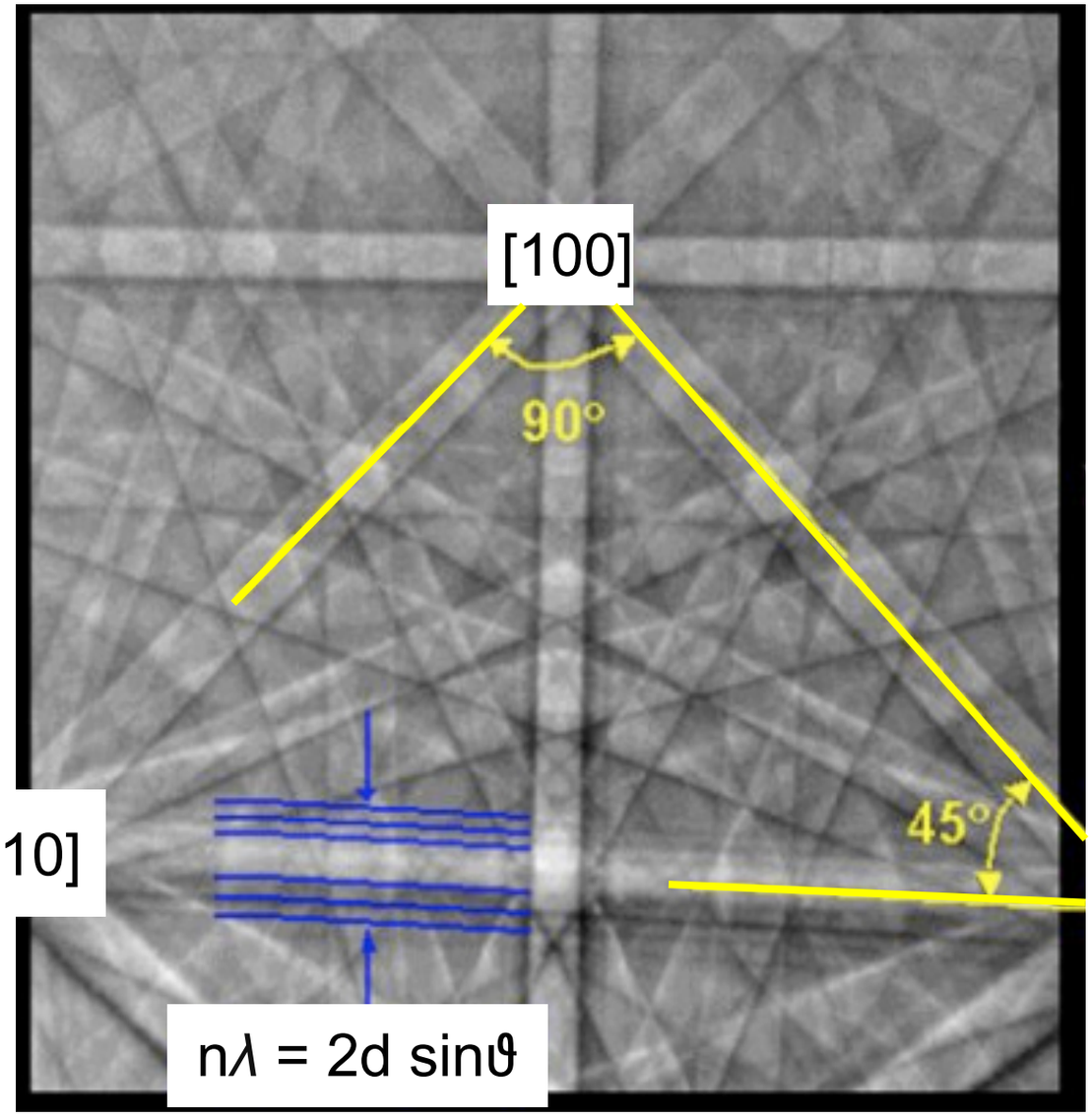


2ϑ ~ 1° (nearly straight at the screen)

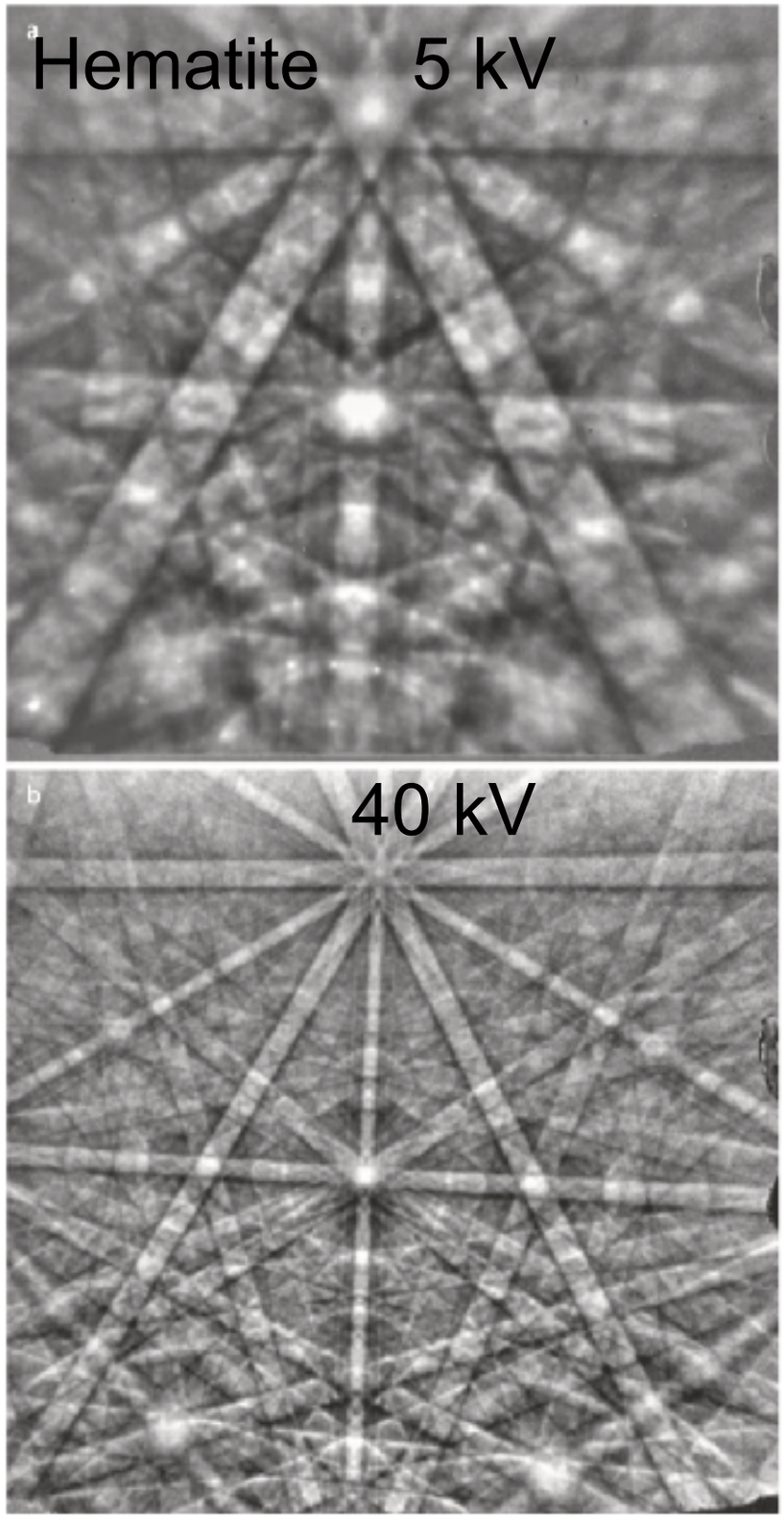
width = $2\vartheta_{hkl}$

direction of band center = lattice plane x phosphor screen

Interpretation of Kikuchi bands:



interplanar angles are relative to projection center

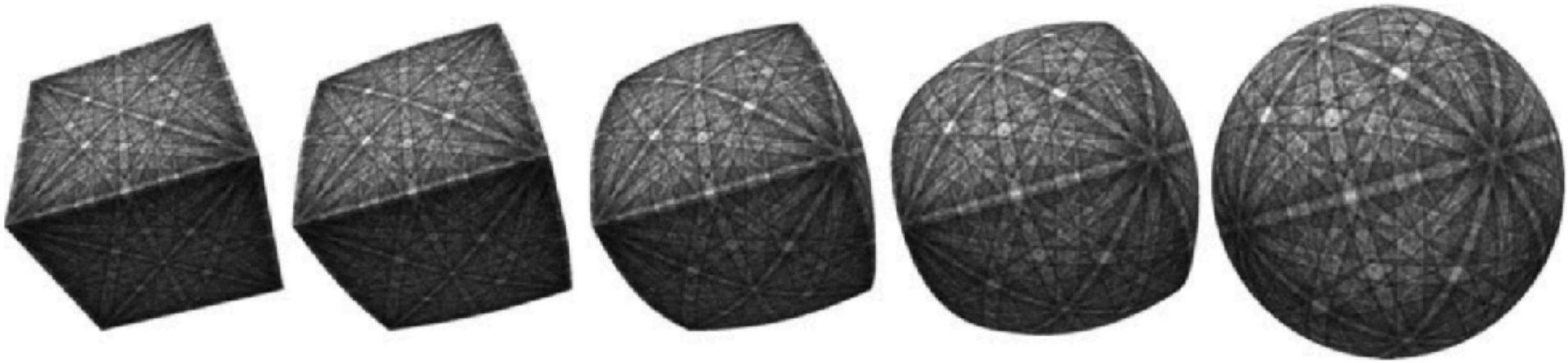


Goldstein 2018

Interpretation of Kikuchi bands:

spherical Kikuchi maps for quartz and copper, courtesy of Geoff Lloyd

Interpretation of Kikuchi bands:



cubic material - > from a cube to a spherical Kikuchi map (Day, 2009)

How does an EBSD work:

(FEG)SEM with low or high vacuum conditions:
beam or stage mapping (or combined for large area mapping)
SEM setup 10-25 kV, 5-40 nA (!)
step sizes: ~(2)30 nm to >100 µm
indexing: ~10-100 pts/s (for silicates) / 1000s for metals

band identification through Hough/Radon transform or better methods

solving bands against a known material file

optional:
EDX: counts at 40 pts/s are rather low but sometimes sufficient for phase identification
exporting pattern: enable possibility for offline re-indexing or other exciting methods (cross correlation, dictionary indexing, fore-scatter simulation, identifying unknown phases ...)

Geometry of an EBSD system

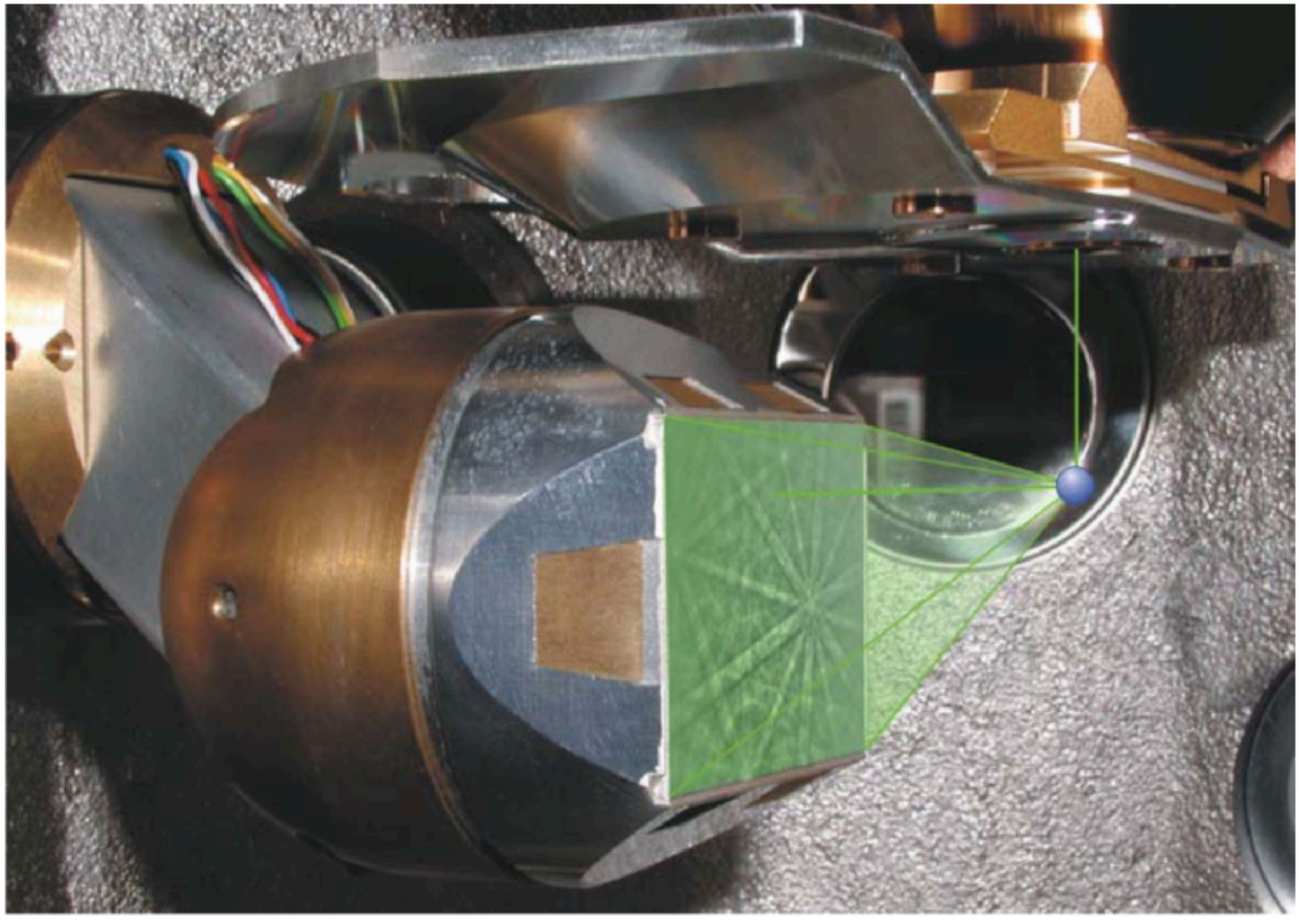
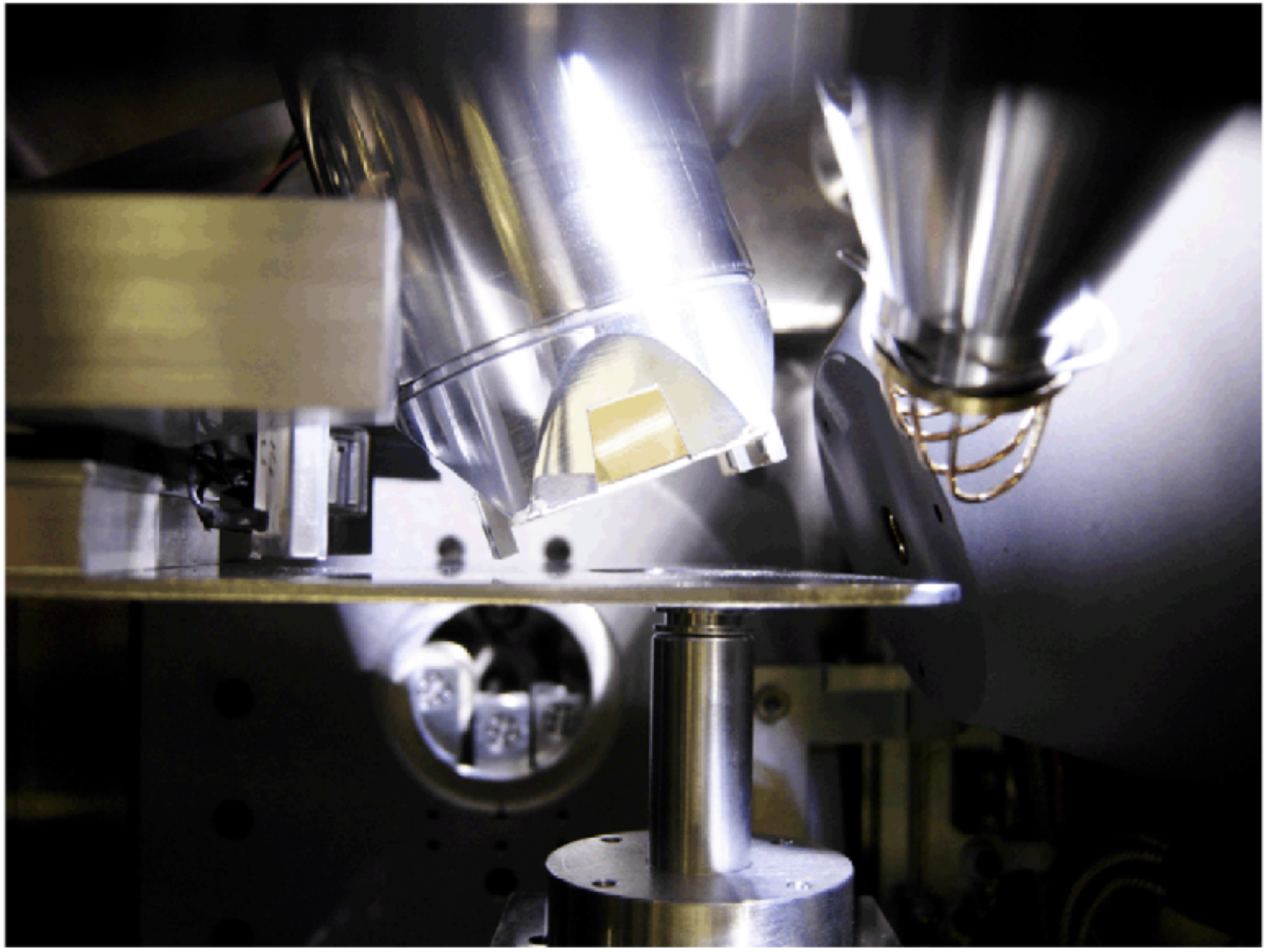
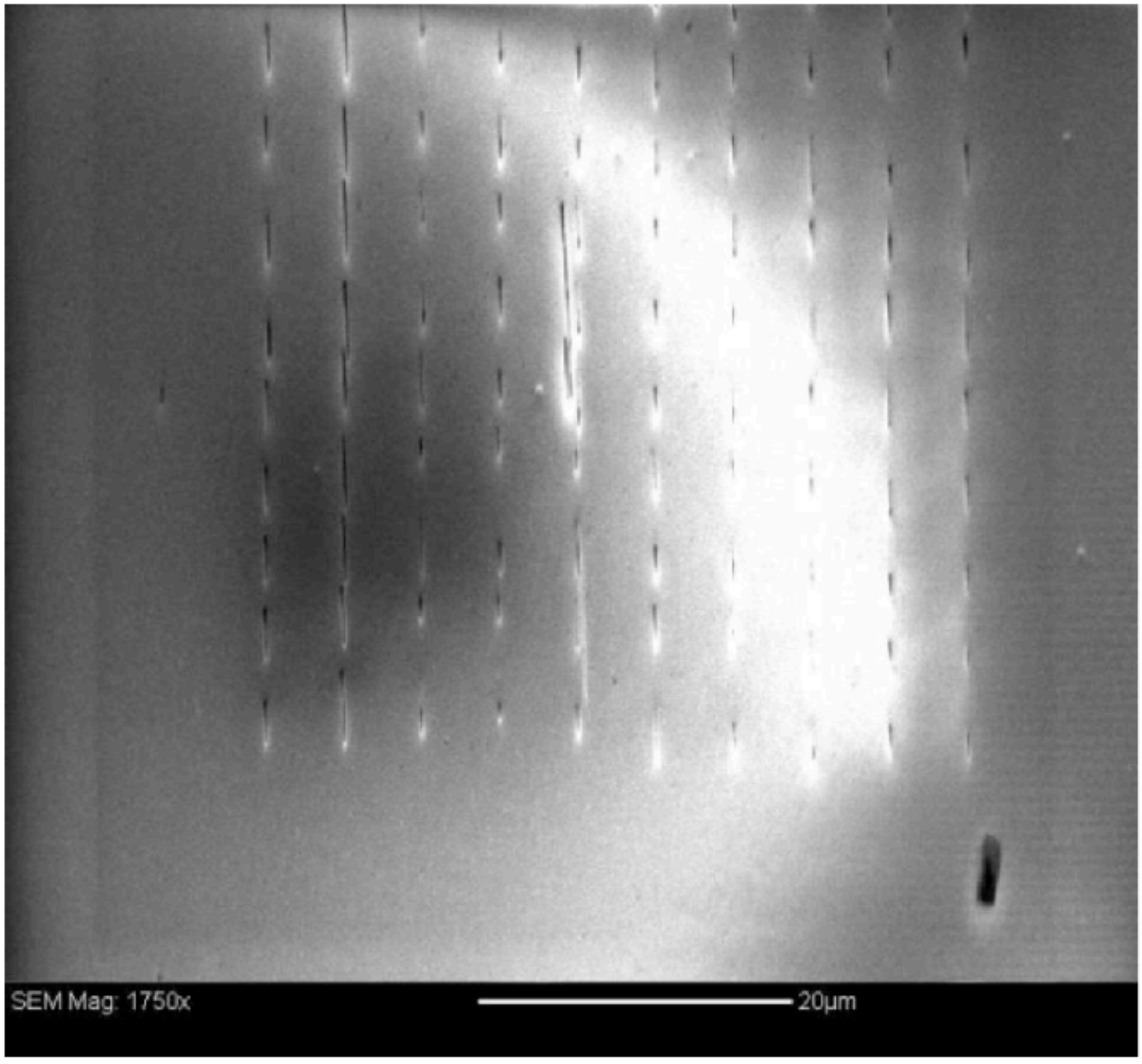


