

# putting rock properties in their place

– meshing orientation imaging with grain shape and size distribution

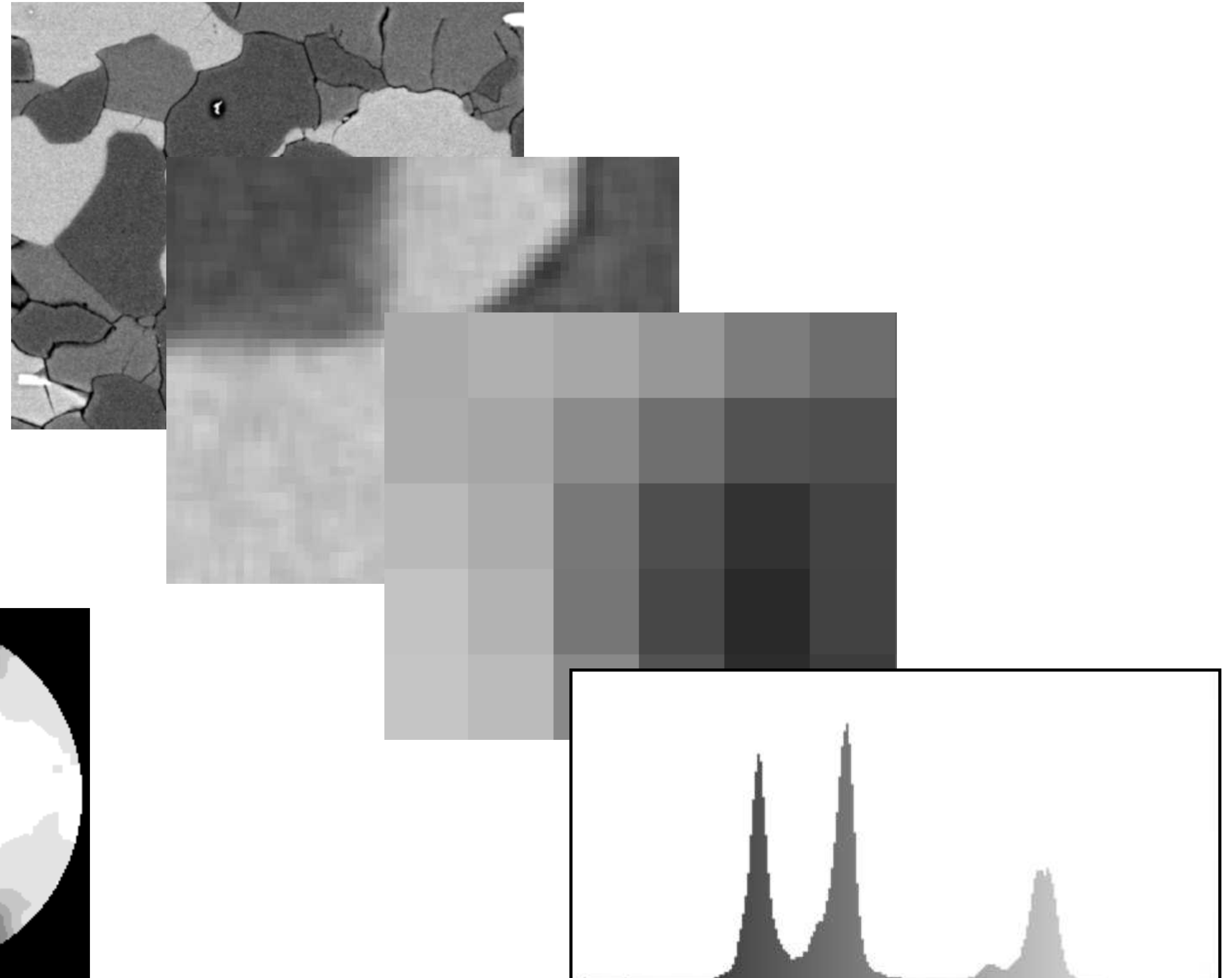
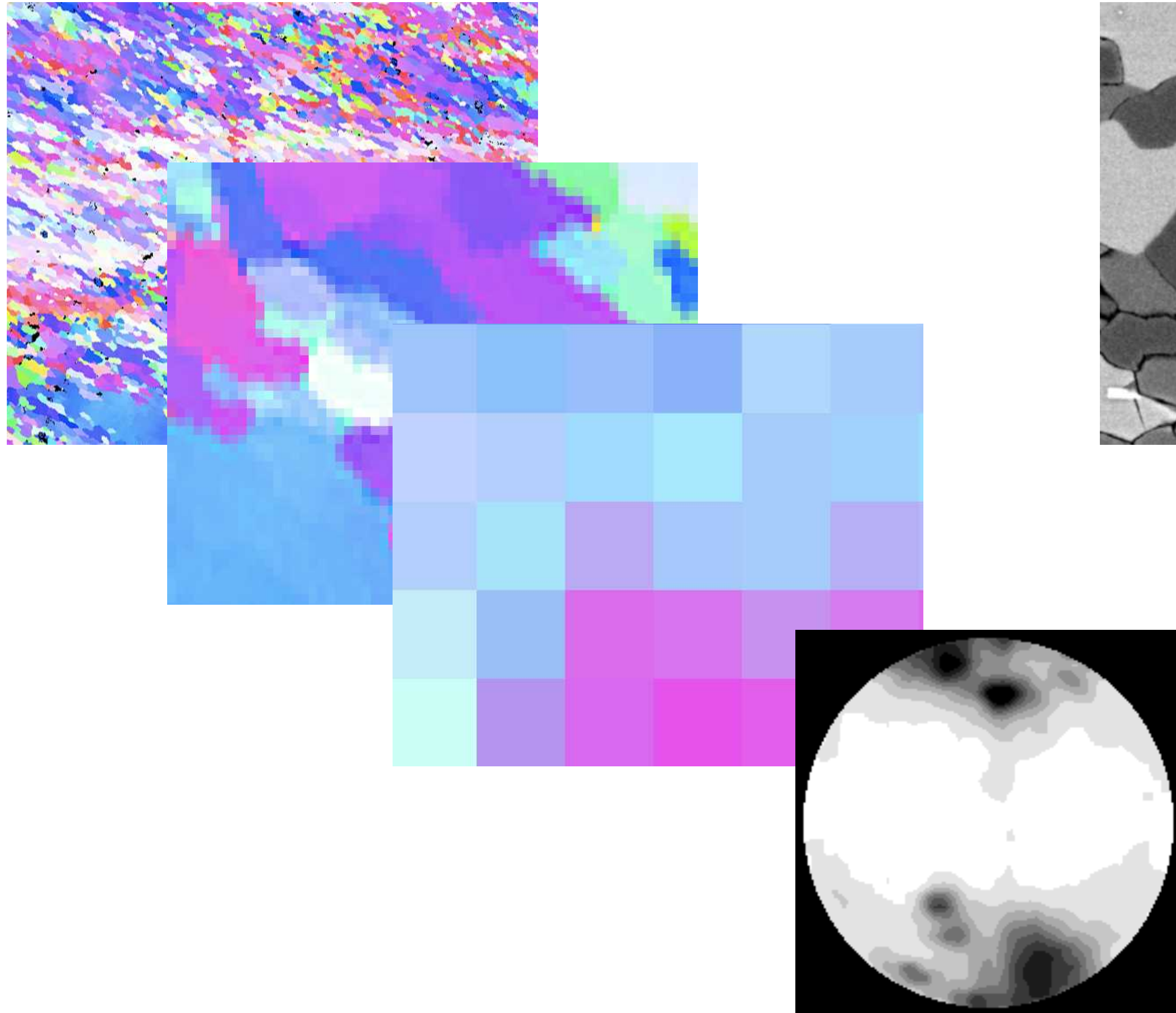
[renee.heilbronner@unibas.ch](mailto:renee.heilbronner@unibas.ch)