Image courtesy of John Bonevich and Mark Vaudin



tilted gun setup
(D. Mainprice)

Sample coating?



Post-Acquisition SE on ice, Gill Pennock

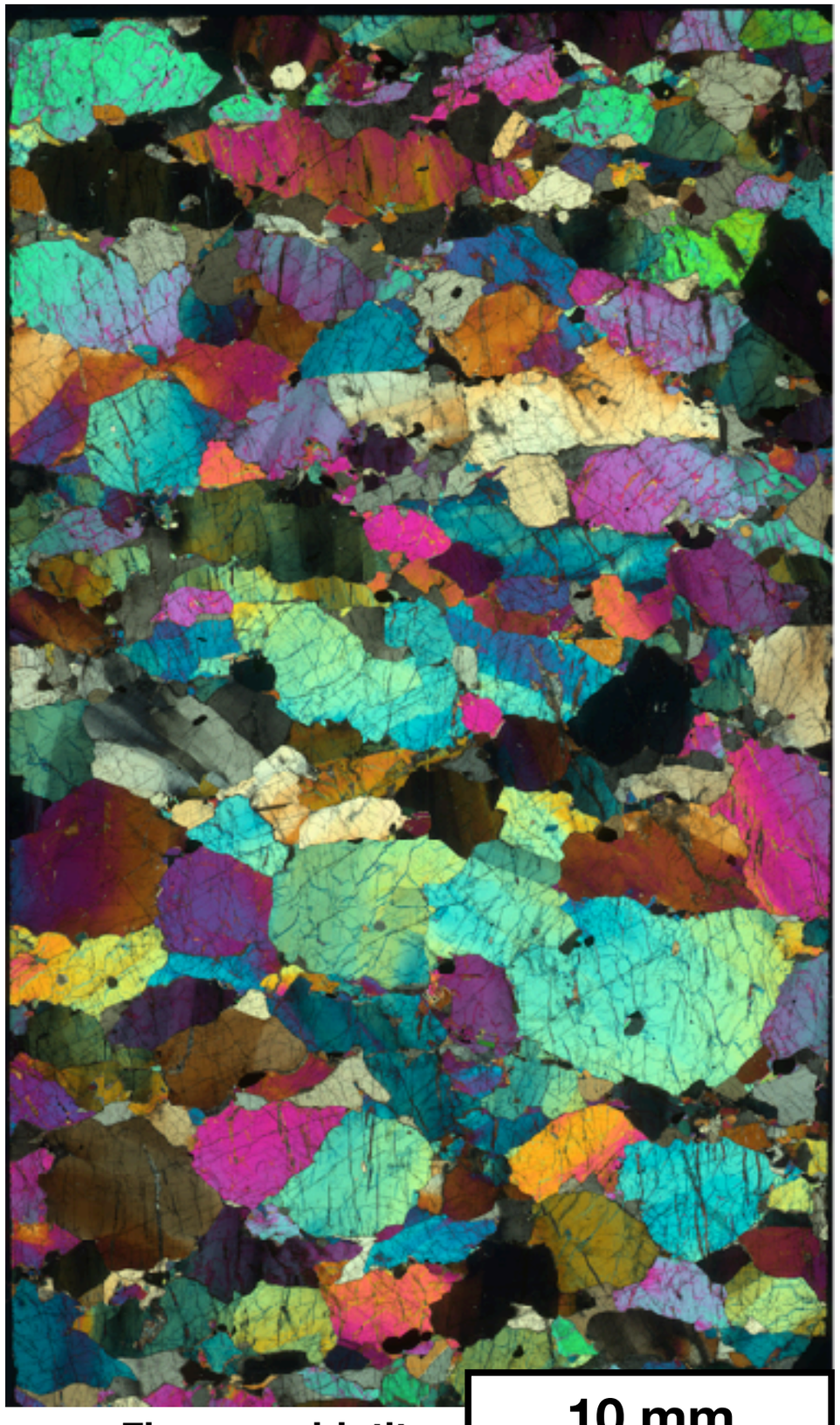
Sample coating for non-conductors?

no coating -> charging

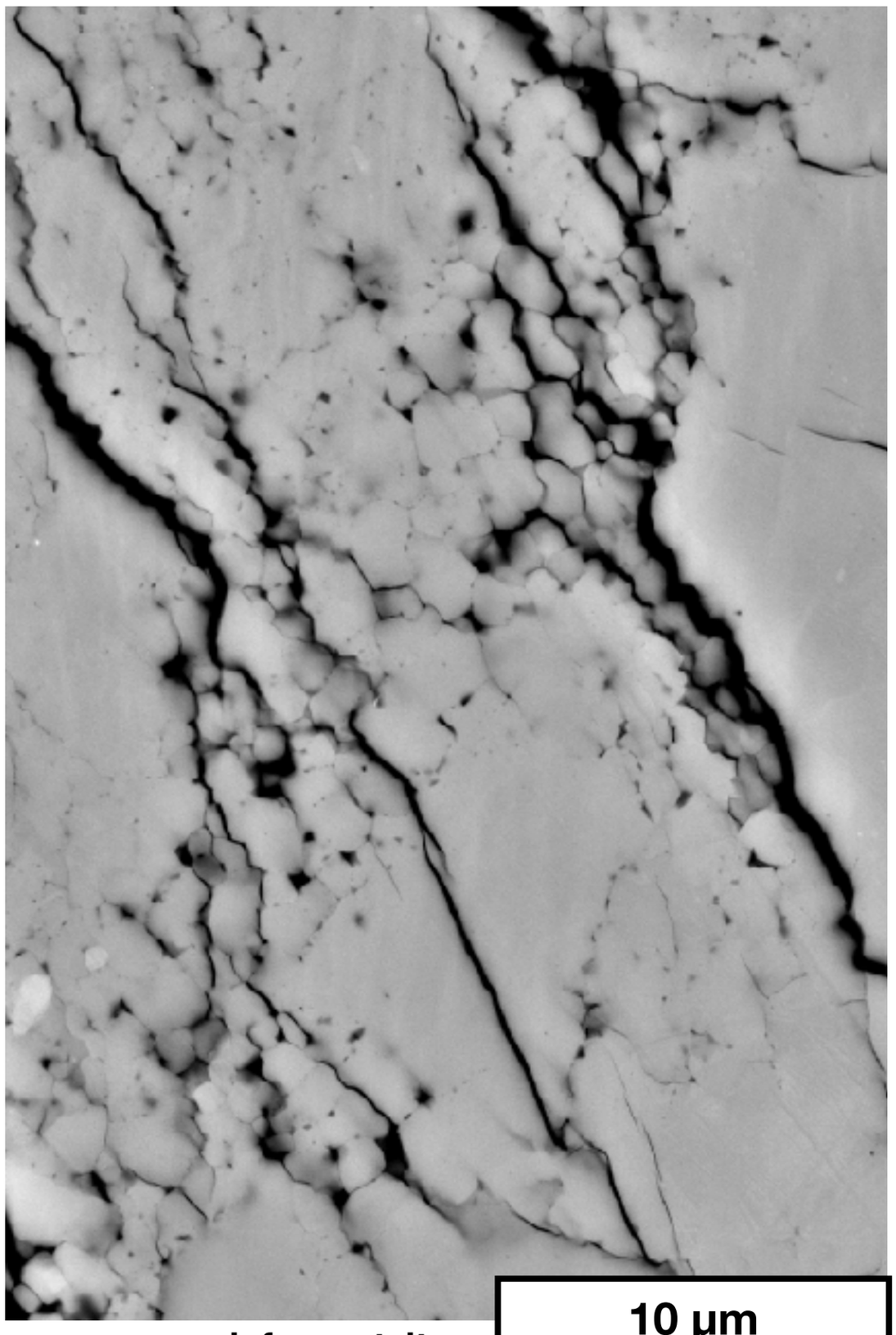
low vacuum / no coating
- gas removes charging
- gas spreads beam/BSE
- gas decreases EBSD quality
- charging often suppressed at P>30Pa (if VP is available)

high vacuum / thin coating (~10Å C)
- no beam spread
- no charging
- coating degrades pattern quality

More considerations prior to data acquisition: resolution

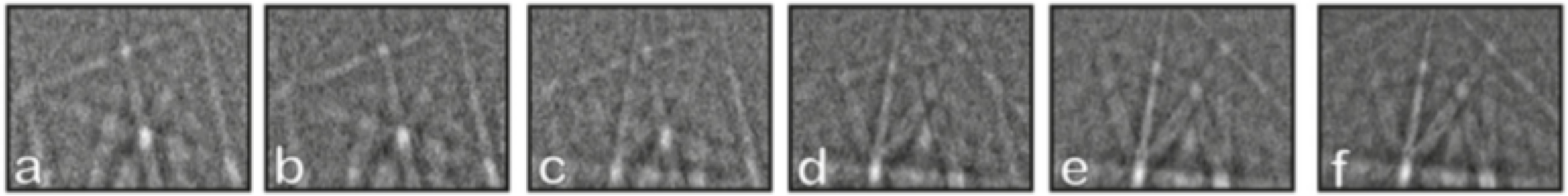


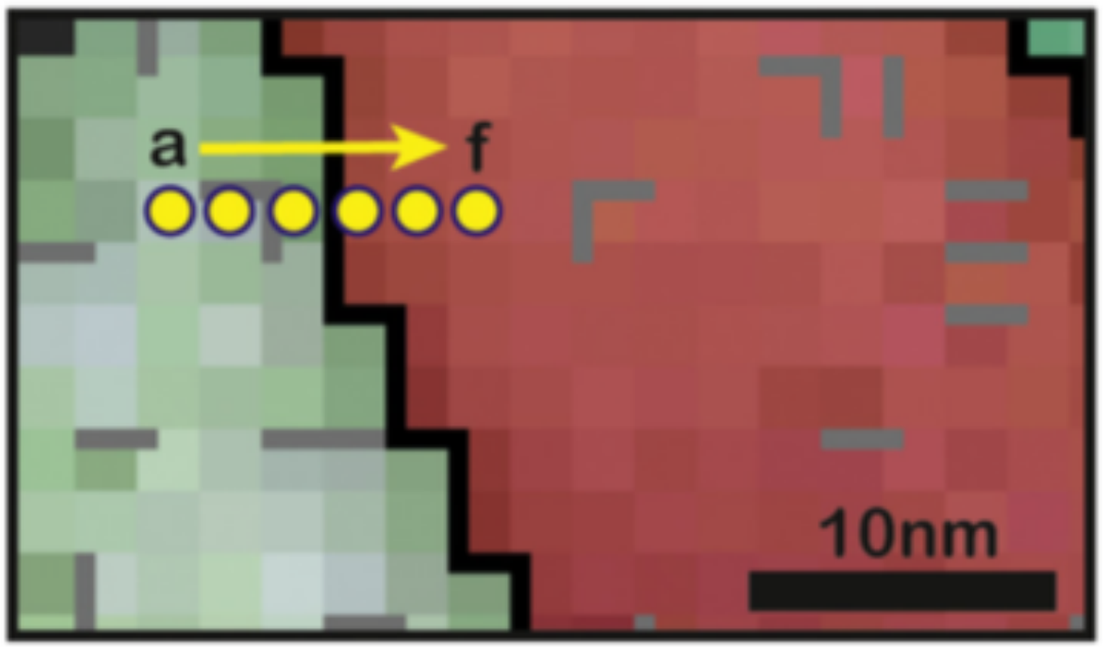
Finero peridotite

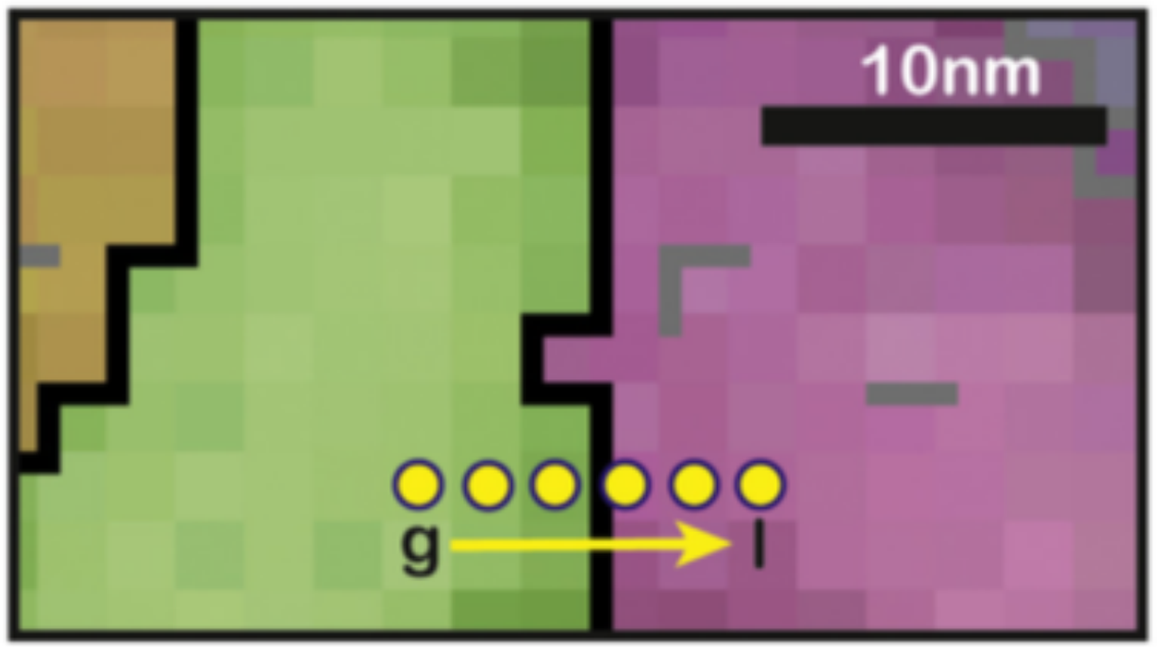


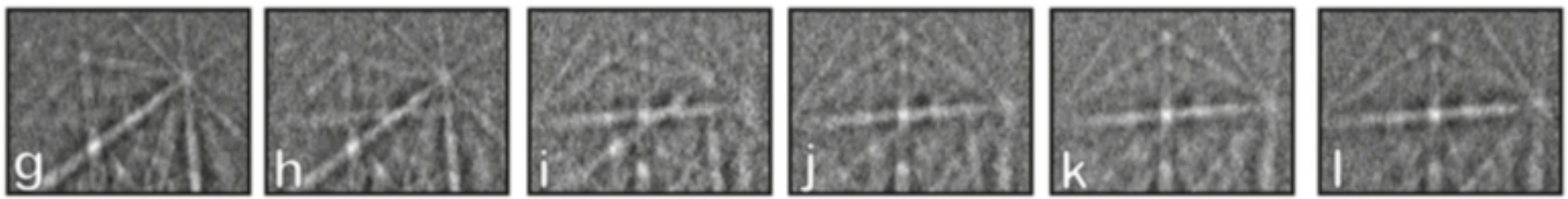
exp. def. quartzite

Very high resolution: Transmission Kikuchi diffraction







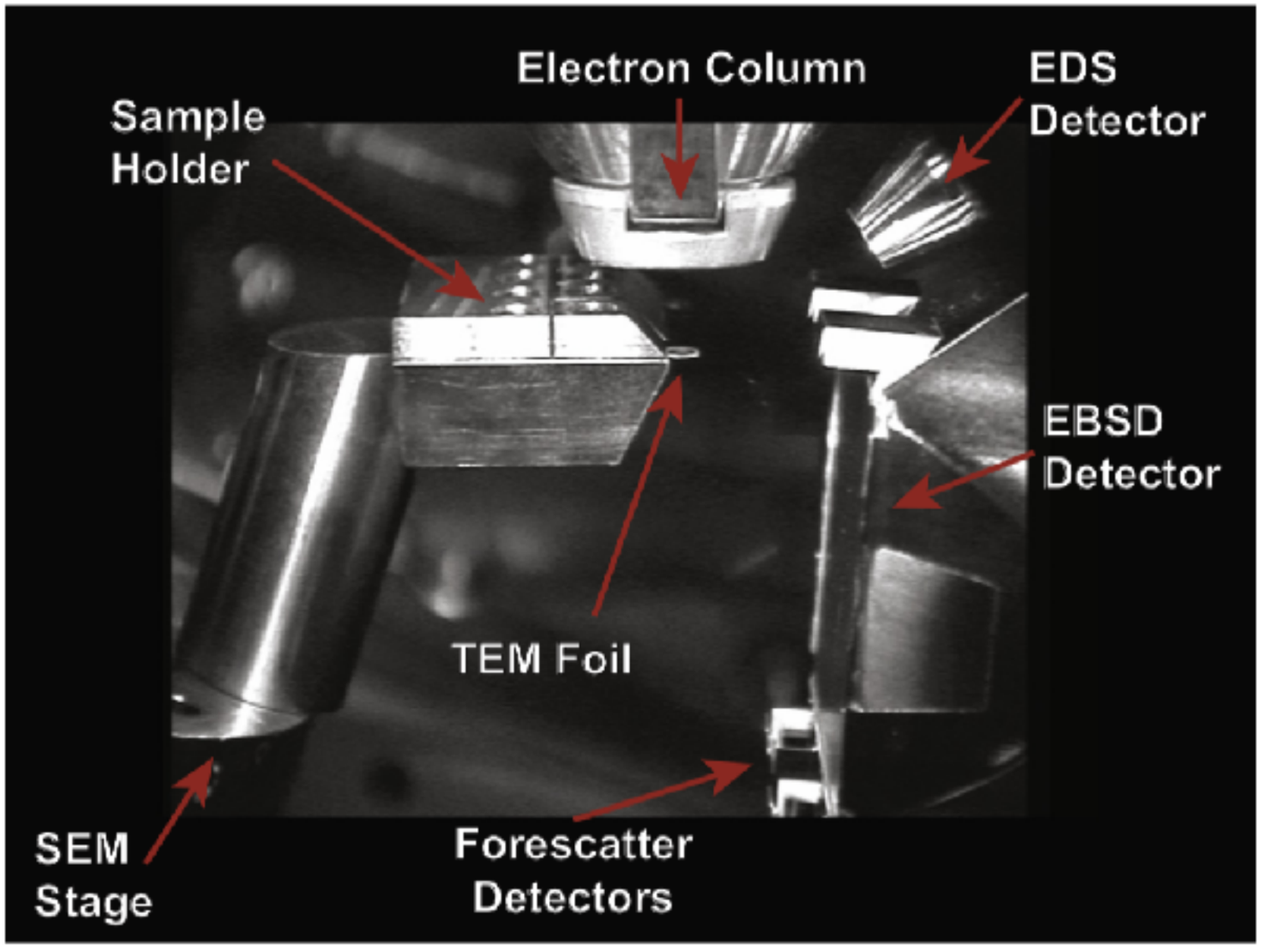


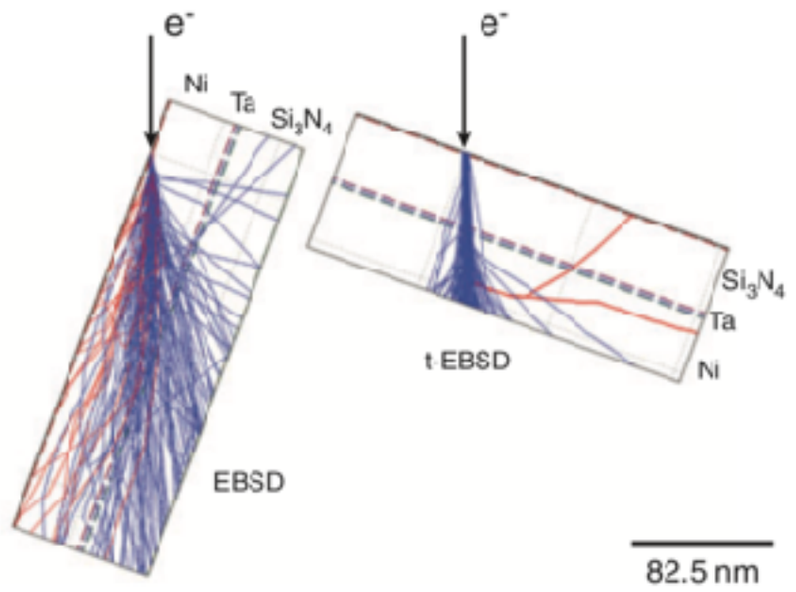
100-200nm foil

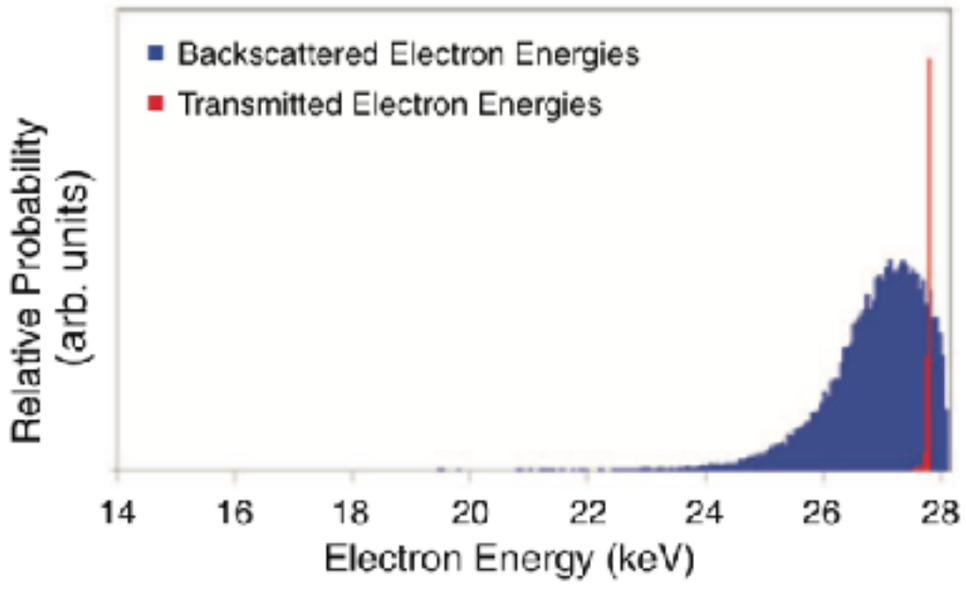
2 nm step size

Trimby, 2012

Very high resolution: Transmission Kikuchi diffraction



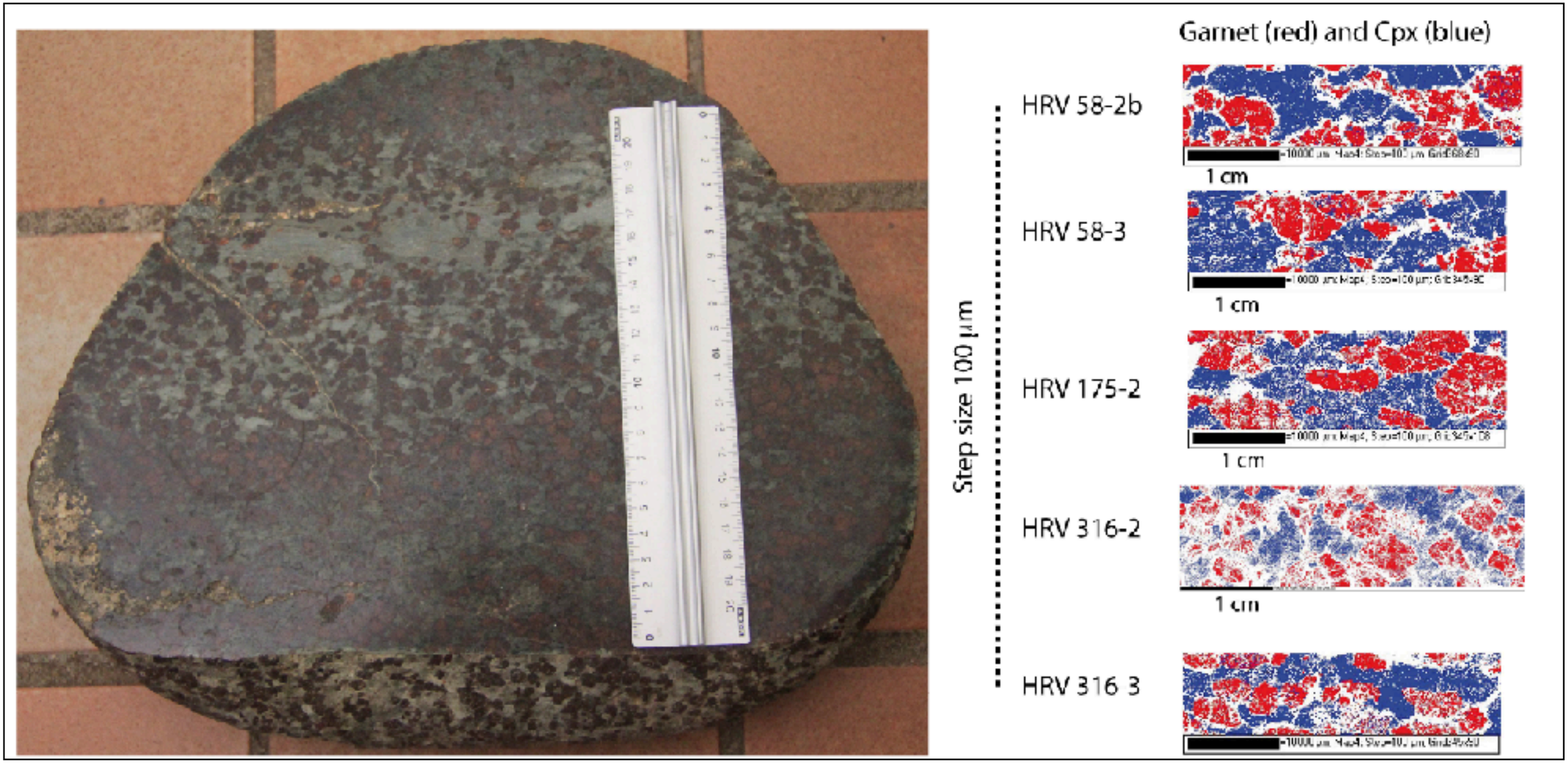




Keller & Geiss 2012

- small sample size
- complicated sample preparation
- on a nice FEGSEM < 50 nm possible for quartz