Holger Stünitz Symposium, Orléans, June 25, 2024

# physical properties – at each pixel

$$A_{\text{phase}}/A_{\text{total}} = V_{\text{phase}}/V_{\text{total}}$$

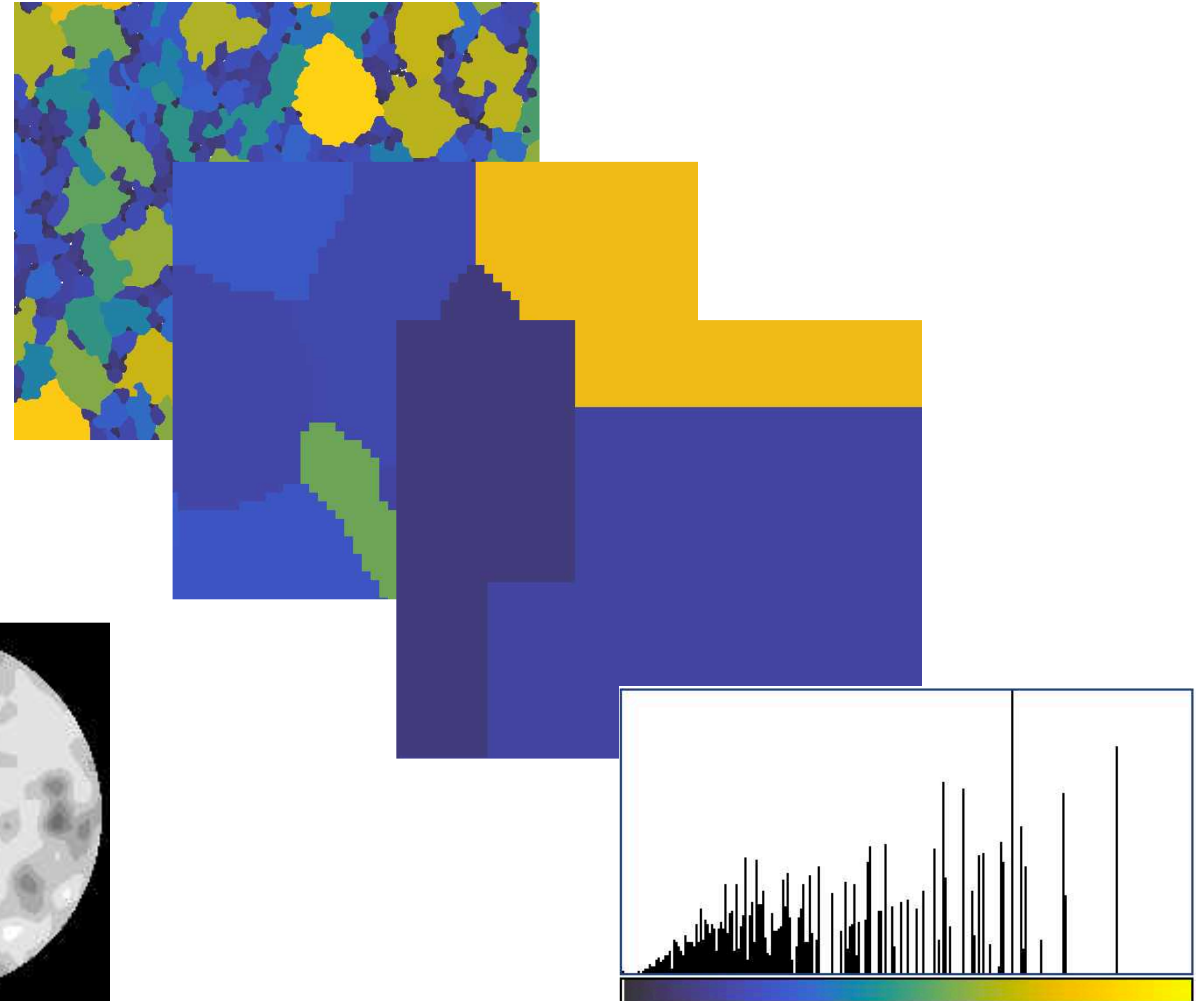
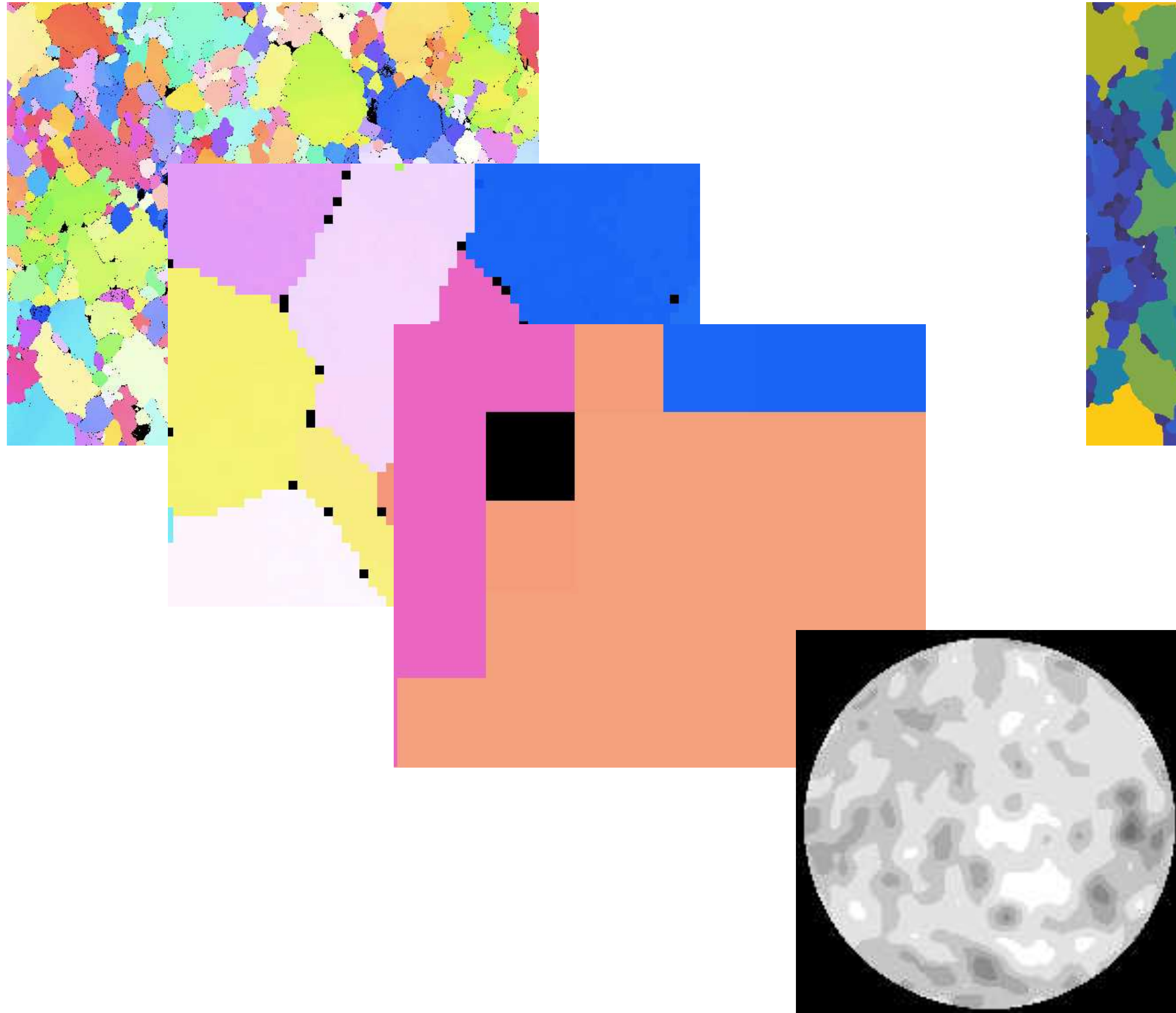
$$A_{\text{phase}}/A_{\text{total}} = V_{\text{phase}}/V_{\text{total}}$$



# properties of pixels – properties of grains

$$A_{\text{phase}}/A_{\text{total}} = V_{\text{phase}}/V_{\text{total}}$$

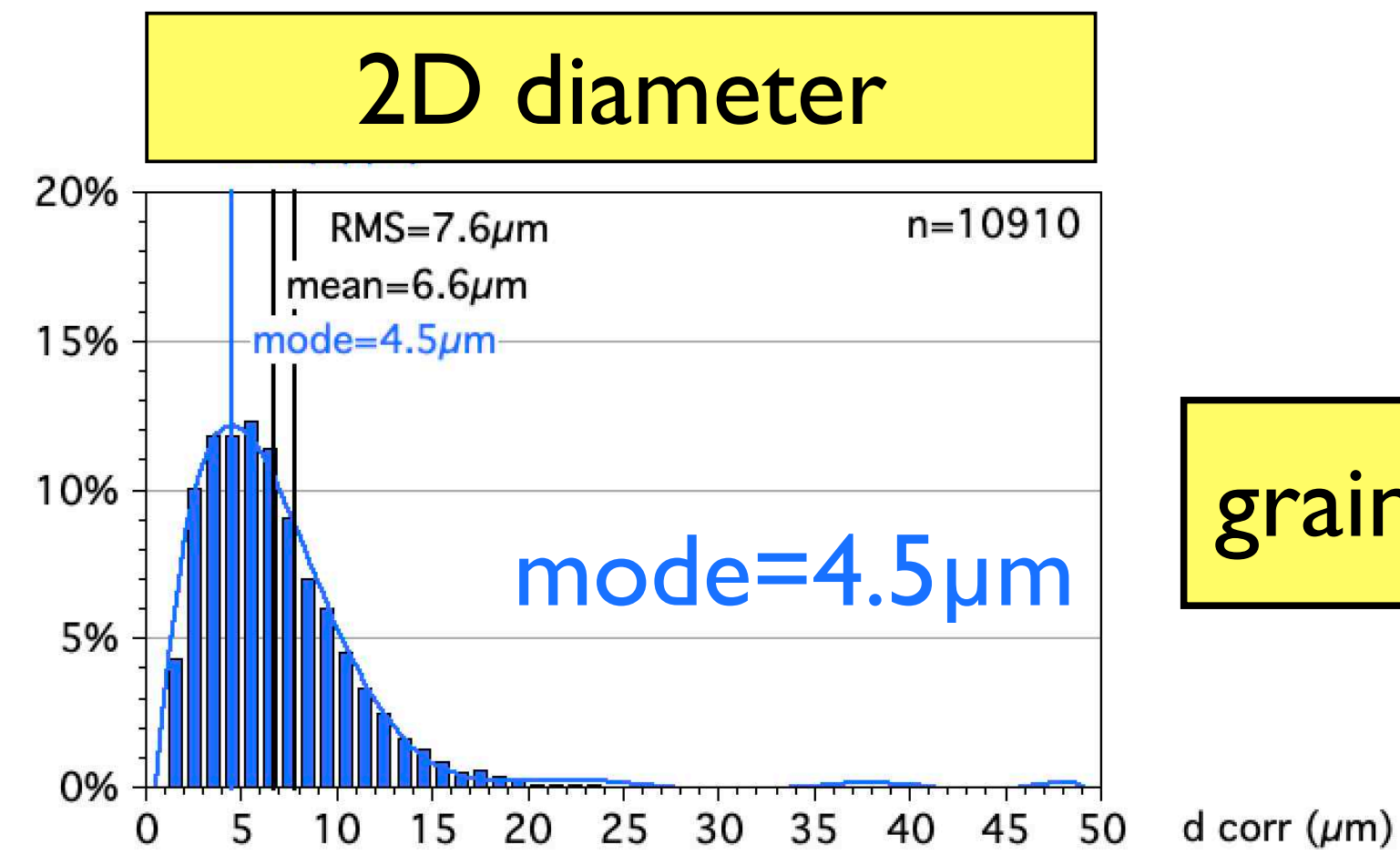
$$A_{\text{grainsize}}/A_{\text{total}} \neq V_{\text{grainsize}}/V_{\text{total}}$$



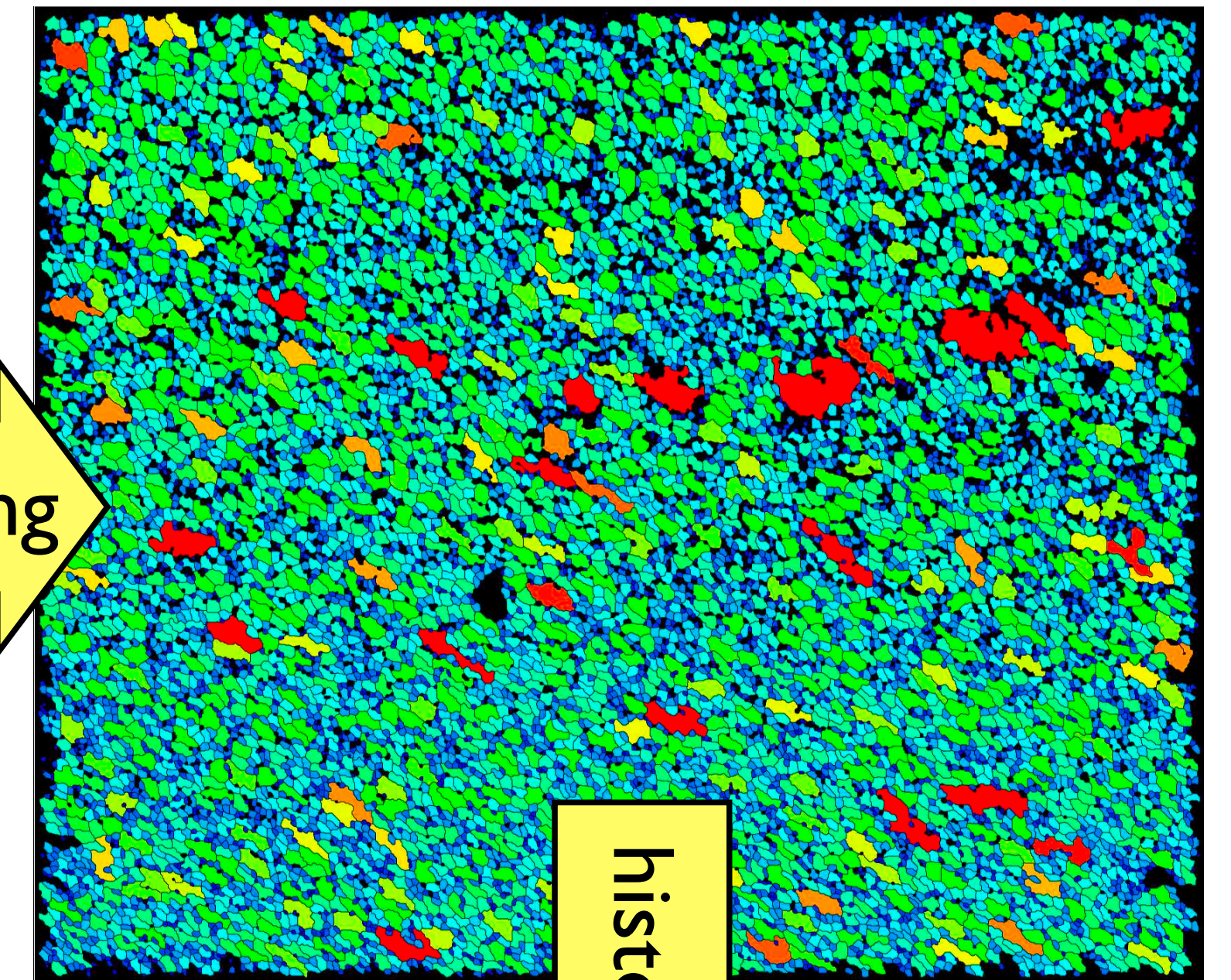
# my mission on earth ... 2D sections $\rightarrow$ 3D grains