Very large area: stitching and/or a large chamber

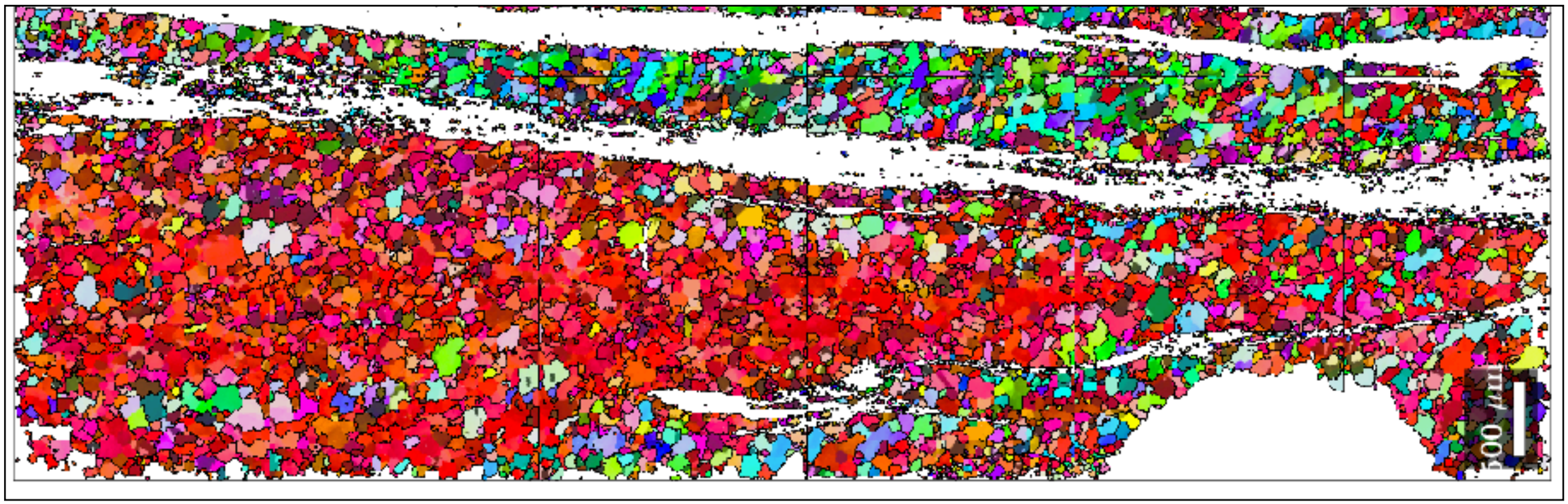


step size = 100 µm

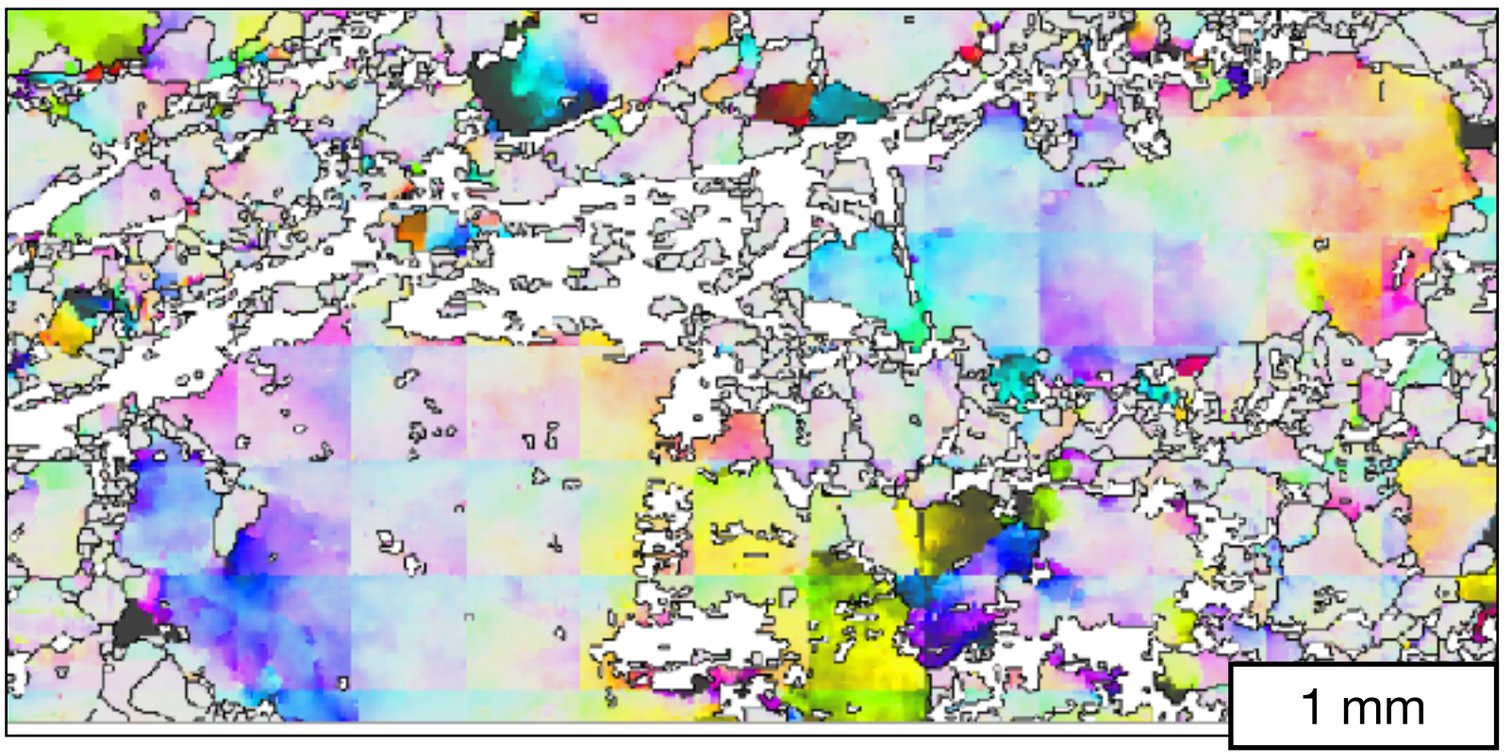
image courtesy of David Mainprice

Very large area: stitching and/or a large chamber

useless stitching



stitching ok, but orientation distortion

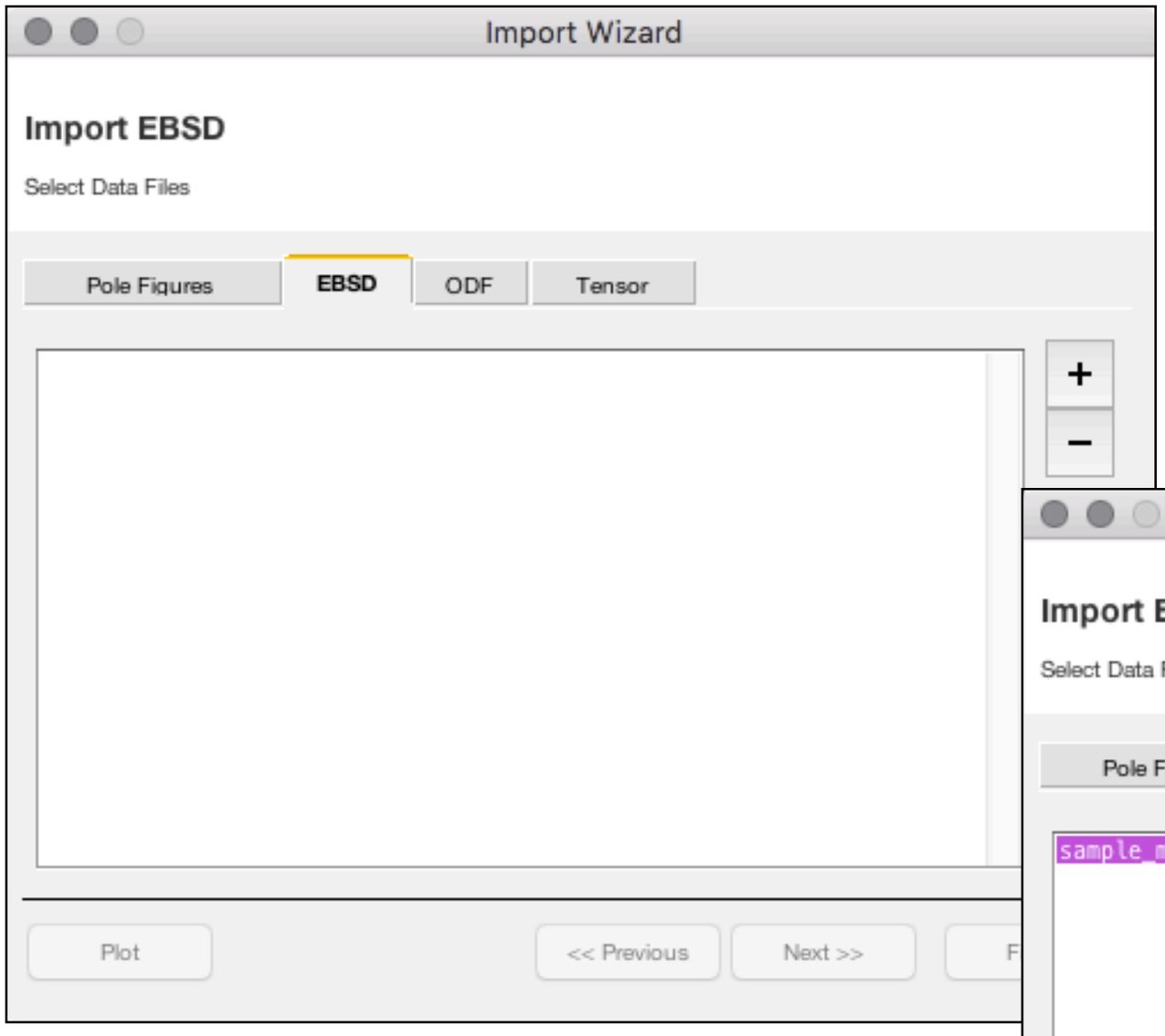


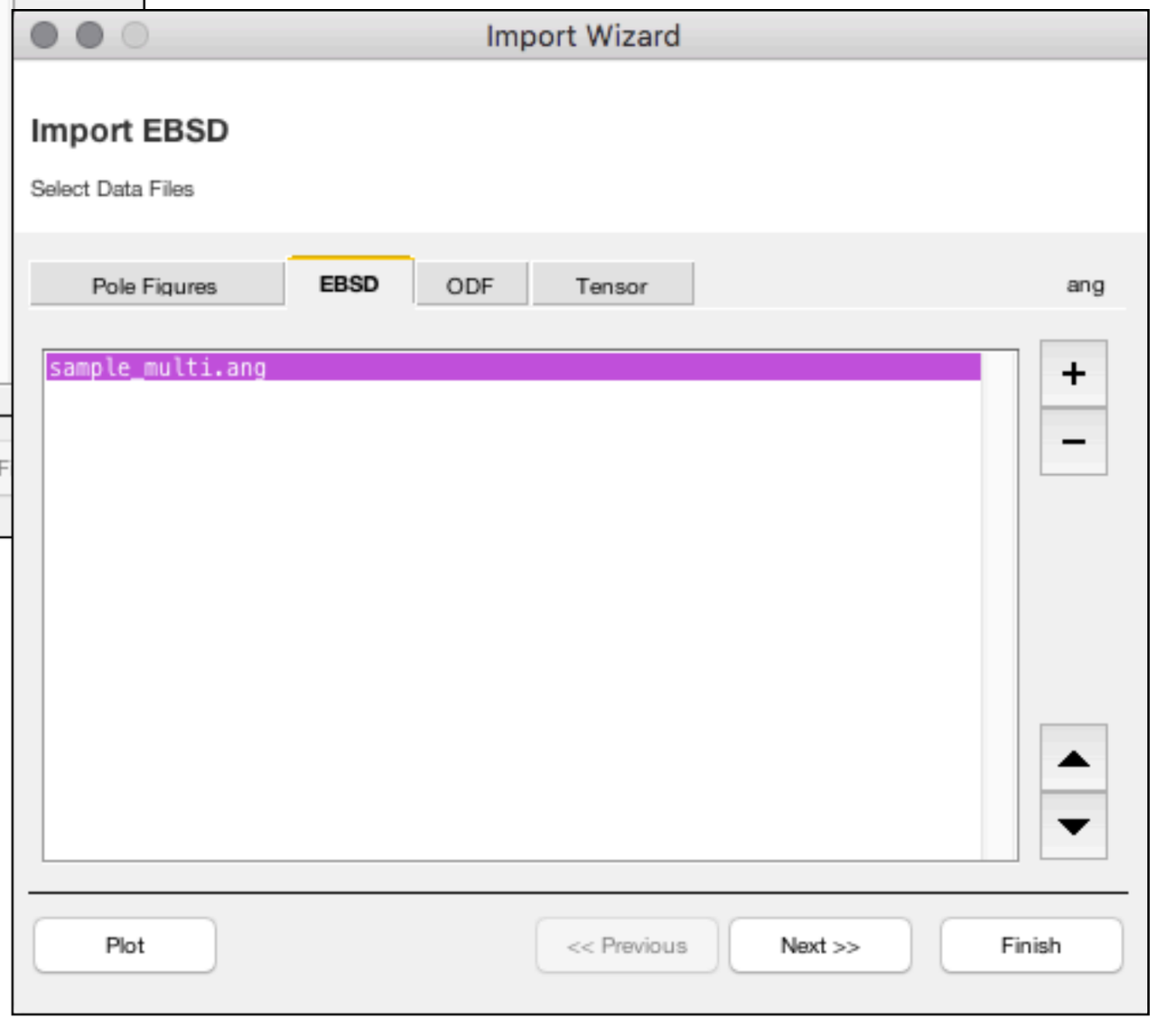
Importing EBSD data into MTEX

currently available interfaces:

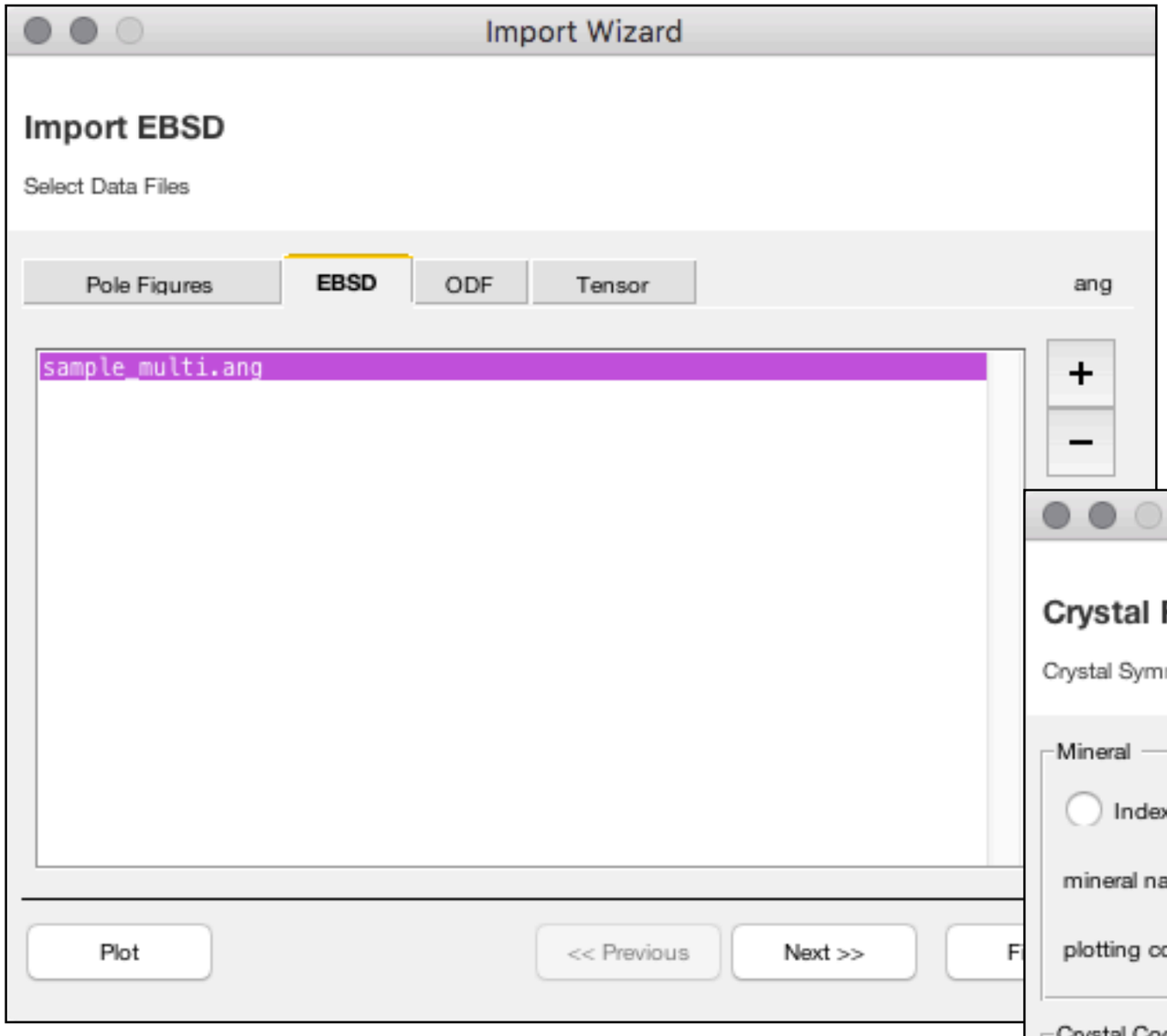
```
loadEBSD_ACOM.m
loadEBSD_ang.m
loadEBSD_brukertxt.m
loadEBSD_crc.m
loadEBSD_csv.m
loadEBSD_ctf.m
loadEBSD_ebsdimage.m
loadEBSD_dream3d.m
loadEBSD_h5.m
loadEBSD_hdf5.m
loadEBSD_osc.m
loadEBSD_Oxfordcsv.m
loadEBSD_sor.m

loadEBSD_generic.m
```