segmentation



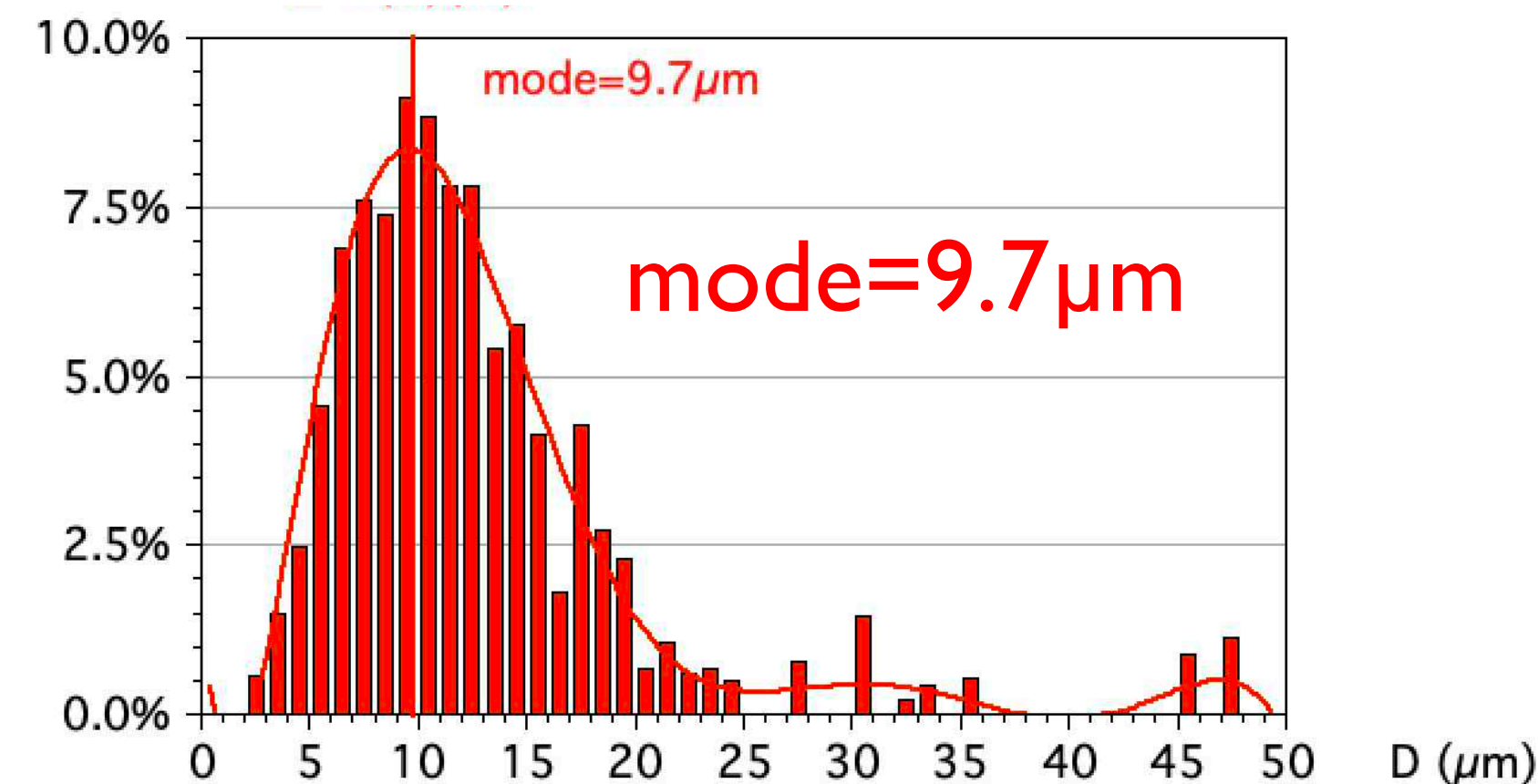
grain size mapping



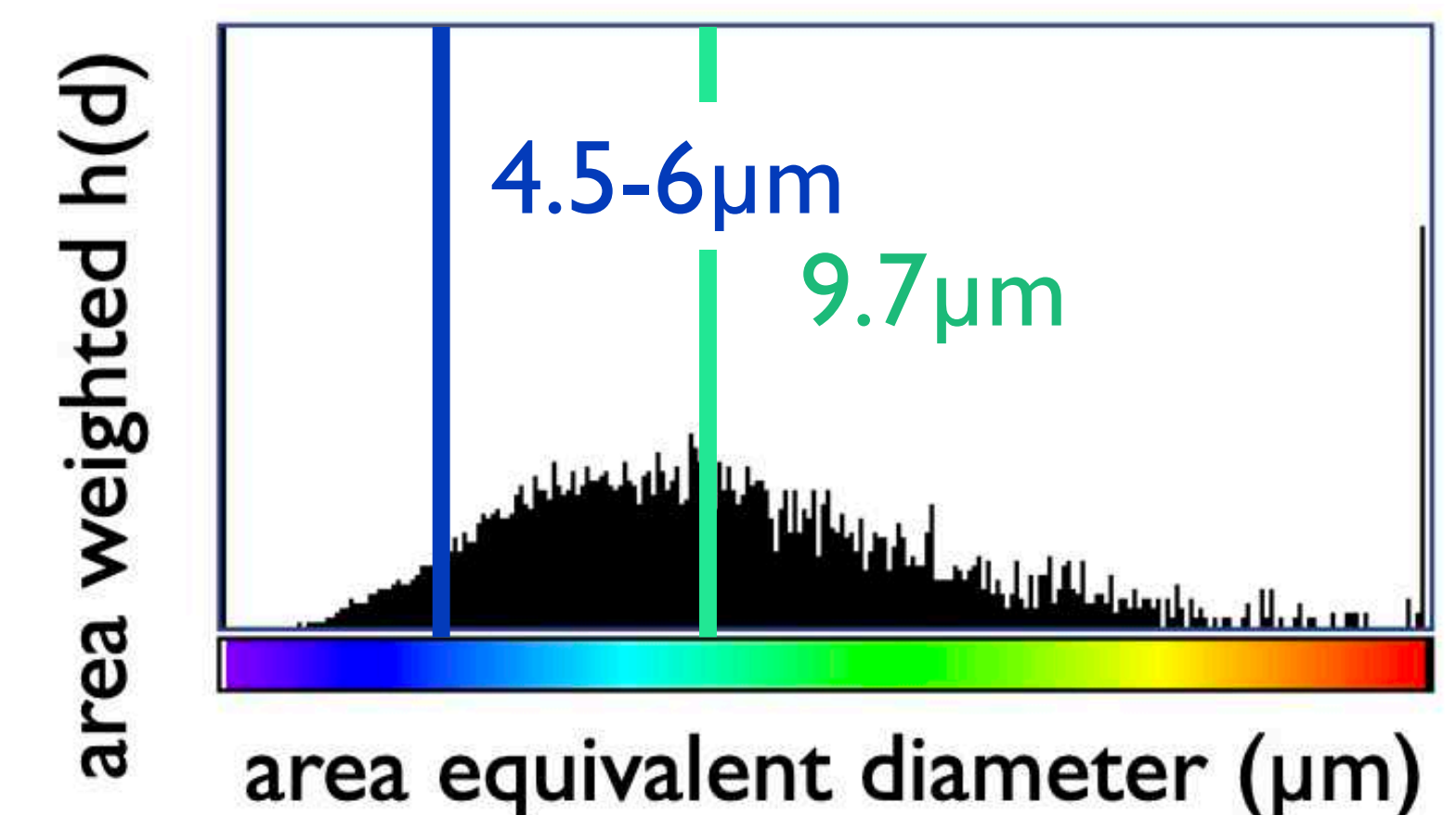
histogram

area-weighting of 2D diameters (grainsize mapping) confirms visually that the dominant grain size  $\neq$  the numerical mean (or RMS) of  $h(d)$

vol-weighted 3D diameter



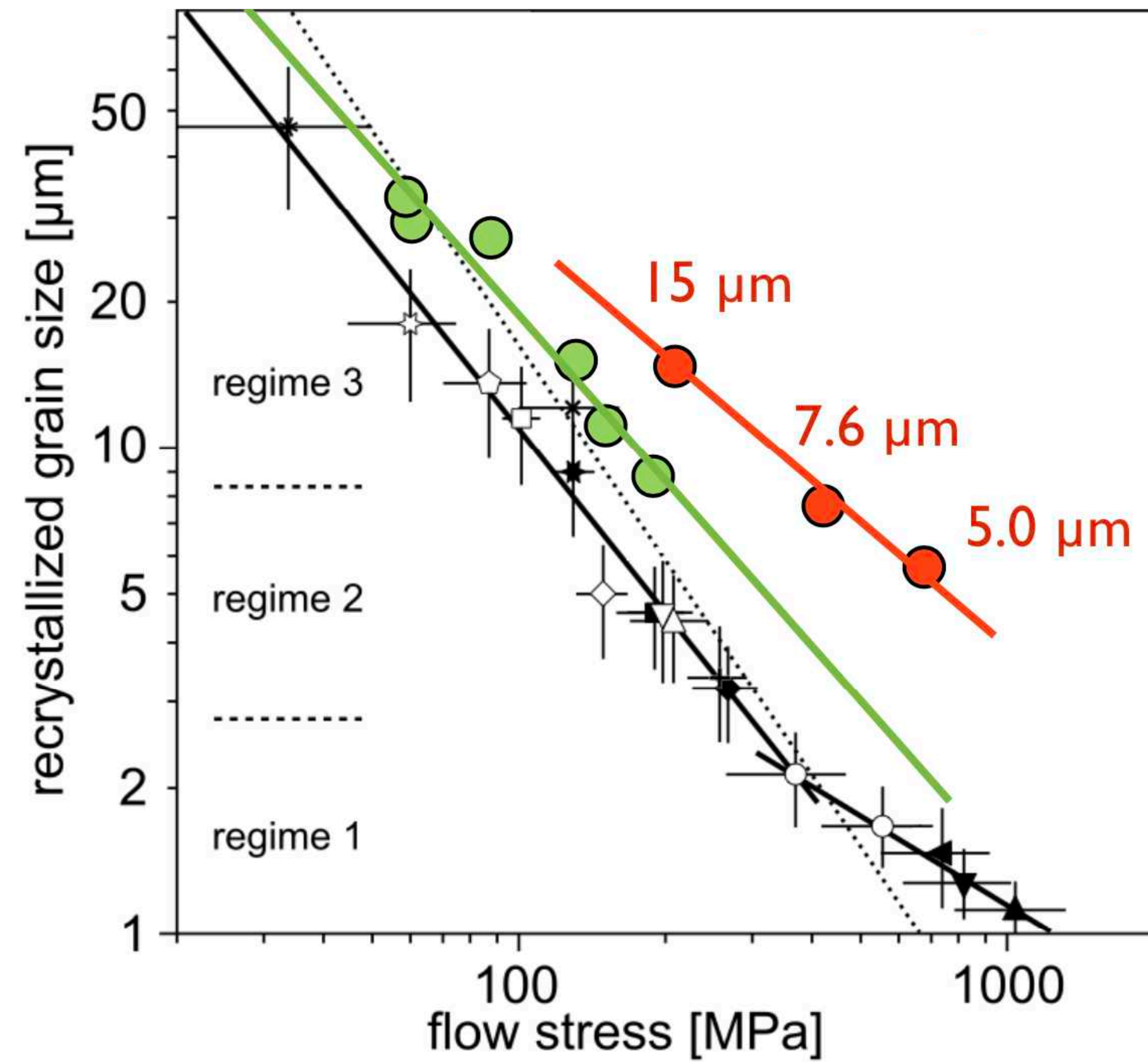
area-weighted 2D diameter



where to start ?

... the piezometer ...?

# EGU 2016 – surprise surprise !!



RMS  $h(d)$   
 $d$  = diameter of 2D sections

— Stipp & Tullis 2003

..... Twiss 1977

mode vol%(D)

D = diameter of 3D grains

— Stipp & Tullis 2003  
*recalculated from EBSD maps*

mode vol%(D)

D = diameter of 3D grains

— shear 'piezometer'

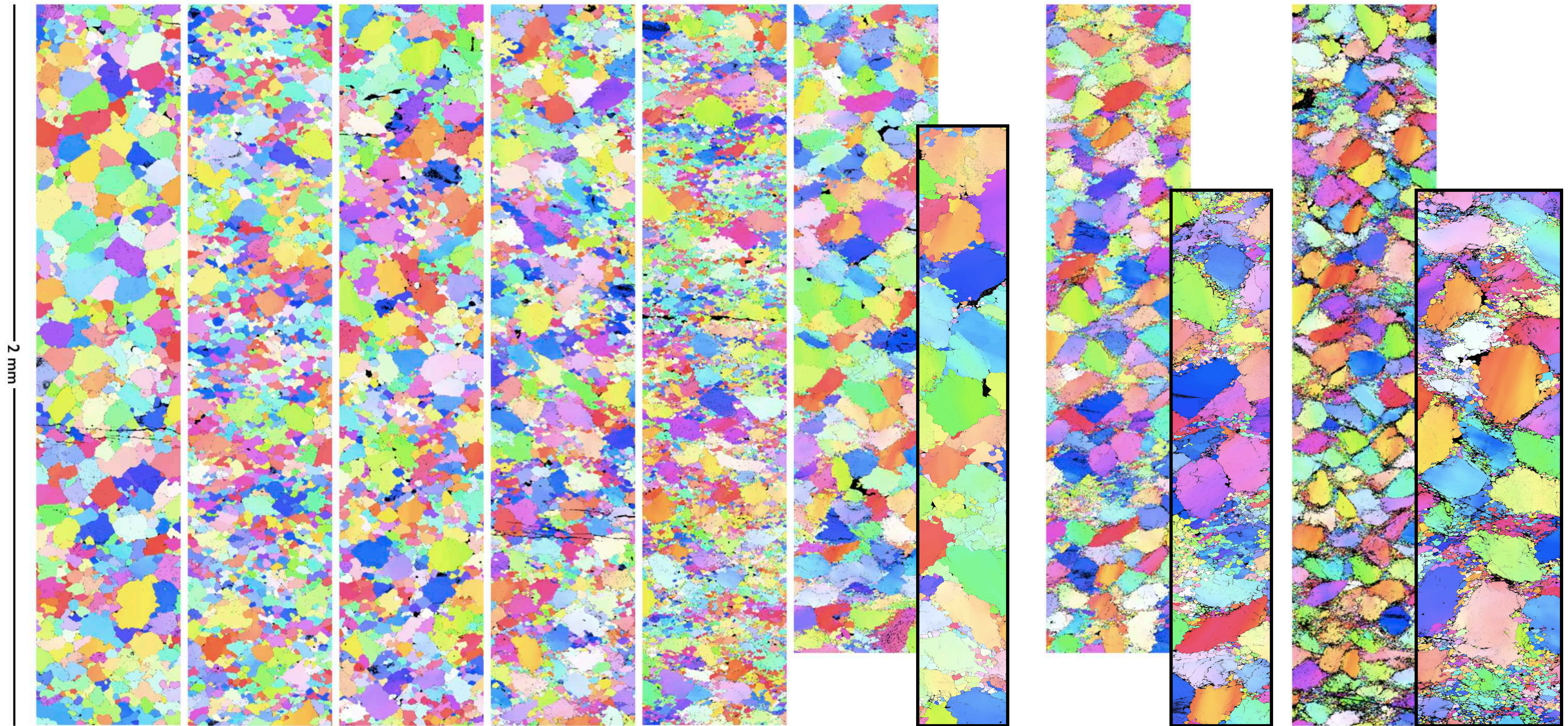
*coaxial experiments  
molten salt assembly*

*shearing experiments  
solid confining medium*

questions:

- is BHQ stronger in shear compared to coaxial deformation ?!  
i.e., piezometer not valid for shearing deformation?
- is determination of  $\Delta\sigma$  from shearing experiments incorrect?