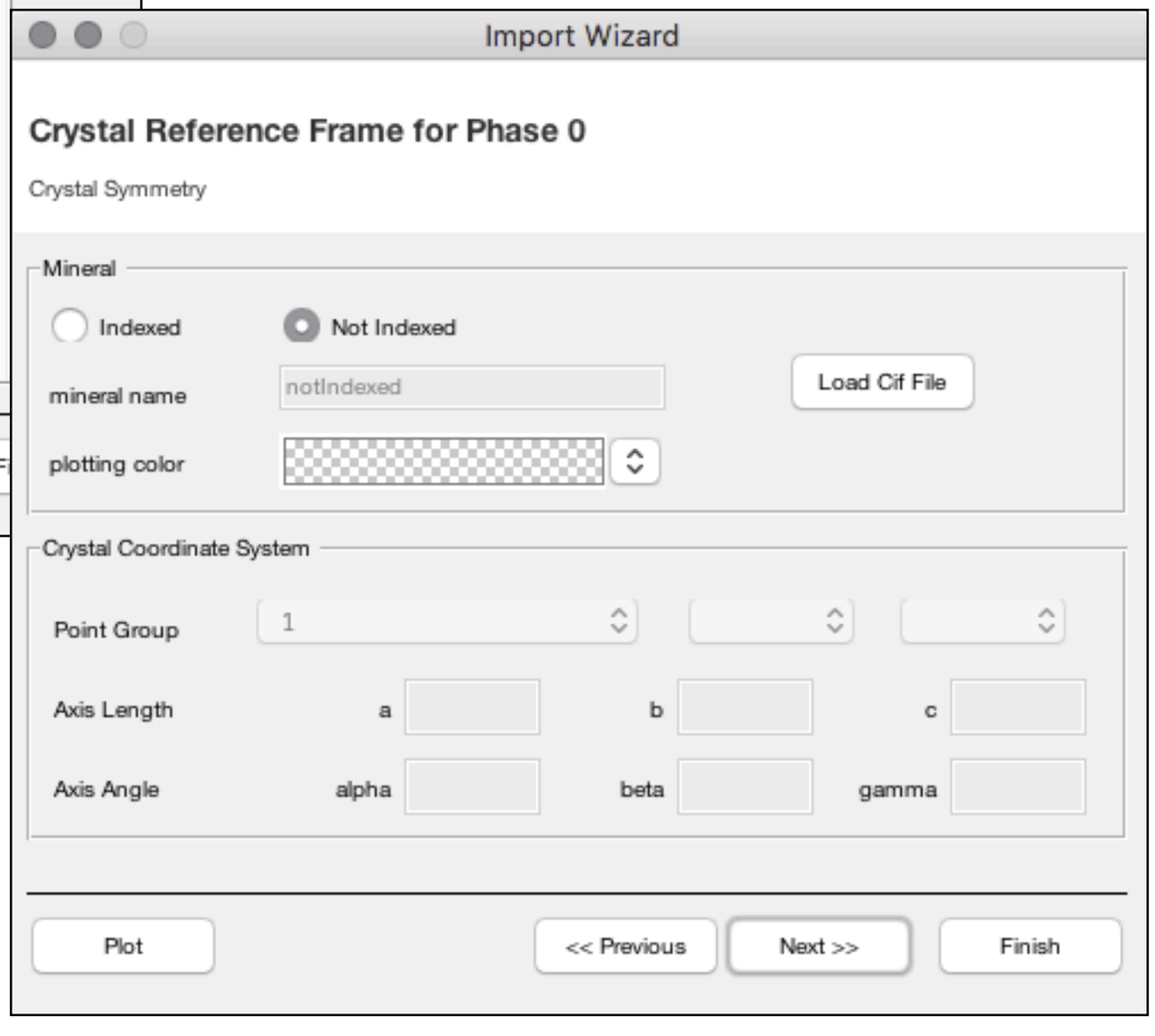
Importing EBSD data into MTEX





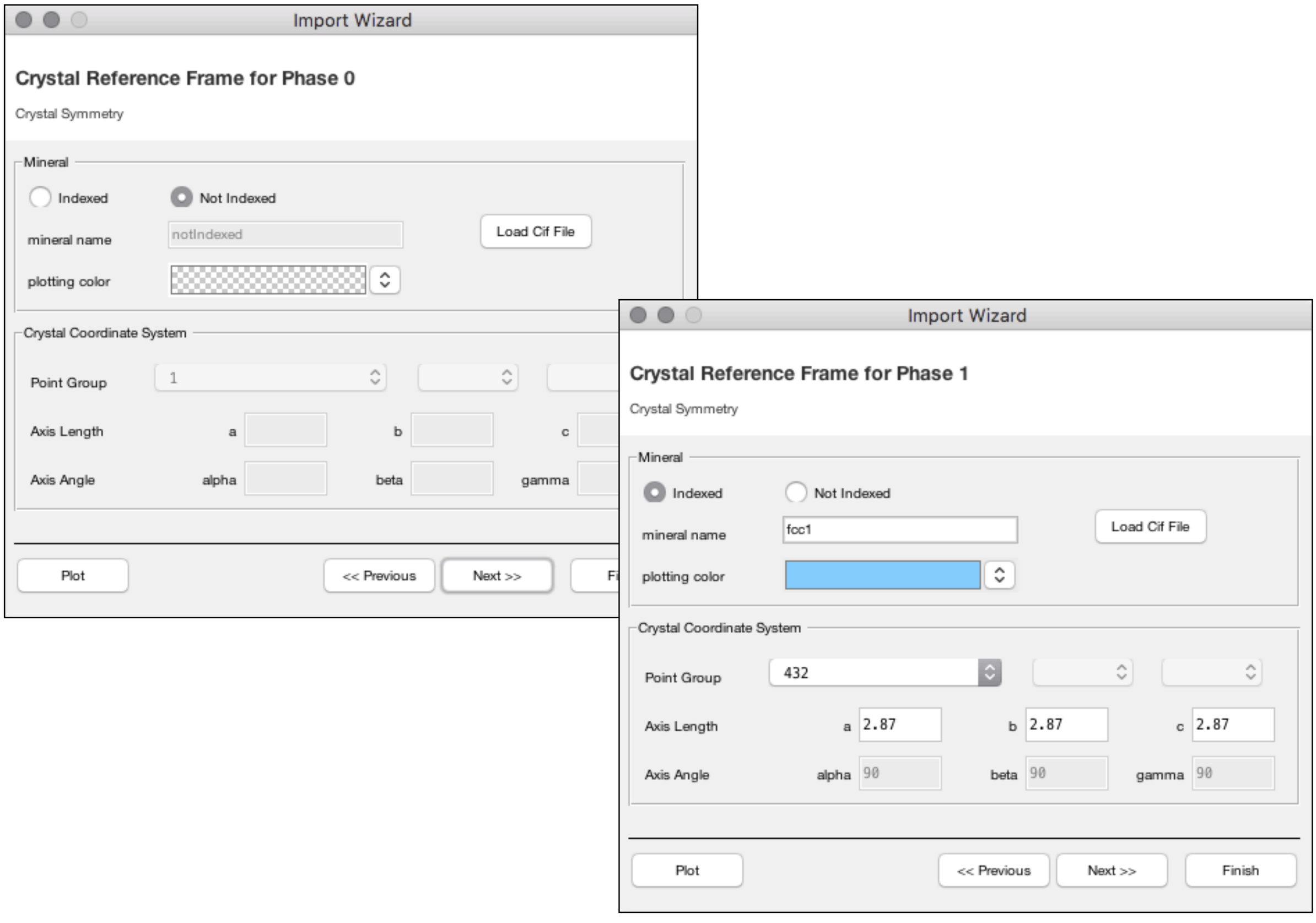
Importing EBSD data into MTEX



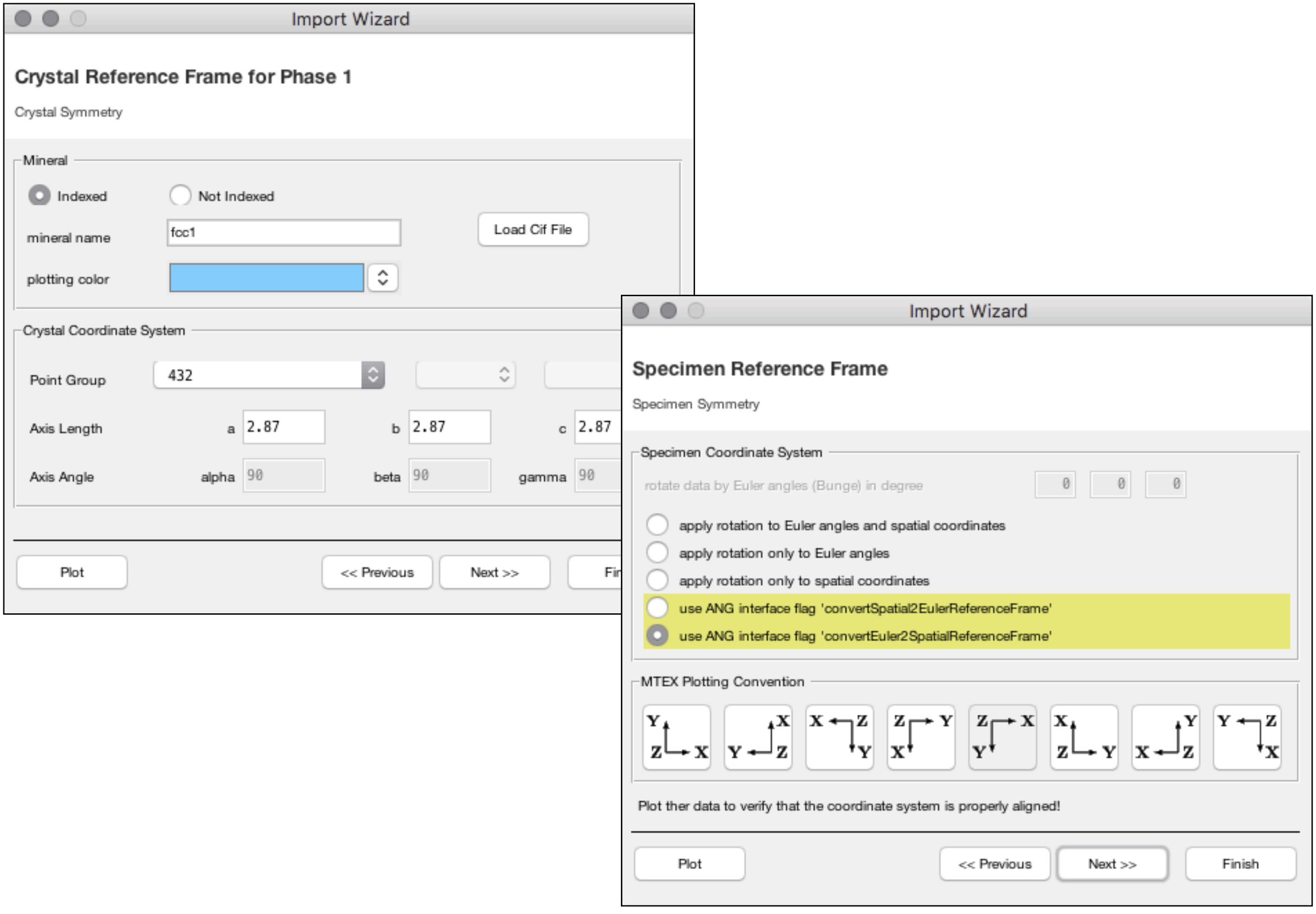


* in MTEX, there are two types of datapoints which do not appear in the map, more on that later

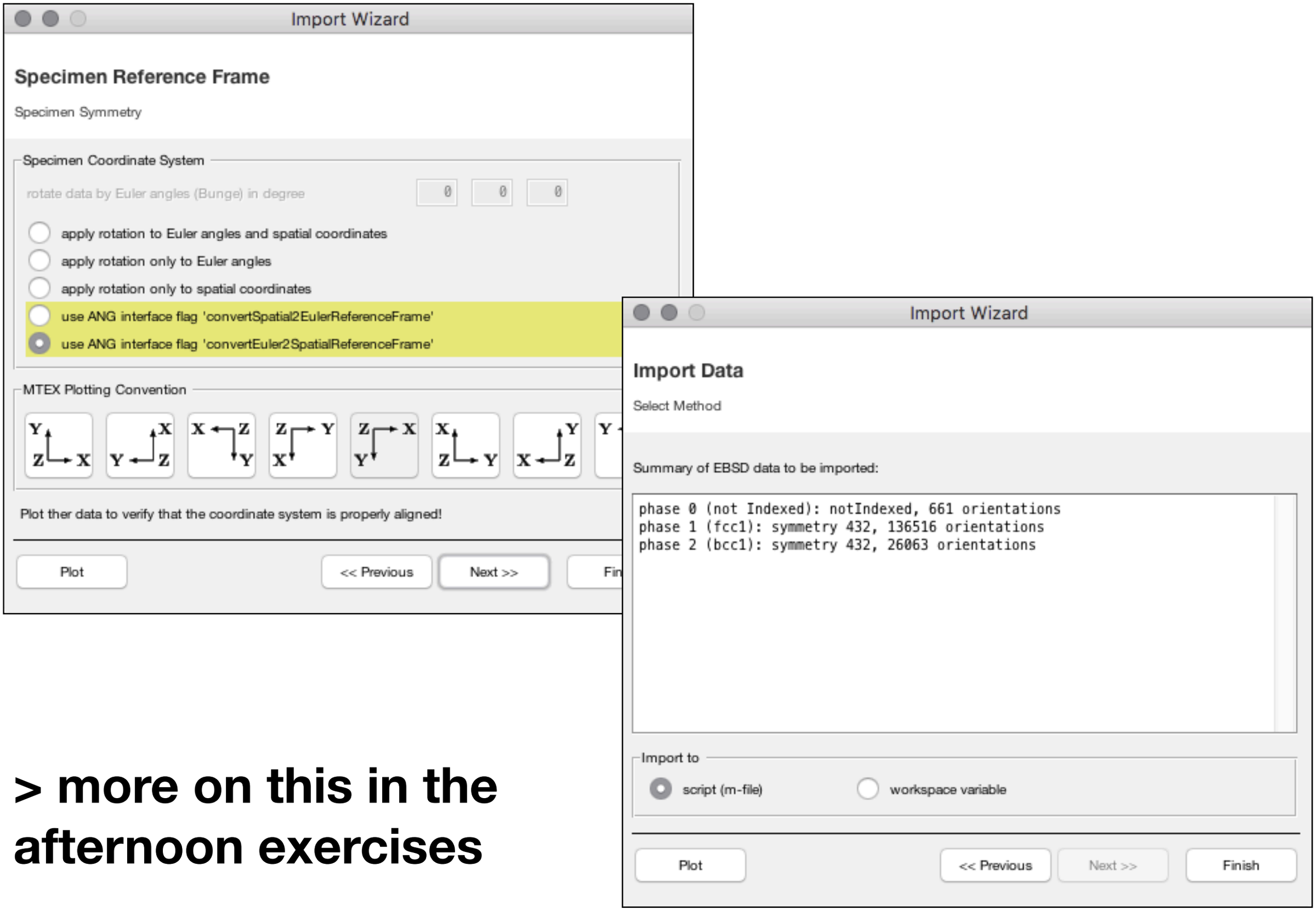
Importing EBSD data into MTEX



Importing EBSD data into MTEX



Importing EBSD data into MTEX



> more on this in the afternoon exercises

how does the ebsd object look like

```
>> ebsd
ebsd = EBSD (show methods, plot)
 Phase  Orientations       Mineral        Color  Symmetry  Crystal reference frame
     1   44953 (90%)       olivine   light blue       222
     2   1370 (2.8%)      Dolomite  light green         3        X||a, Y||b*, Z||c
     3   2311 (4.6%)     Enstatite    light red       222
     4   1095 (2.2%)  Chalcopyrite         cyan       422

 Properties: ci, fit, iq, sem_signal, unknown1, unknown2, unknown3, unknown4, x, y
 Scan unit : um
```

```
>> ebsd.prop
ans =
  struct with fields:

            ci: [49729×1 double]
           fit: [49729×1 double]
            iq: [49729×1 double]
    sem_signal: [49729×1 double]
      unknown1: [49729×1 double]
      unknown2: [49729×1 double]
      unknown3: [49729×1 double]
      unknown4: [49729×1 double]
             x: [49729×1 double]
             y: [49729×1 double]
```

select ebsd by phase:

```
>> ebsd
ebsd = EBSD (show methods, plot)
 Phase  Orientations       Mineral        Color  Symmetry  Crystal reference frame
     1   44953 (90%)       olivine   light blue       222
     2   1370 (2.8%)      Dolomite  light green         3        X||a, Y||b*, Z||c
     3   2311 (4.6%)     Enstatite    light red       222
     4   1095 (2.2%)  Chalcopyrite         cyan       422

 Properties: ci, fit, iq, sem_signal, unknown1, unknown2, unknown3, unknown4, x, y
 Scan unit : um
```

```
>> ebsd('Chalcopyrite')
ans = EBSD (show methods, plot)
 Phase  Orientations       Mineral  Color  Symmetry  Crystal reference frame
     4   1095 (100%)  Chalcopyrite   cyan       422
…
```

```
ebsd('Chalcopyrite')
ebsd('Chalco')
ebsd('C')
```

Note: multiply defined abbreviations: e.g. Ti-alpha and Ti-beta ebsd(’Ti’) will obviously select two phases!

select ebsd by phase:

```
>> ebsd('Chalcopyrite')

ans = EBSD (show methods, plot)
 Phase  Orientations       Mineral  Color  Symmetry  Crystal reference frame
     4   1095 (100%)  Chalcopyrite   cyan       422

 Properties: ci, fit, iq, sem_signal, unknown1, unknown2, unknown3, unknown4, x, y
 Scan unit : um


>> ebsd('C').phase
4
4
4
...


>> ebsd(ebsd.phase==4)
ans = EBSD (show methods, plot)
 Phase  Orientations       Mineral  Color  Symmetry  Crystal reference frame
     4   1095 (100%)  Chalcopyrite   cyan       422
```

select ebsd by logical index / property

```
>> ebsd.ci(1:10)
ans =
    0.4500
    0.4910
    0.6150
    0.2630
    0.4250
    0.2810
...
```

```
>> ebsd.ci(1:10)>0.3
ans = 10×1 logical array
   1
   1
   1
   0
   1
   0
...
```

```
>> ebsd(ebsd.ci>0.5)
ans = EBSD (show methods, plot)
 Phase  Orientations    Mineral        Color  Symmetry  Crystal reference frame
     1   21350 (99%)    olivine   light blue       222
     2   131 (0.61%)   Dolomite  light green         3        X||a, Y||b*, Z||c
     3    5 (0.023%)  Enstatite    light red       222

...
```

```
>> ebsd(ebsd.ci>0.5 & ebsd.phase==1)

ans = EBSD (show methods, plot)

 Phase  Orientations  Mineral       Color  Symmetry  Crystal reference frame
     1  21350 (100%)  olivine  light blue       222

...
```

select ebsd by phase:

```
>> ebsd({'o' 'e'})

ans = EBSD (show methods, plot)

 Phase  Orientations    Mineral       Color  Symmetry  Crystal reference frame
     1   44953 (95%)    olivine  light blue       222
     3   2311 (4.9%)  Enstatite   light red       222

 Properties: ci, fit, iq, sem_signal, unknown1, unknown2, unknown3, unknown4, x, y
 Scan unit : um
```

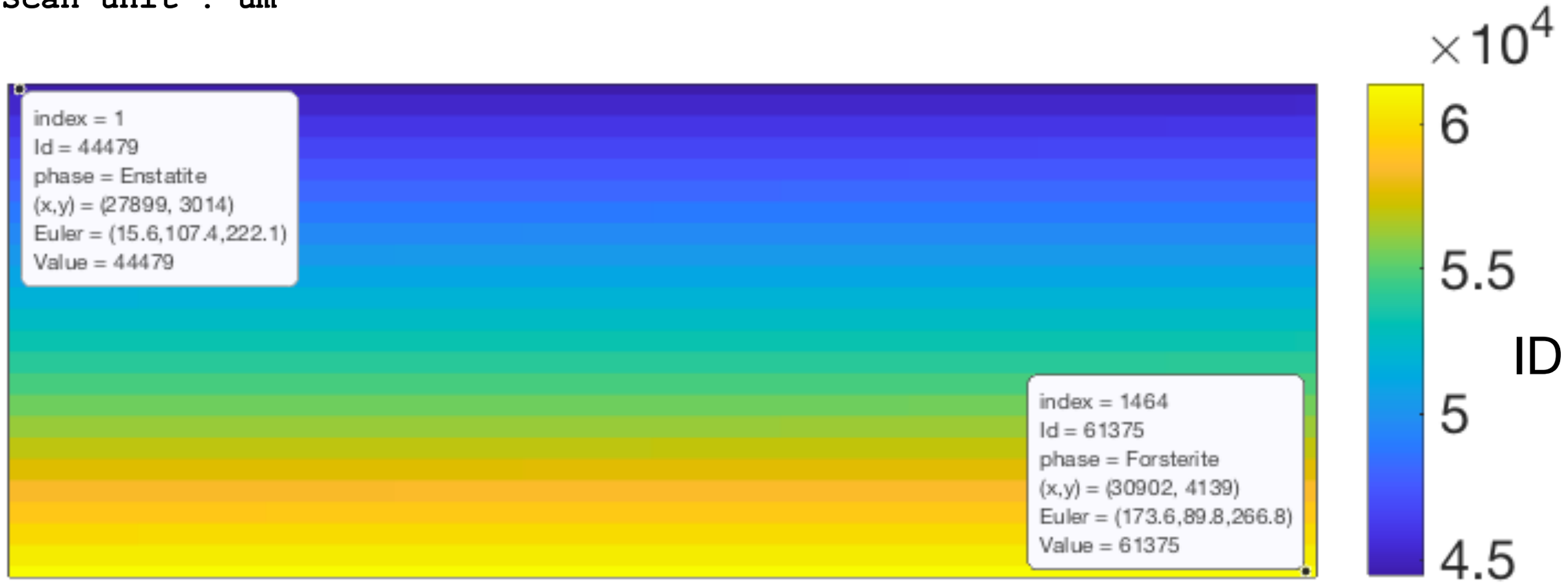
but!

```
>> ebsd({'o' 'e'}).orientations
Error using phaseList/checkSinglePhase (line 278)

------------------------------------------------------------
Your variable contains the phases: olivine, Enstatite

However, your are executing a command that is only permitted for a single phase!

Please see modify EBSD data for how to restrict EBSD data or grains to a single
phase.
------------------------------------------------------------
```

ebsd concepts in MTEX : id/index

id: once assigned during import (ebsd{...} or ebsd('id',..)
index: sequential number in list (ebsd(...))

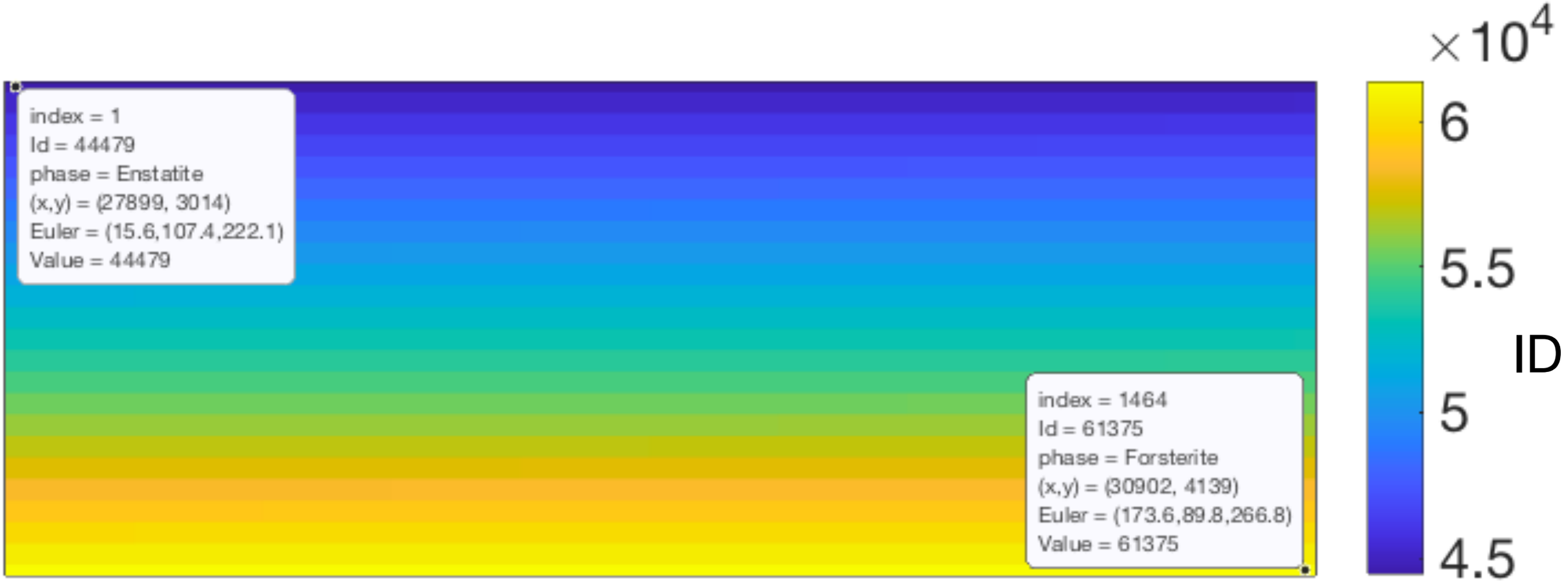
```
>> ebsd{44479}
ans = EBSD (show methods, plot
 Phase  Orientations    Mineral          Color  Symmetry  Crystal reference frame
     2      1 (100%)  Enstatite  light green       mmm
    Id   Phase   phi1    Phi   phi2   bands   bc    bs   error   mad       x      y
 44479       2     16   107    222       7   86   141       0   0.7   27900   3000
Scan unit : um
```



ebsd concepts in MTEX : id/index

id: once assigned during import (ebsd{...} or ebsd('id',..)
index: sequential number in list (ebsd(...))

```
>> ebsd(1)
ans = EBSD (show methods, plot
 Phase  Orientations    Mineral         Color  Symmetry  Crystal reference frame
     2       1 (100%)  Enstatite  light green       mmm
    Id   Phase   phi1    Phi   phi2   bands   bc    bs   error   mad        x       y
 44479       2     16   107    222       7   86   141       0   0.7    27900    3000
 Scan unit : um
```



ebsd concepts in MTEX : id/index

Why might id and index not be the same?

- crop from a larger map, deleted points ...
- common source of errors

Sometimes it's useful to reset the id

```
>> ebsd.id= id2ind(ebsd,ebsd.id)
```

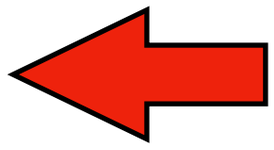
```
>> ebsdc{1}

ans = EBSD (show methods, plot)

 Phase  Orientations    Mineral        Color  Symmetry  Crystal reference frame
     2      1 (100%)  Enstatite  light green       mmm

 Id   Phase   phi1   Phi   phi2   bands   bc    bs   error   mad       x      y
  1       2     16   107    222       7   86   141       0   0.7   27900   3000
 Scan unit : um
```

In MTEX, there are two types of datapoints which do not appear in the map

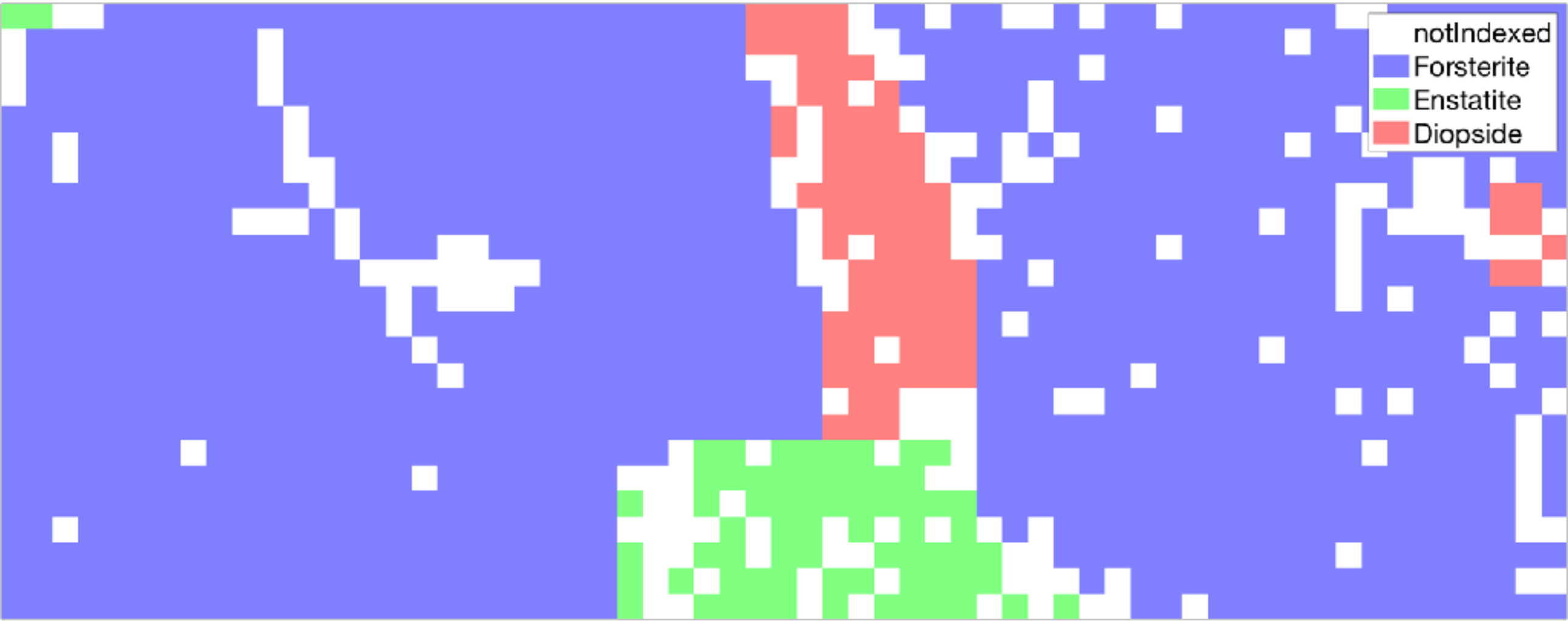
1) explicitly "notIndexed"
   - think of them as a phase
   - known to be not indexed

2) points which are just not there
   - points missing in the list
   - can be data format
   - user choice to delete points

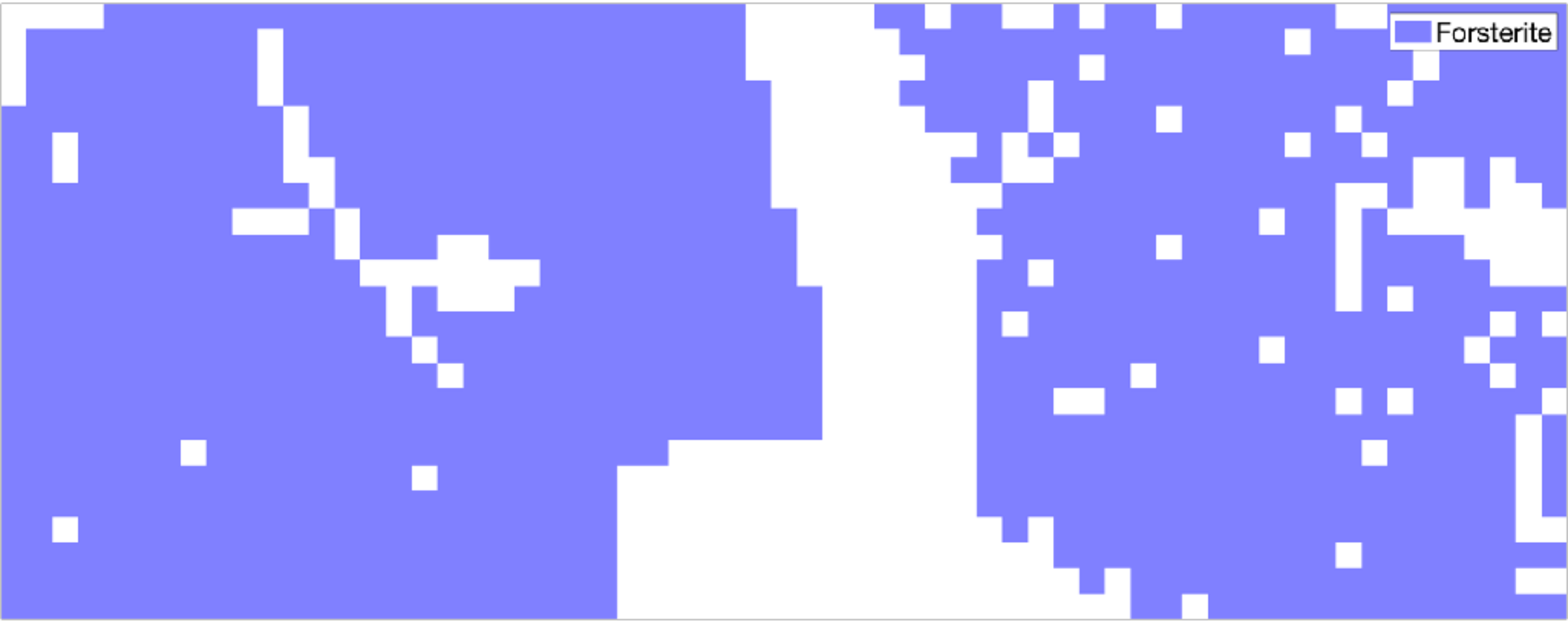
x	y	phase	...
0	0	1	
1	0	1	
2	0	1	
3	0	0	
4	0	1	
5	0	1	
6	0	1	
8	0	1	
9	0	1	
10	0	1	



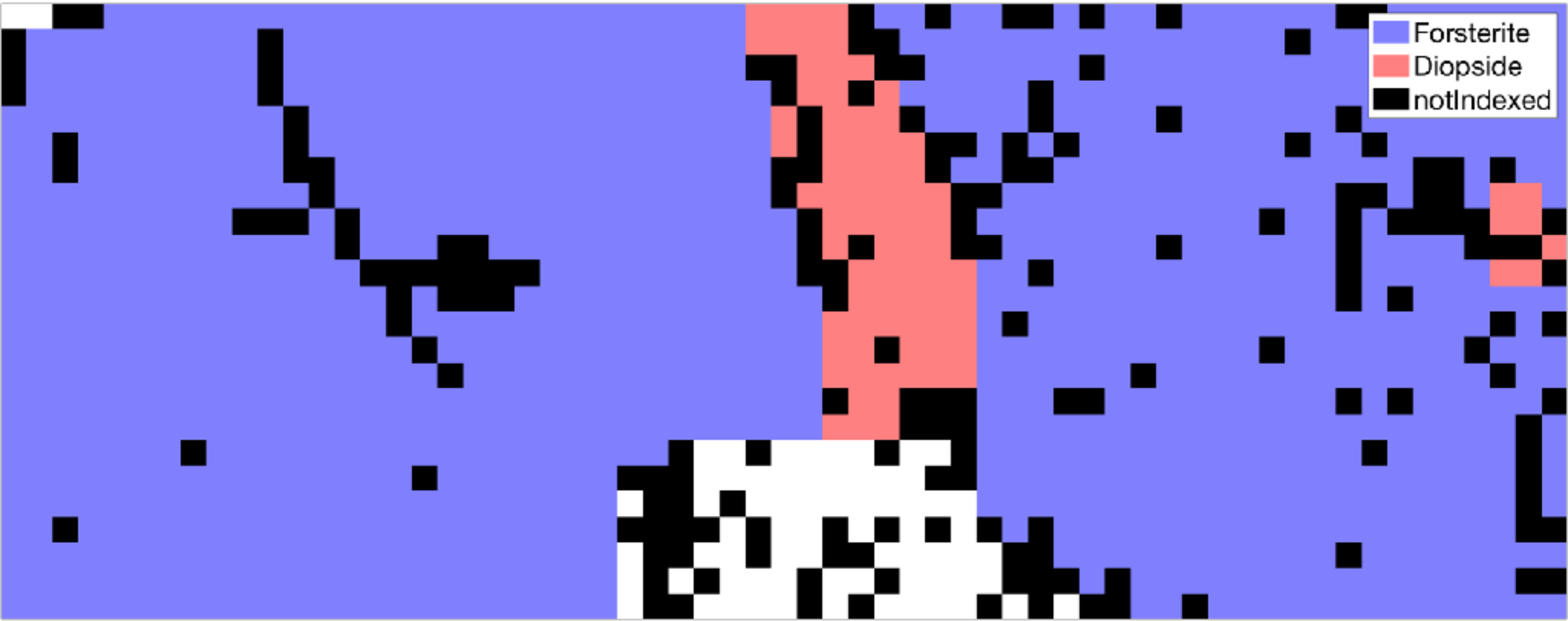
```
>> ebsd
ebsd = EBSD (show methods, plot)

 Phase  Orientations     Mineral        Color  Symmetry  Crystal reference frame
     0     190 (13%)  notIndexed
     1    1124 (77%)  Forsterite   light blue       mmm
     2     69 (4.7%)   Enstatite  light green       mmm
     3     81 (5.5%)    Diopside    light red      12/m1       X||a*, Y||b*, Z||c

 Properties: bands, bc, bs, error, mad, x, y
 Scan unit : um
>> plot(ebsd)
```



```
>> ebsd('f')
ans = EBSD (show methods, plot)

 Phase  Orientations     Mineral        Color  Symmetry  Crystal reference frame
     1   1124 (100%)  Forsterite  light blue       mmm

 Properties: bands, bc, bs, error, mad, x, y
 Scan unit : um

>> plot(ebsd('f'))
```

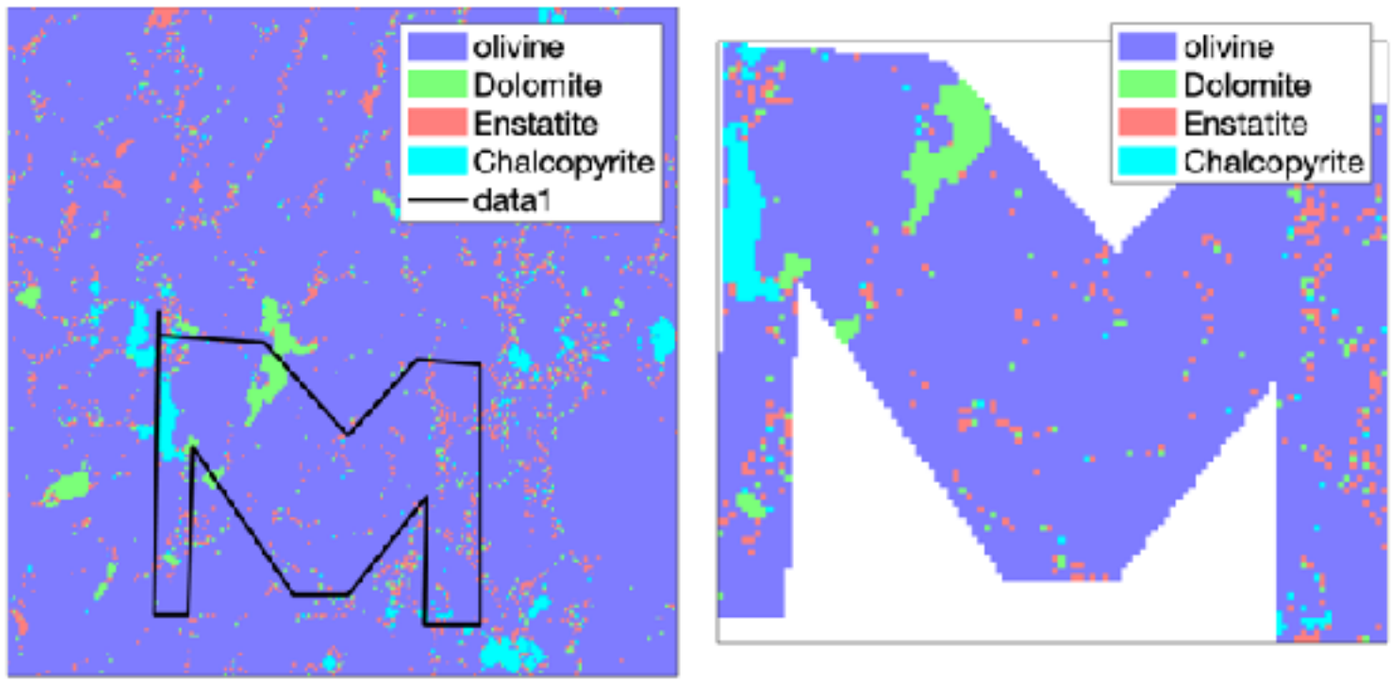


```
>> ebsd('e')=[]
ebsd = EBSD (show methods, plot)

 Phase  Orientations      Mineral       Color  Symmetry  Crystal reference frame
     0     190 (14%)  notIndexed
     1    1124 (81%)  Forsterite  light blue       mmm
     3     81 (5.8%)    Diopside   light red      12/m1        X||a*, Y||b*, Z||c

 Properties: bands, bc, bs, error, mad, x, y
 Scan unit : um
>> plot(ebsdd('indexed')); hold on
>> plot(ebsdd('notIndexed'),'FaceColor','k'); hold off
```



select ebsd area

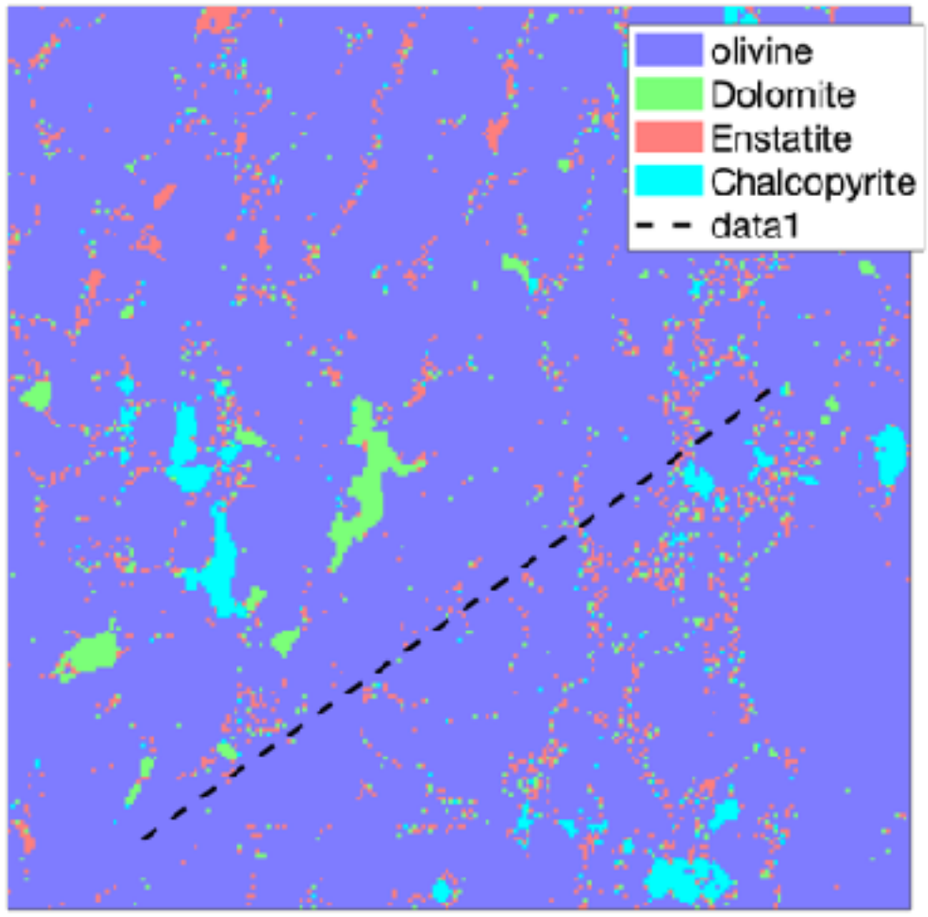
```
plot(ebsd)
xy=ginput() % click around the region, when done hit enter
hold on
plot(xy(:,1),xy(:,2),'k','linewidth',2)
hold off
ebsd_sel = ebsd(inpolygon(ebsd,xy))
nextAxis
plot(ebsd_sel)
```