# EBSD → CIP orientation images of piezometer experiments



w1143-m2

w1066-m2

w1025-m2

w1024-m10

w1029-m3

w1081-m4

w1081-m5

w1050-m5

w1050-m6

w1051-m5

w1051-m6

$\Delta\sigma =$  58 MPa

60 MPa

87 MPa

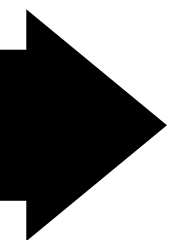
102 MPa

130 MPa

139 MPa

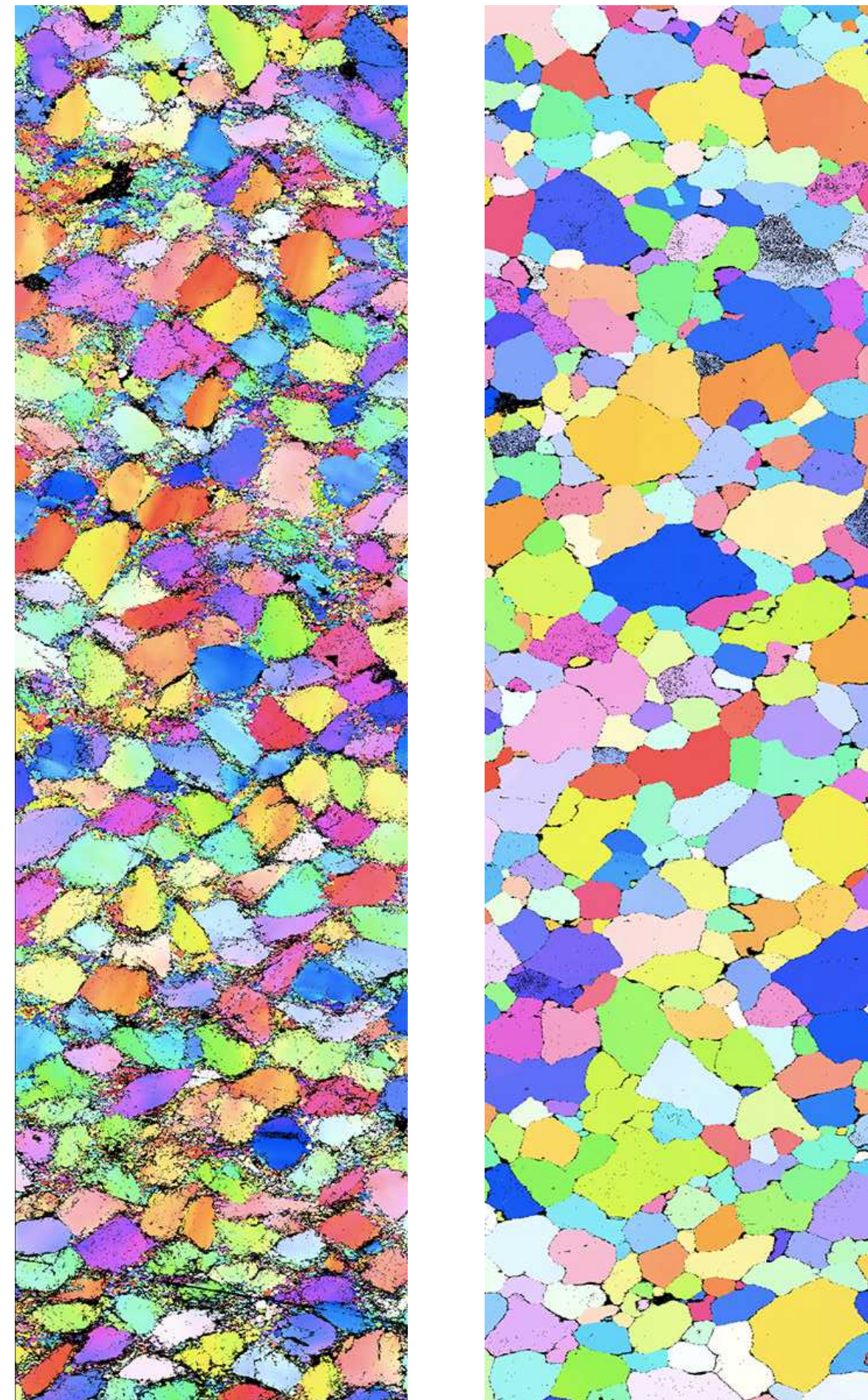
149 MPa

189 MPa

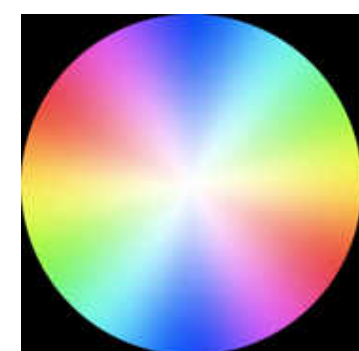


# grain boundaries → diameters → grain size maps

c-axis orientation images



c-axis PDF

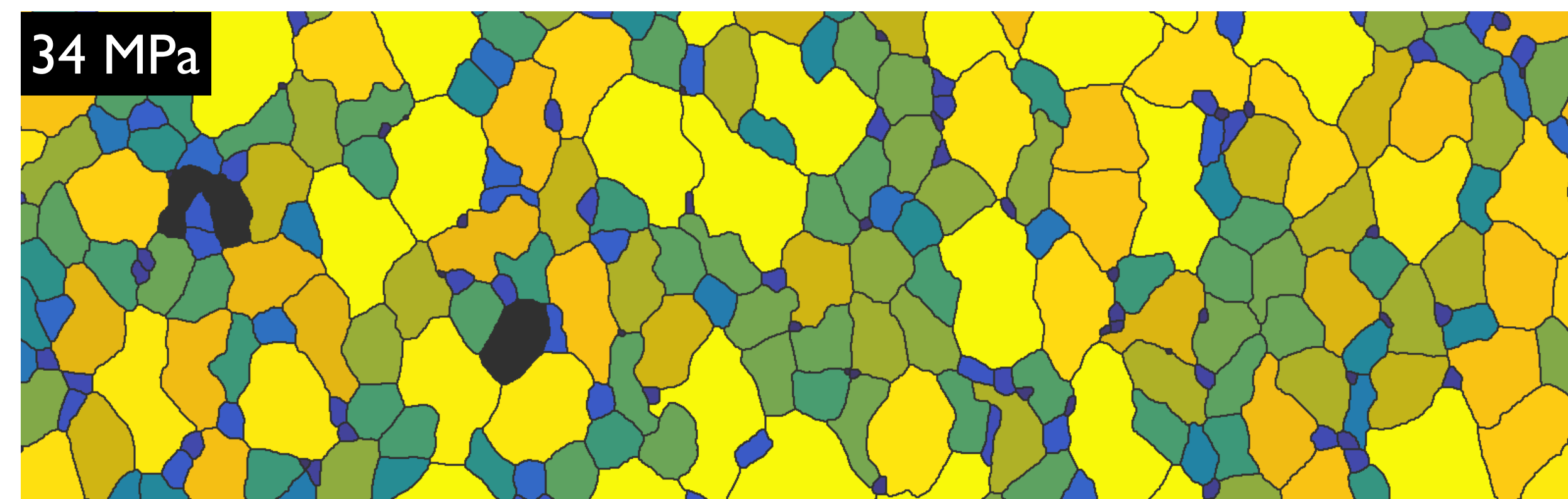
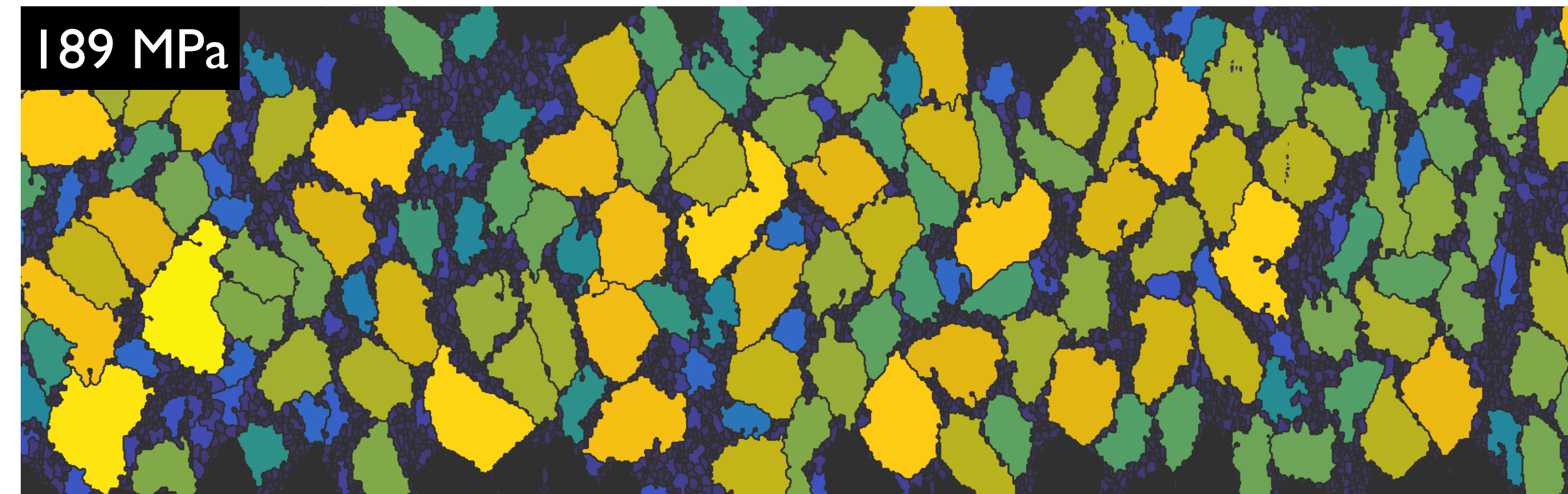


CLUT

189 MPa

34 MPa

grain size maps

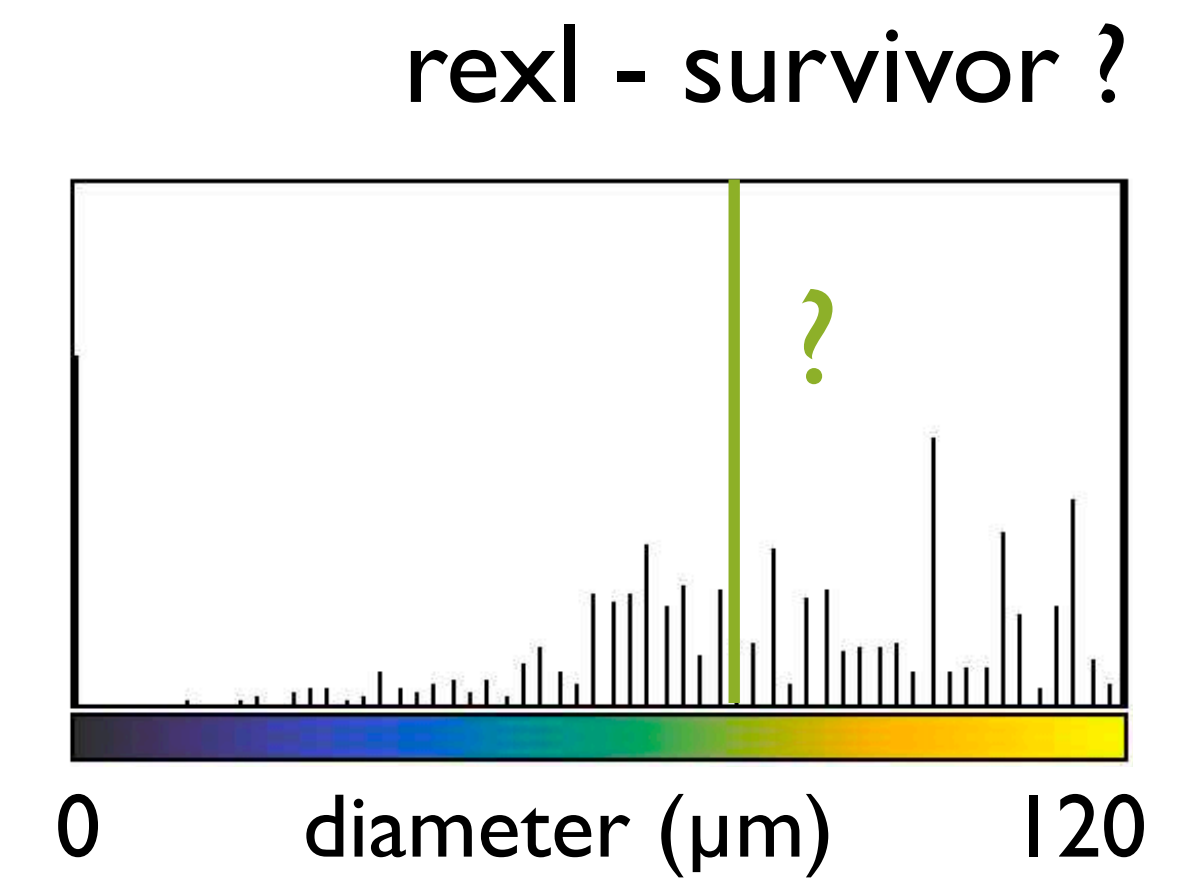
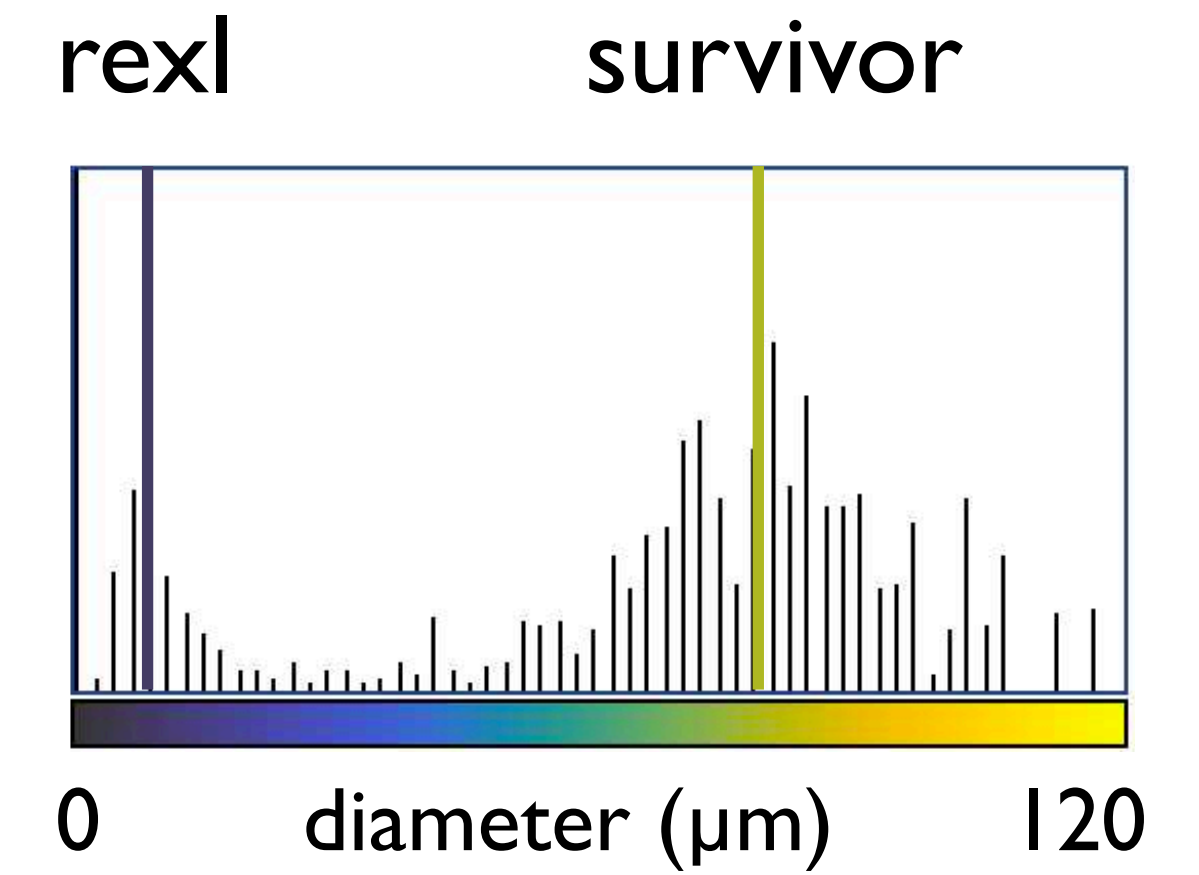


0 μm

$d_{\text{corr}}$

120 μm

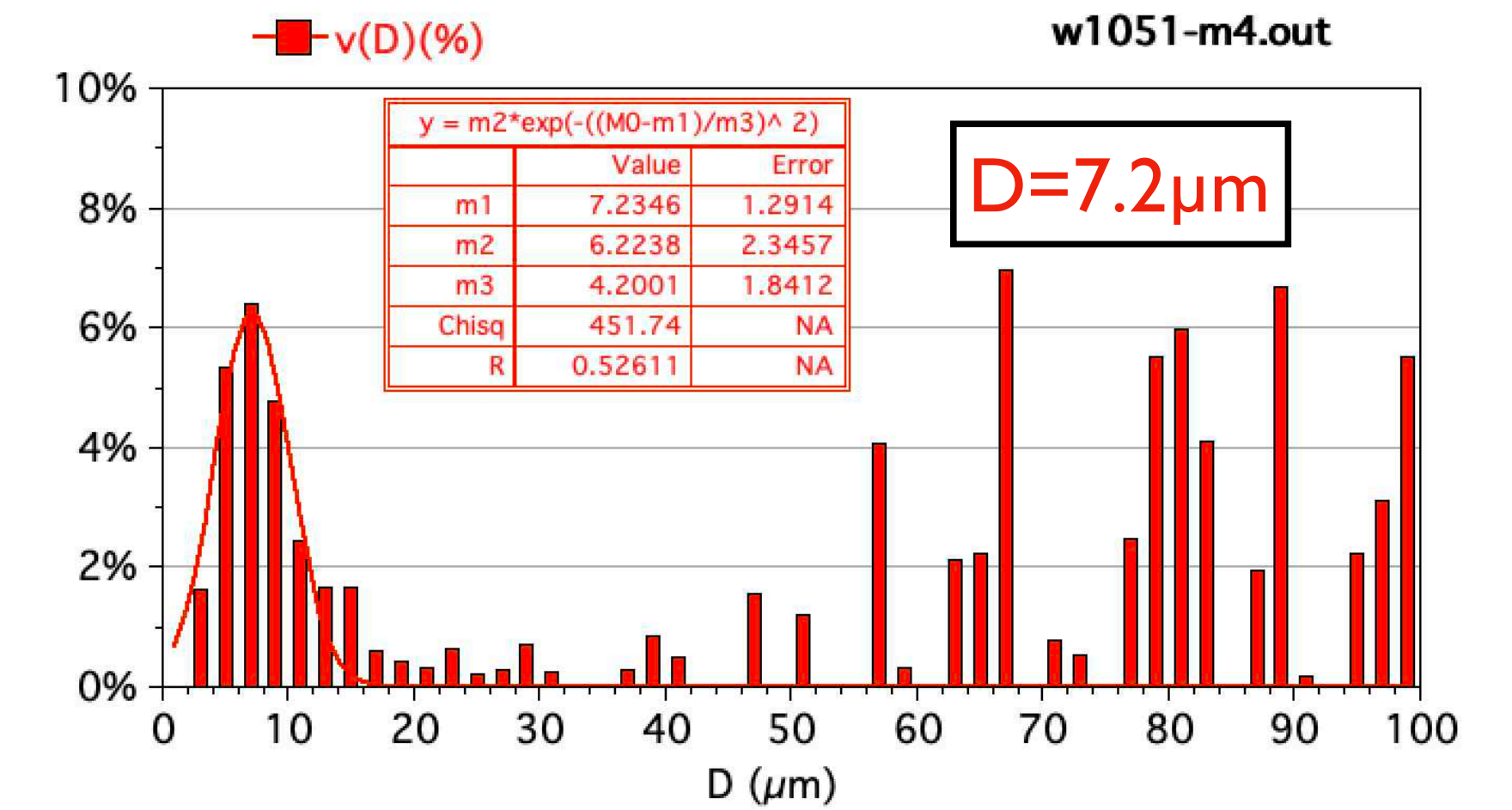
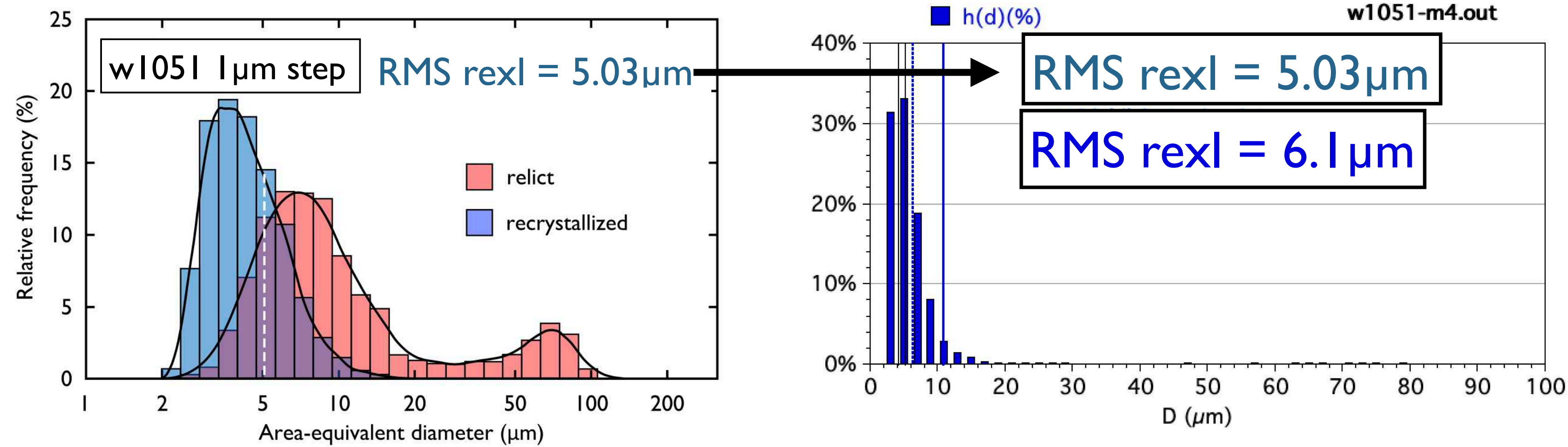
area-weighted histograms



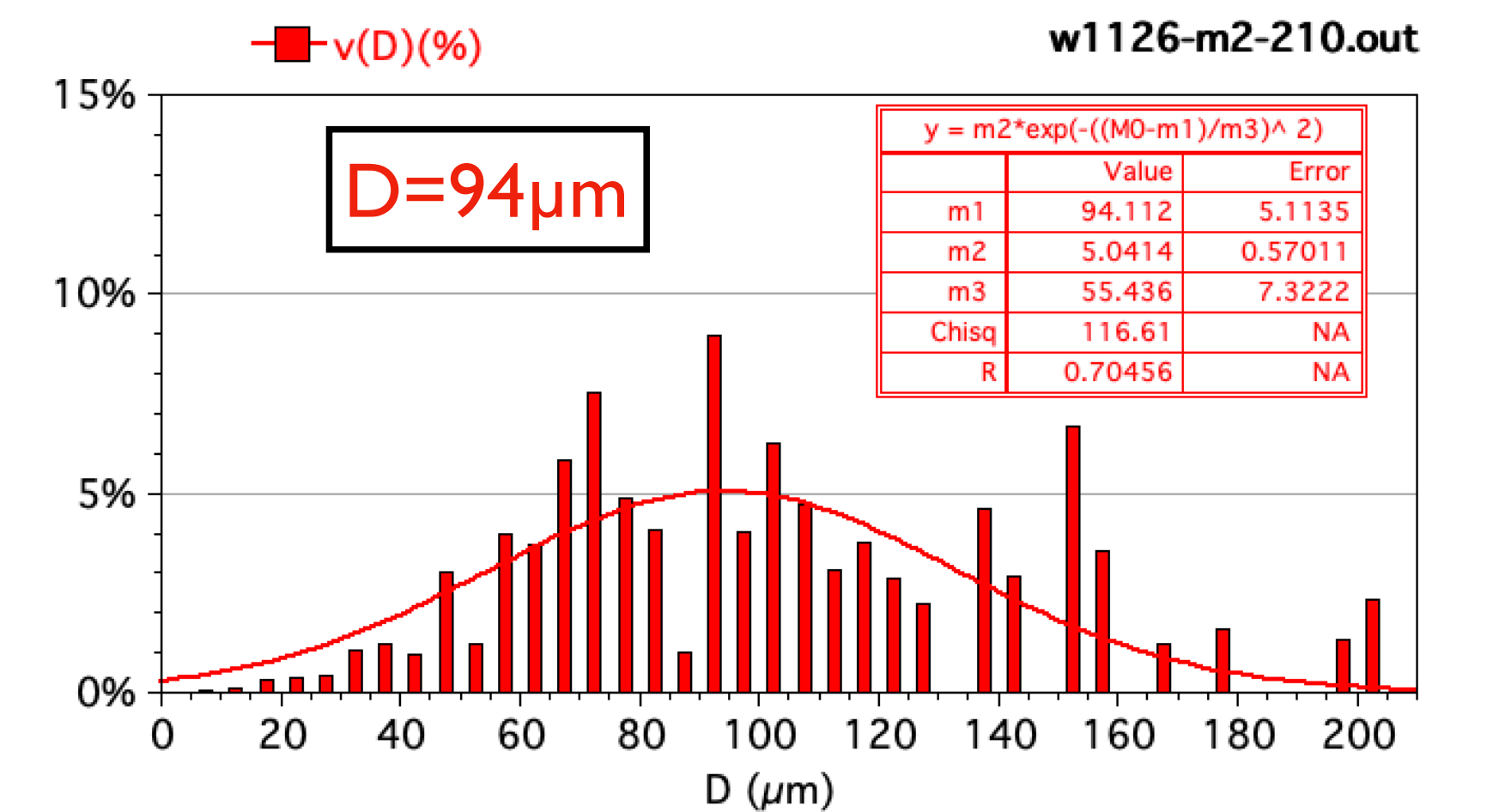
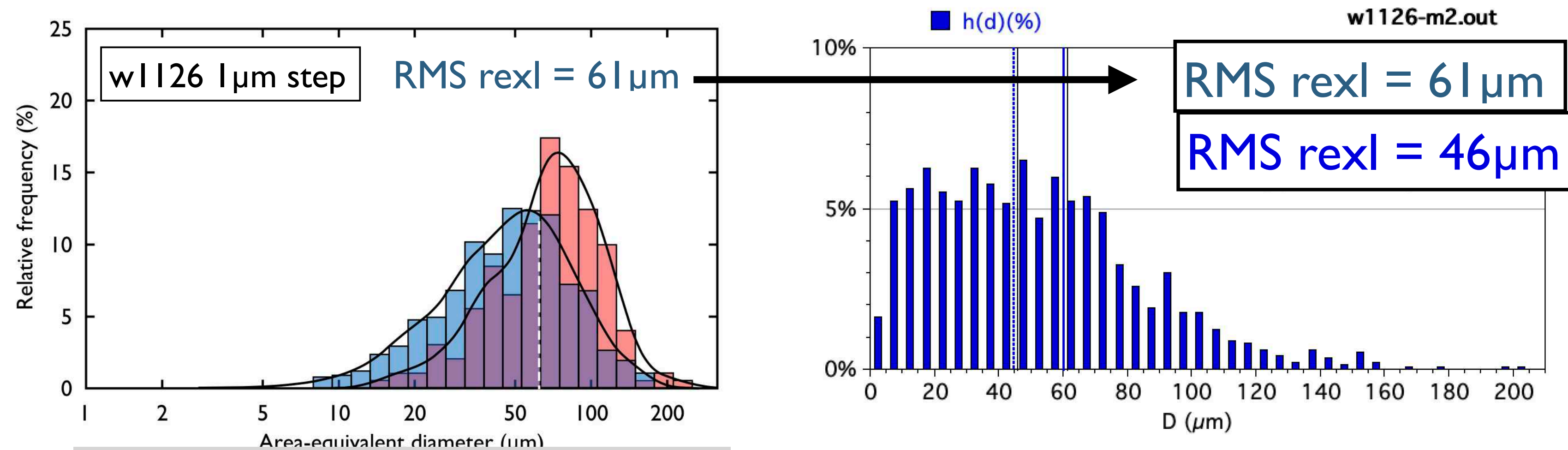
area-weighted of diameters of 2Dsections  $\neq$  diameters of 3D grains !!