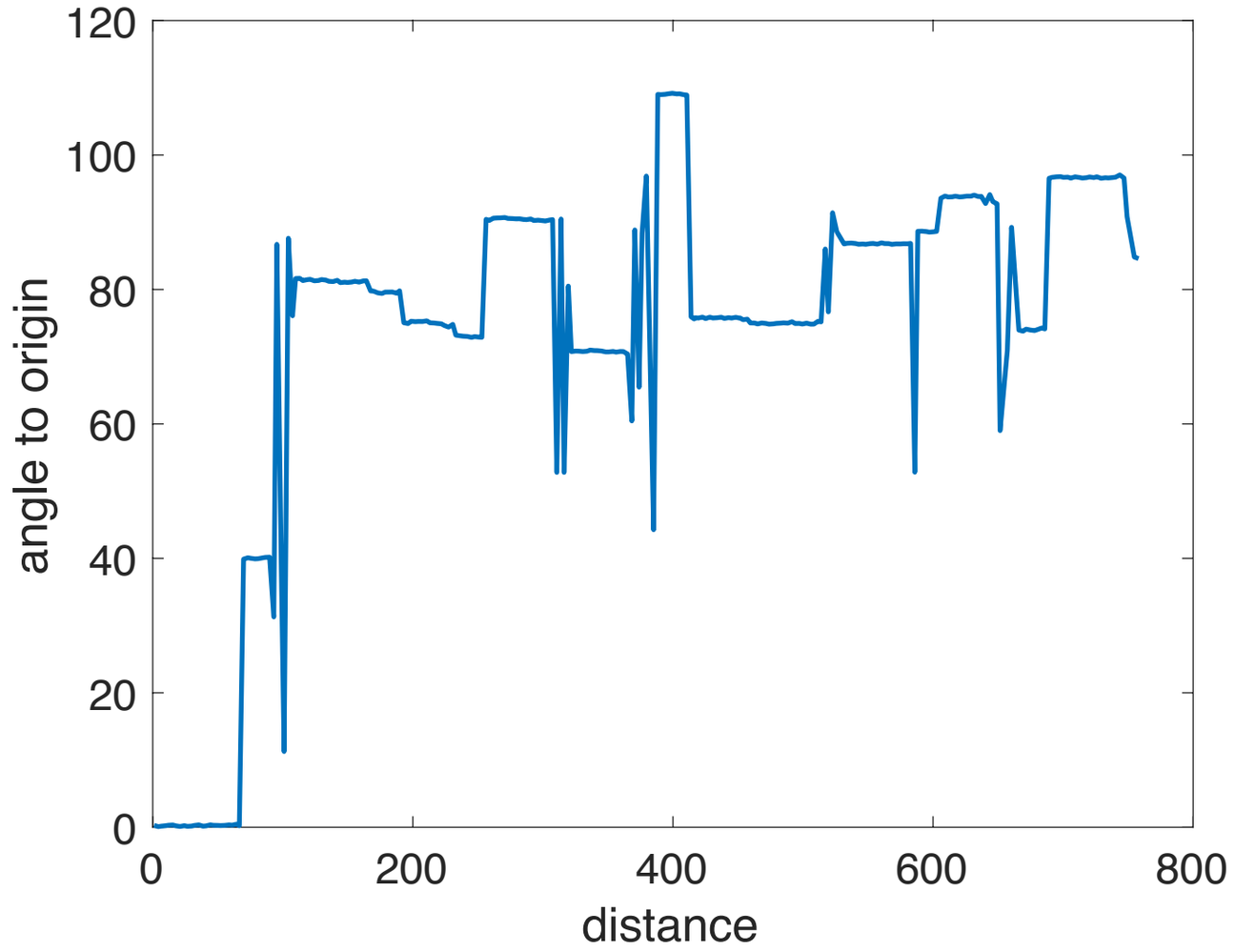


alternatively, in case you only want to select a rectangle manually:

```
ebsd_sel = selectInteractive(ebsd)
```

select ebsd along a line





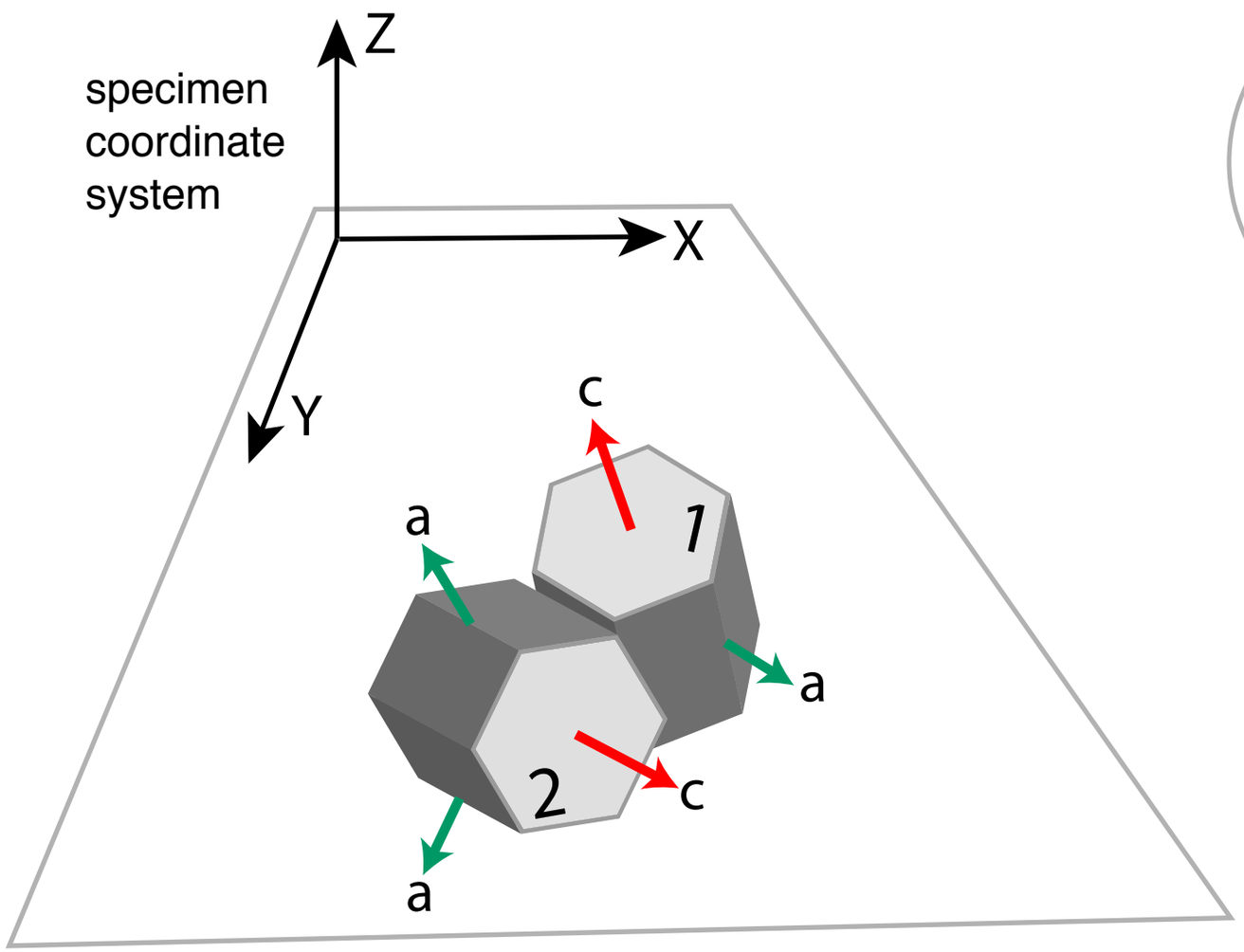

```
plot(ebsd)
xy = ginput(2); hold on
plot(xy(:,1),xy(:,2),'--k','linewidth',2) hold off
```

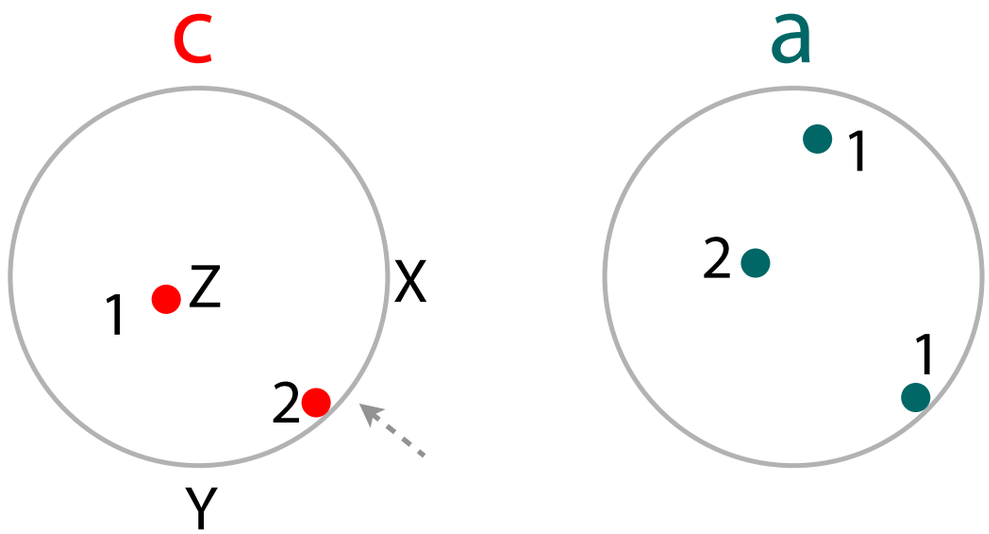
```
[ebsd_line, dist] = spatialProfile(ebsd,xy)
```

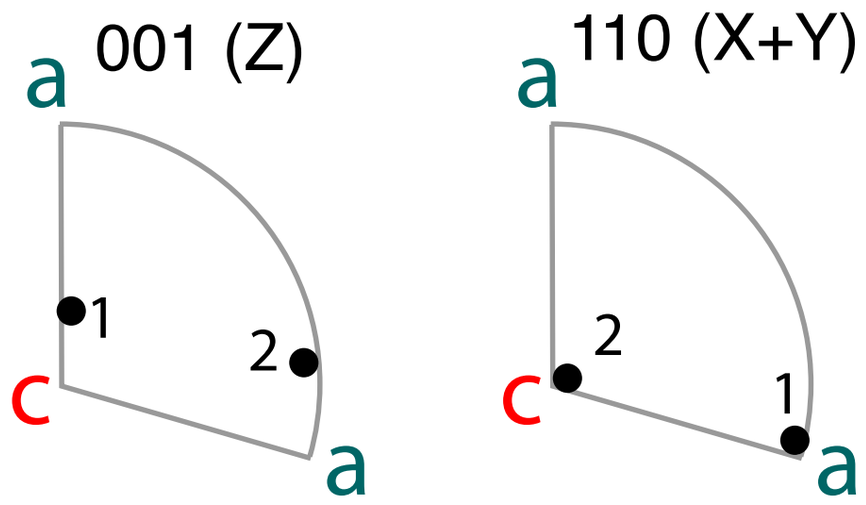
```
cond = ebsd_line.phase == 1;
o = ebsd_line(cond).orientations;
plot(dist(cond(2:end)), angle(o(1),o(2:end))/degree,'linewidth',2)
```

representation of orientations in ebsd maps

pole figures of specimen directions (in specimen coordinates [X,Y,Z]), which are parallel to a given crystal direction (c and a)

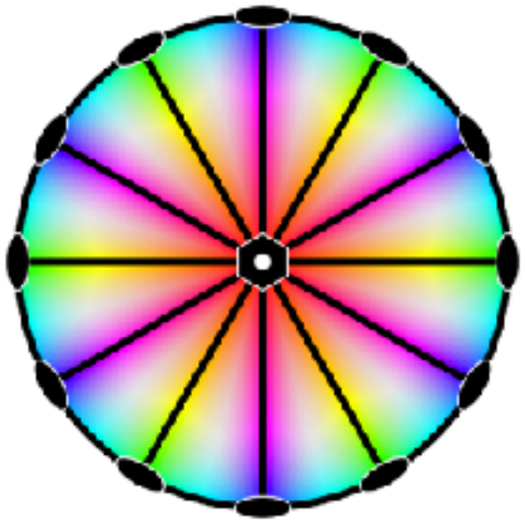


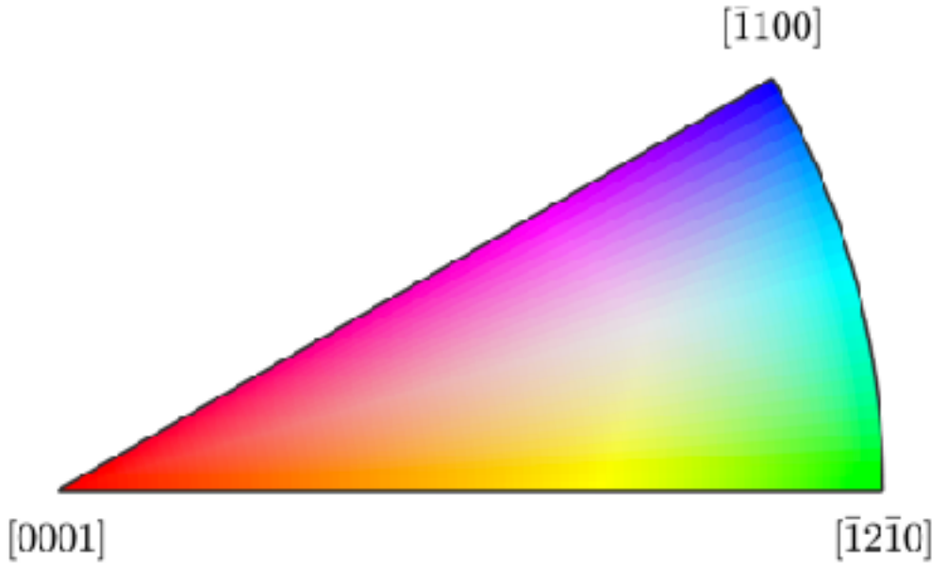


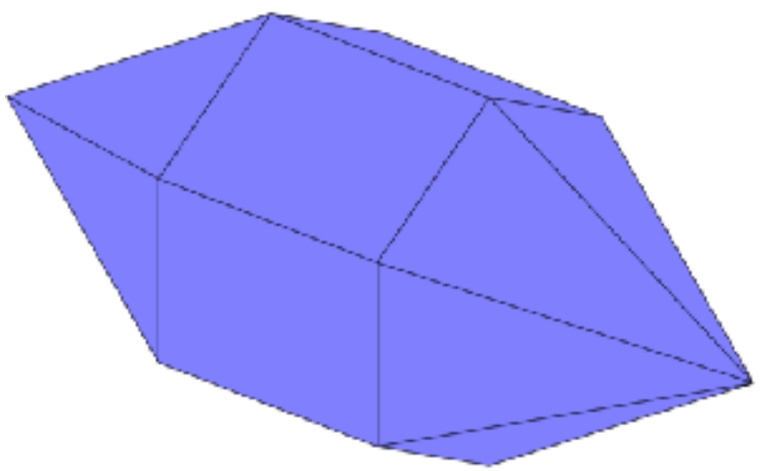


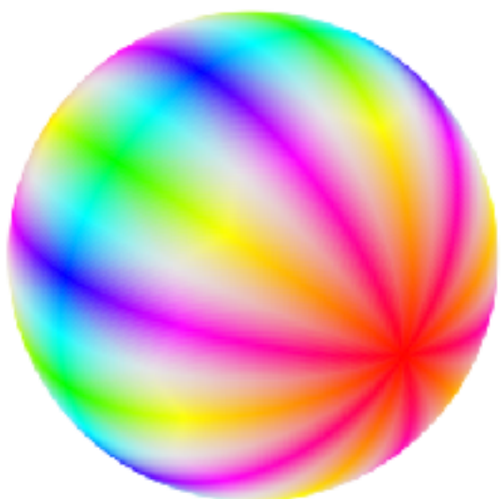
inverse pole figures of crystal directions (in crystal coordinates) which are parallel to a given specimen direction (Z and X+Y)

representation of textures: colorcoding crystal directions



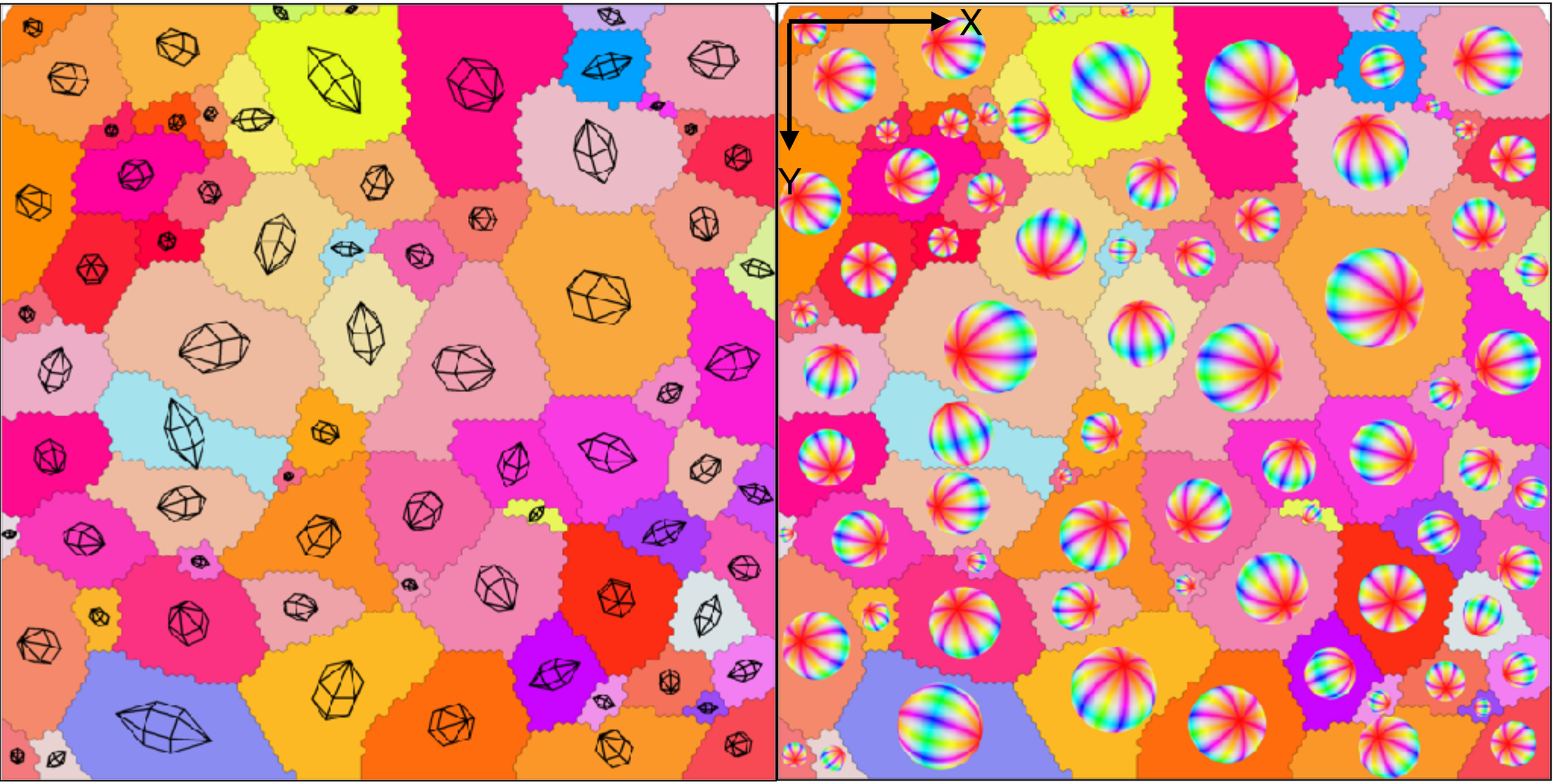






representation of textures: ipf color coding

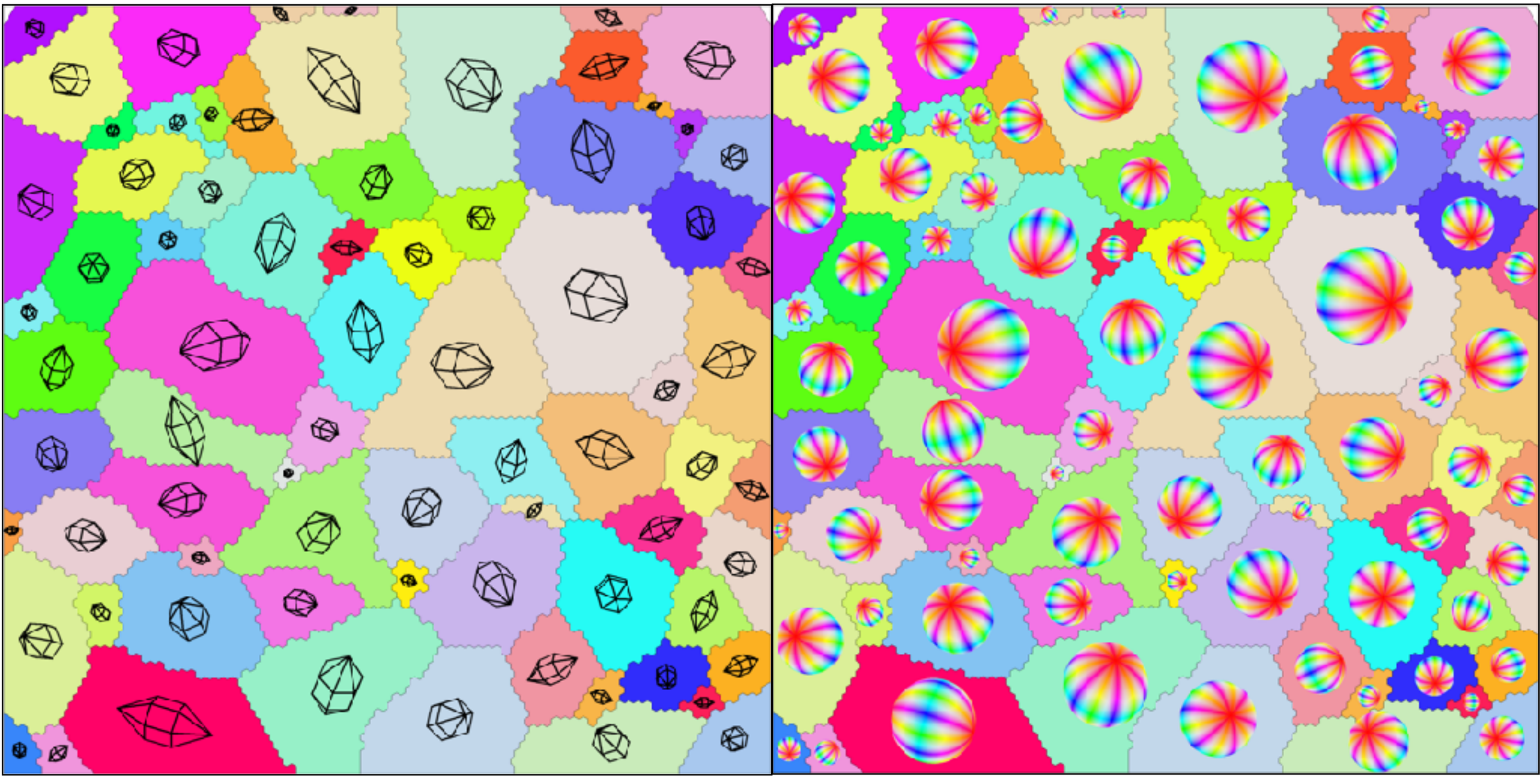
reference direction Z (001)



note: one ipf map is not enough to read the crystal orientation form the map

representation of textures: ipf color coding

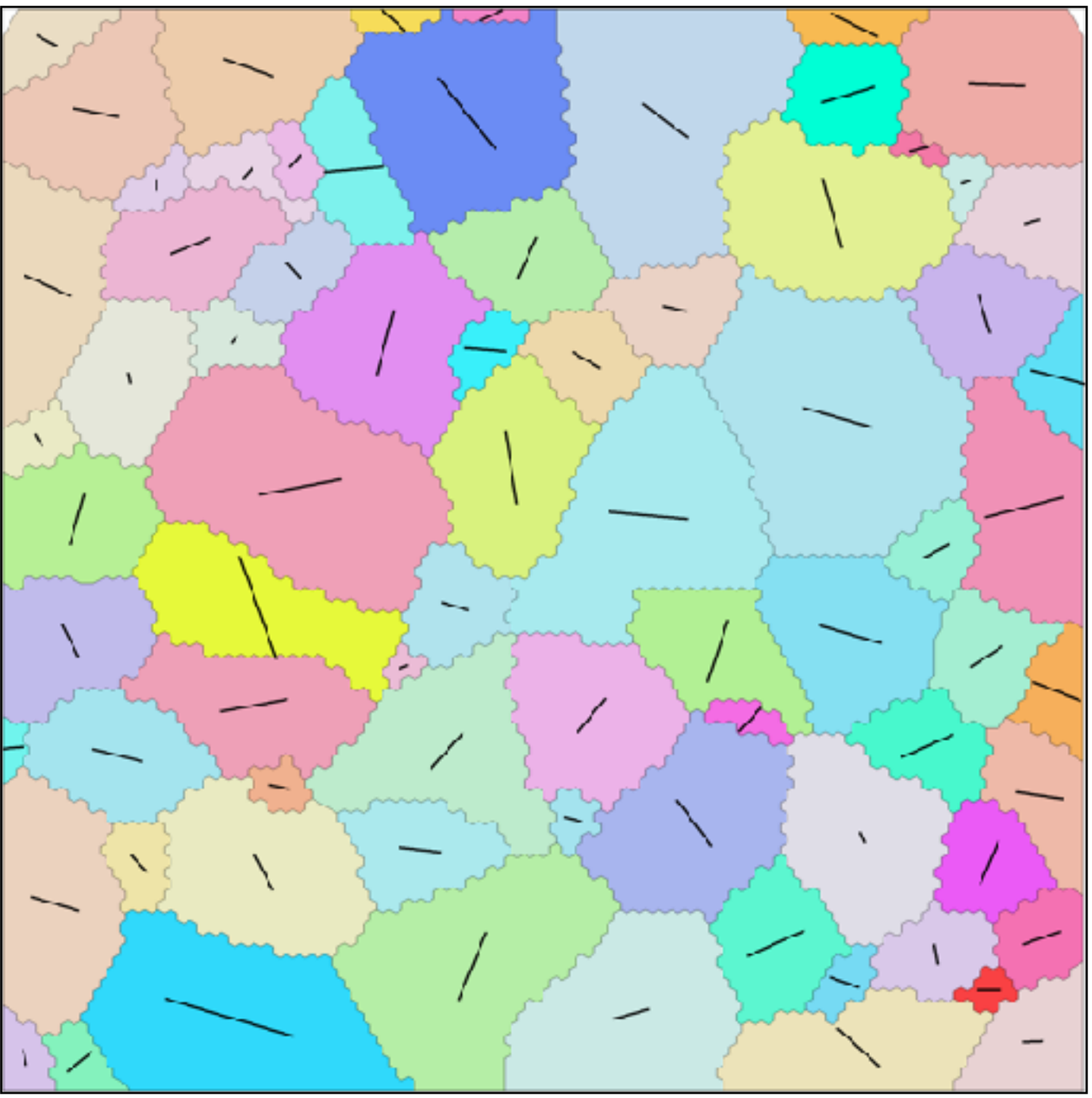
reference direction X (100)

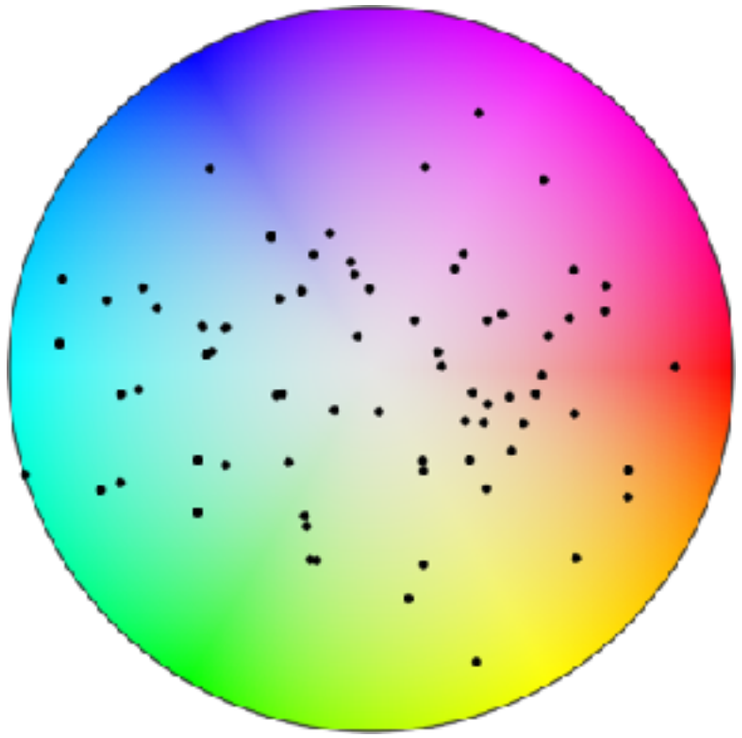


note: one ipf map is not enough to read the crystal orientation form the map

representation of textures: pf color coding

c-axes [0001]

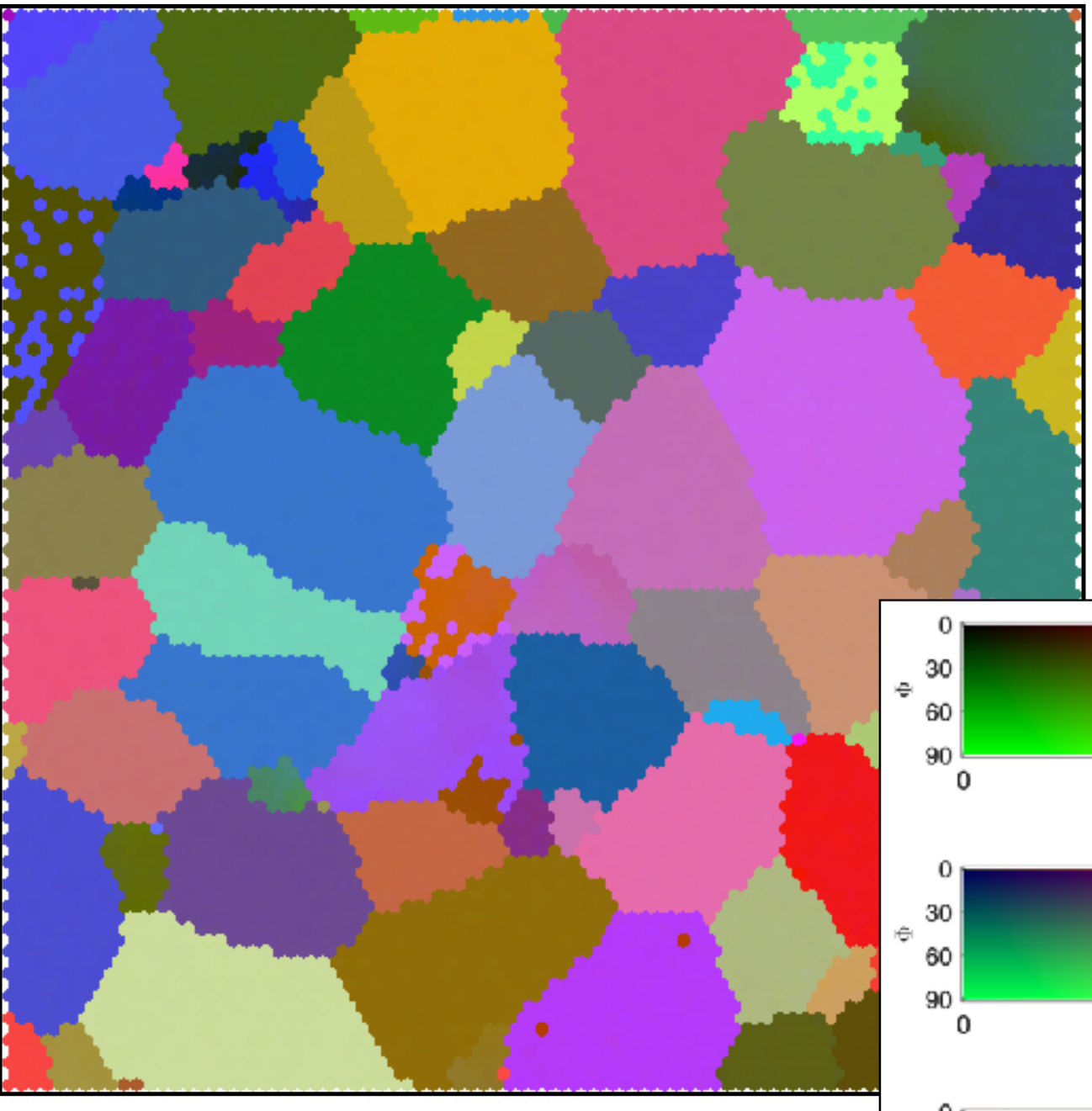


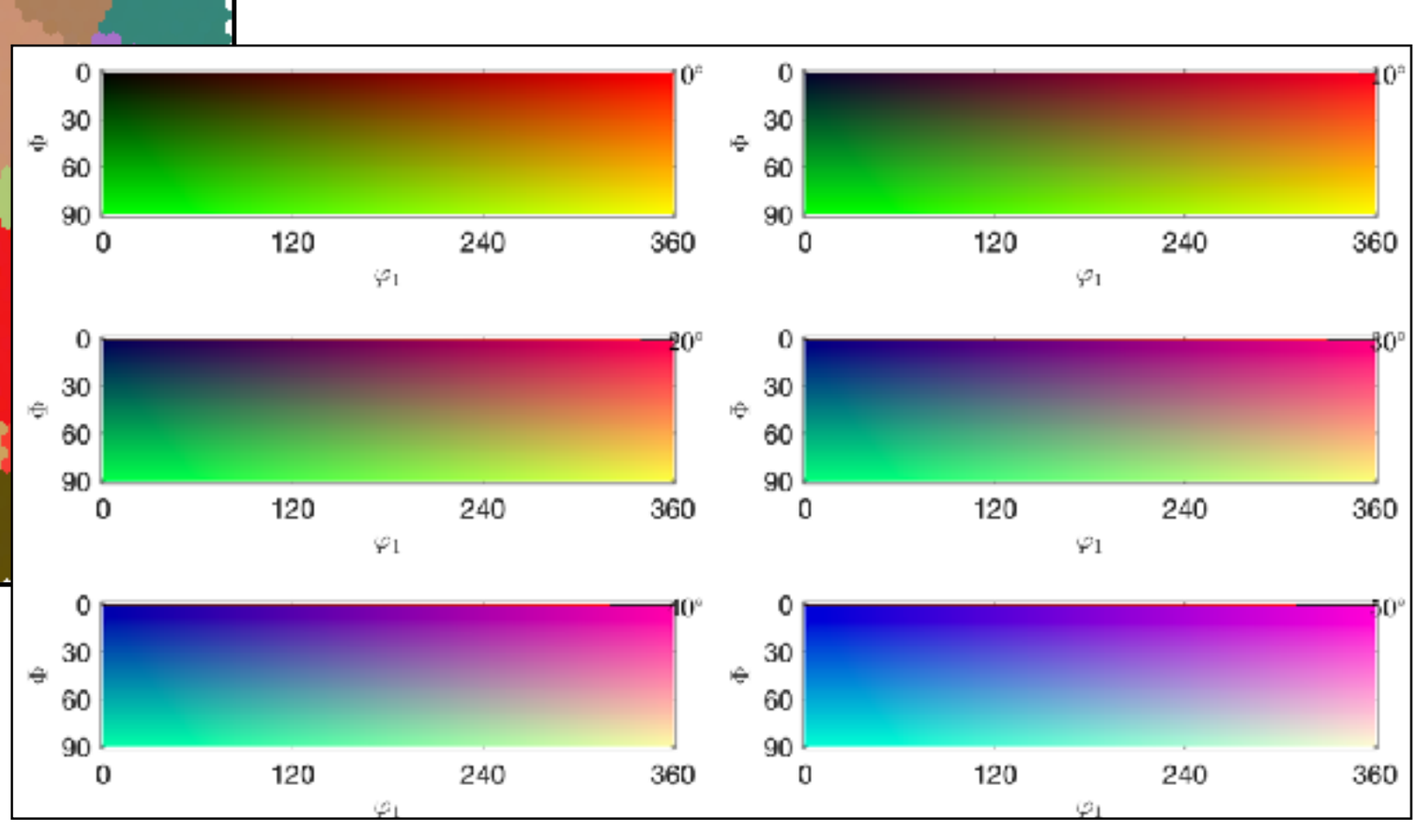


- only possible for unique axes / directions

obviously this colorcoding is also not enough to read the crystal orientation form the map

representation of textures: Euler angle key





representation of textures: axisAngle color key