# optional excursion: harping on the RMS



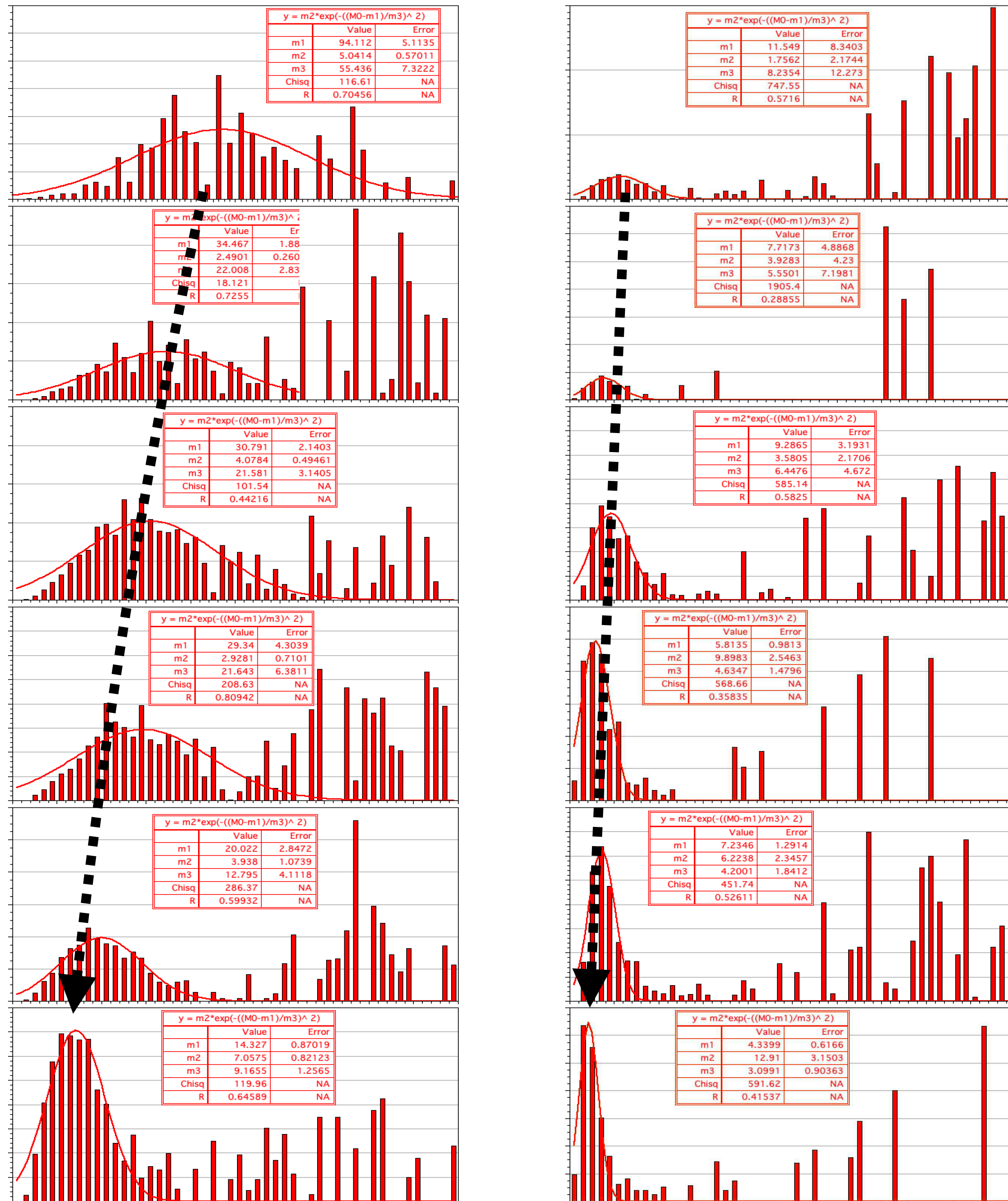
2D ⇒ 3D



Kidder et al. 2016 use GOS to identify rexl grains

recrystallized grains have to be selected from h(d) before calculating RMS (→ bias)  
 stripstar (2D→3D) converts full h(d) to h(D) and volume weighted v(D)  
 the significant grain size is derived from the mode of v(D) and is independent of the range of h(d)

# new EBSD piezo $v(D)$ Gauss fit

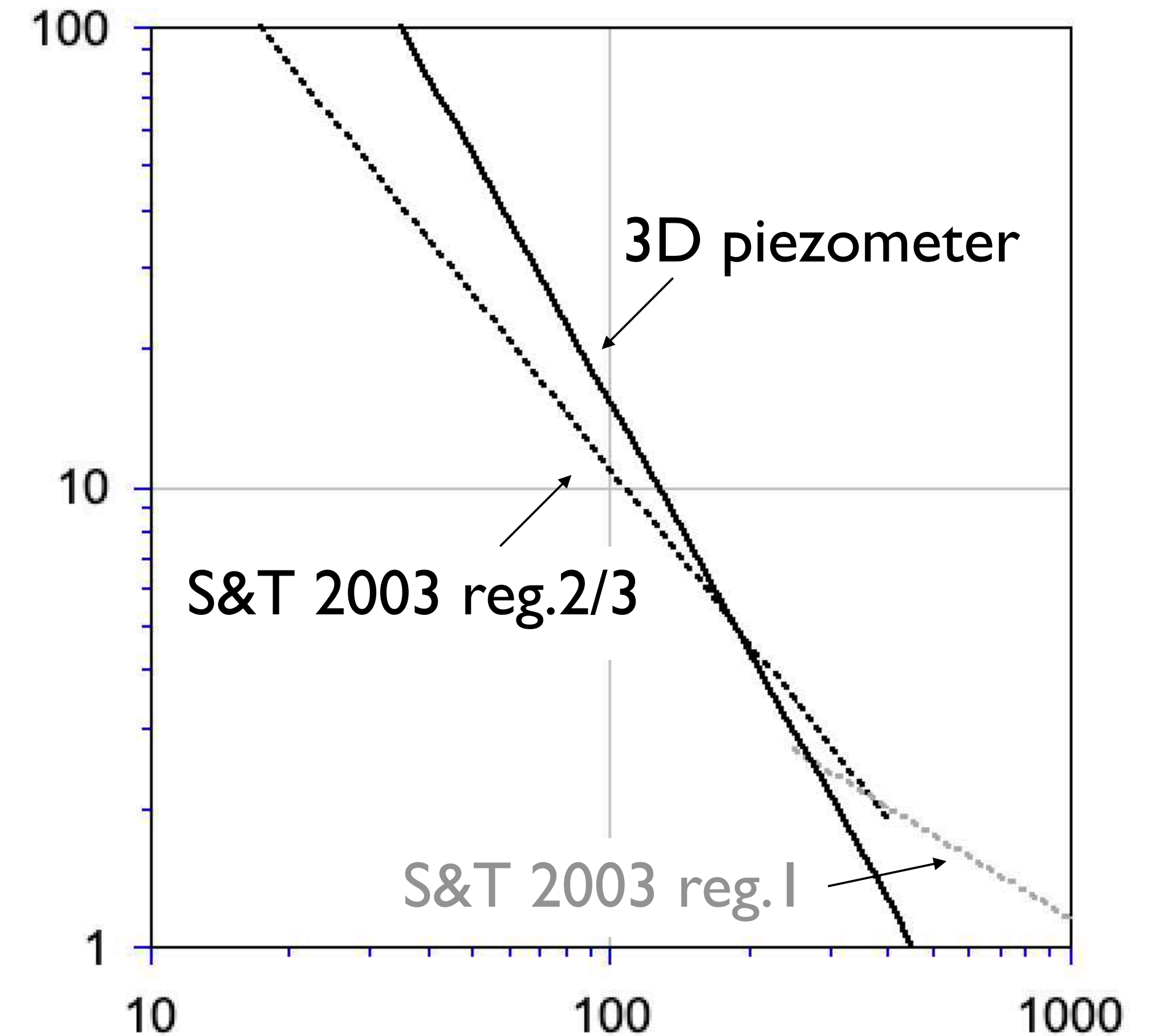
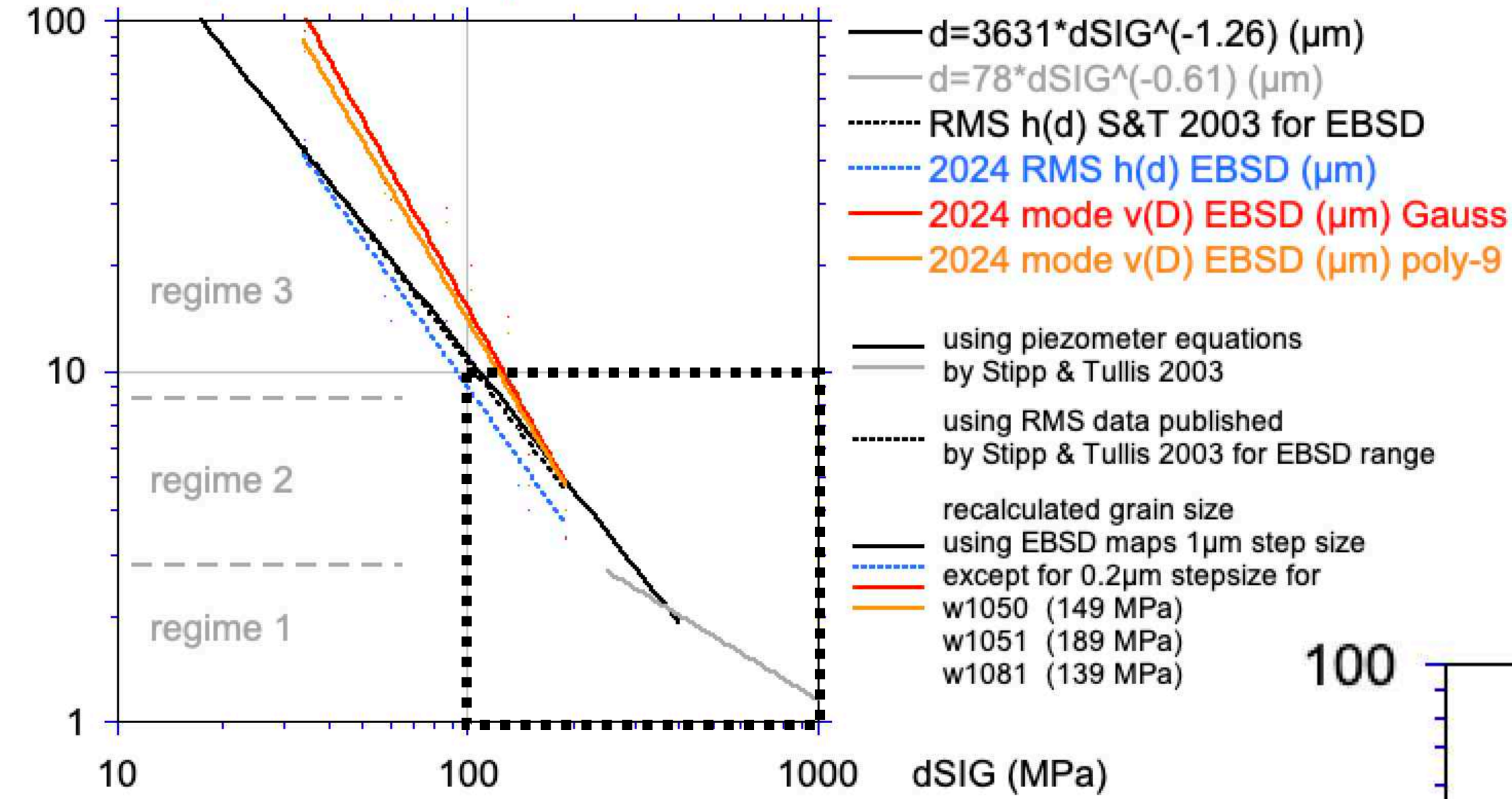
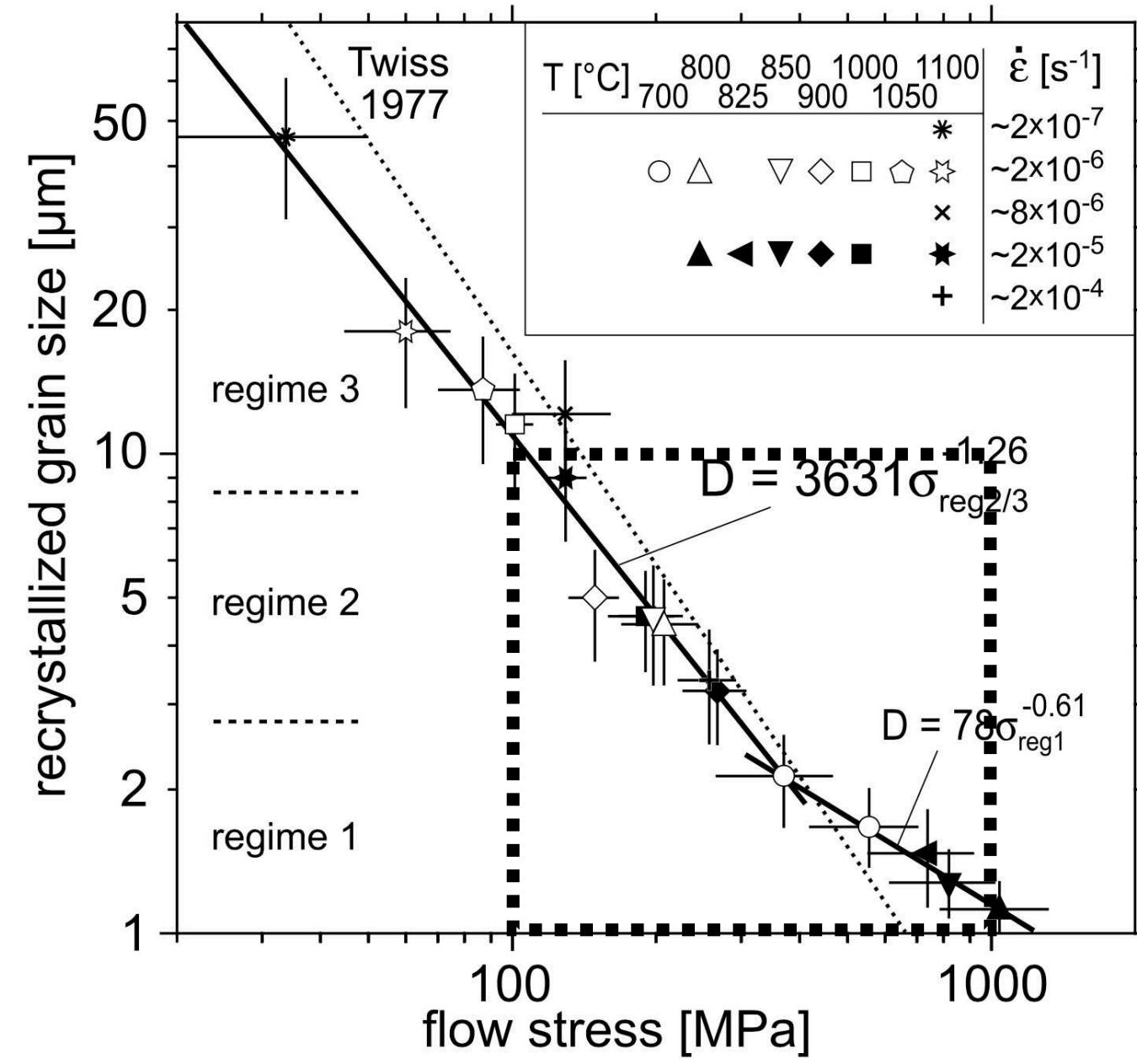


$\Delta\sigma$



	(MPa)	D ( $\mu\text{m}$ )
w1126-m2	34	94.1
w1143-m2	58	34.5
w1066-m2	60	30.8
w1025-m2	87	29.3
w1024-m10	102	20.0
w1029-m3	130	14.3
w1081-m4	139	11.5
w1081-m5	139	7.7
w1050-m6	149	5.8
w1050-m5	149	9.3
w1051-m6	189	3.3
w1051-m4	189	7.2

# derive '3D piezometer'



$$d = 3631 \cdot \Delta\sigma^{-1.26}$$

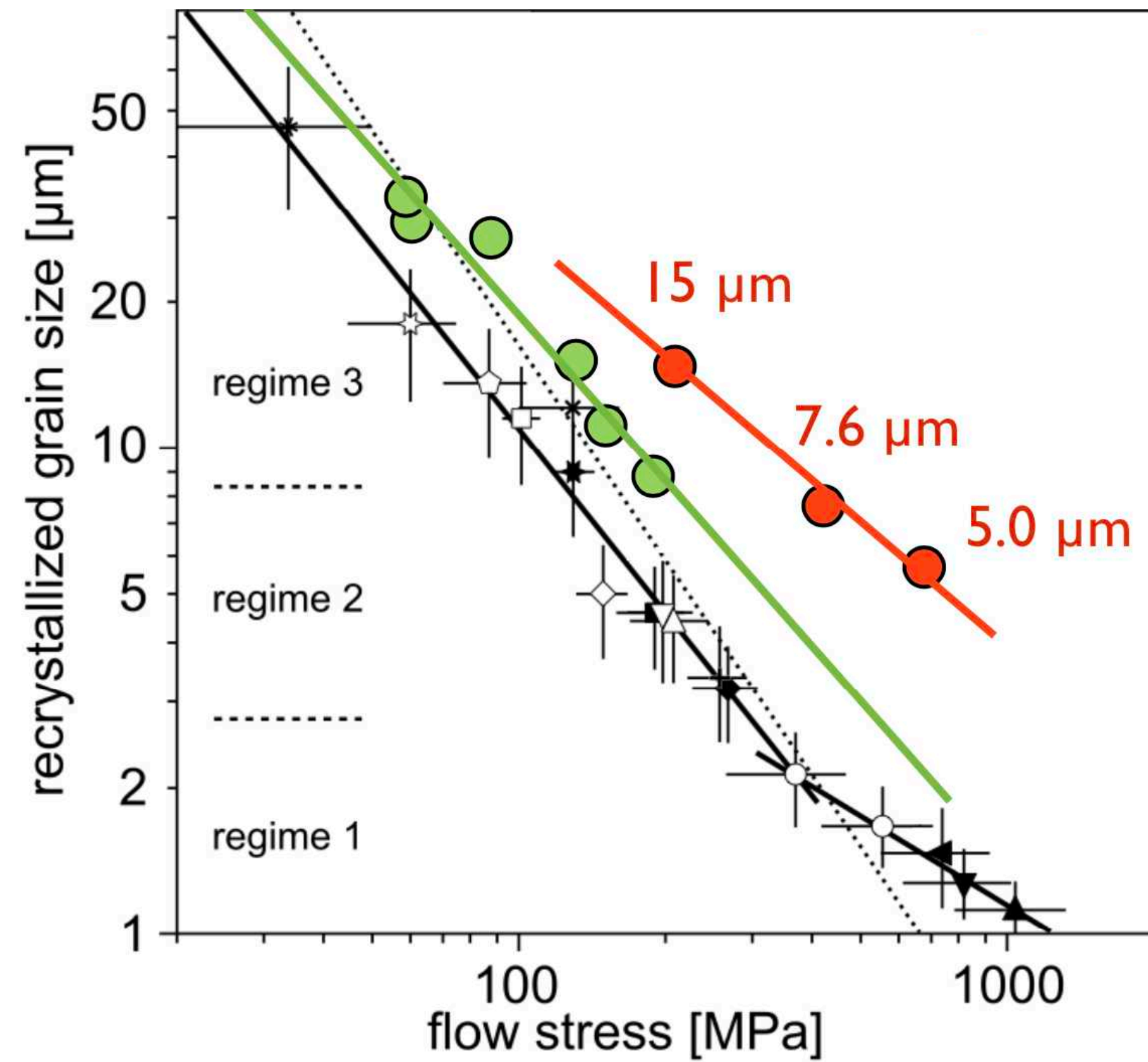
$$d = 78 \cdot \Delta\sigma^{-0.61}$$

$$D = 58060 \cdot \Delta\sigma^{1.79}$$

background for upcoming slides

**back to experimental data**

# EGU 2016 – should we really have been surprised ??



RMS  $h(d)$   
 $d$  = diameter of 2D sections

— Stipp & Tullis 2003

..... Twiss 1977

mode vol%(D)

D = diameter of 3D grains

— Stipp & Tullis 2003  
*recalculated from EBSD maps*

mode vol%(D)

D = diameter of 3D grains

— shear 'piezometer'

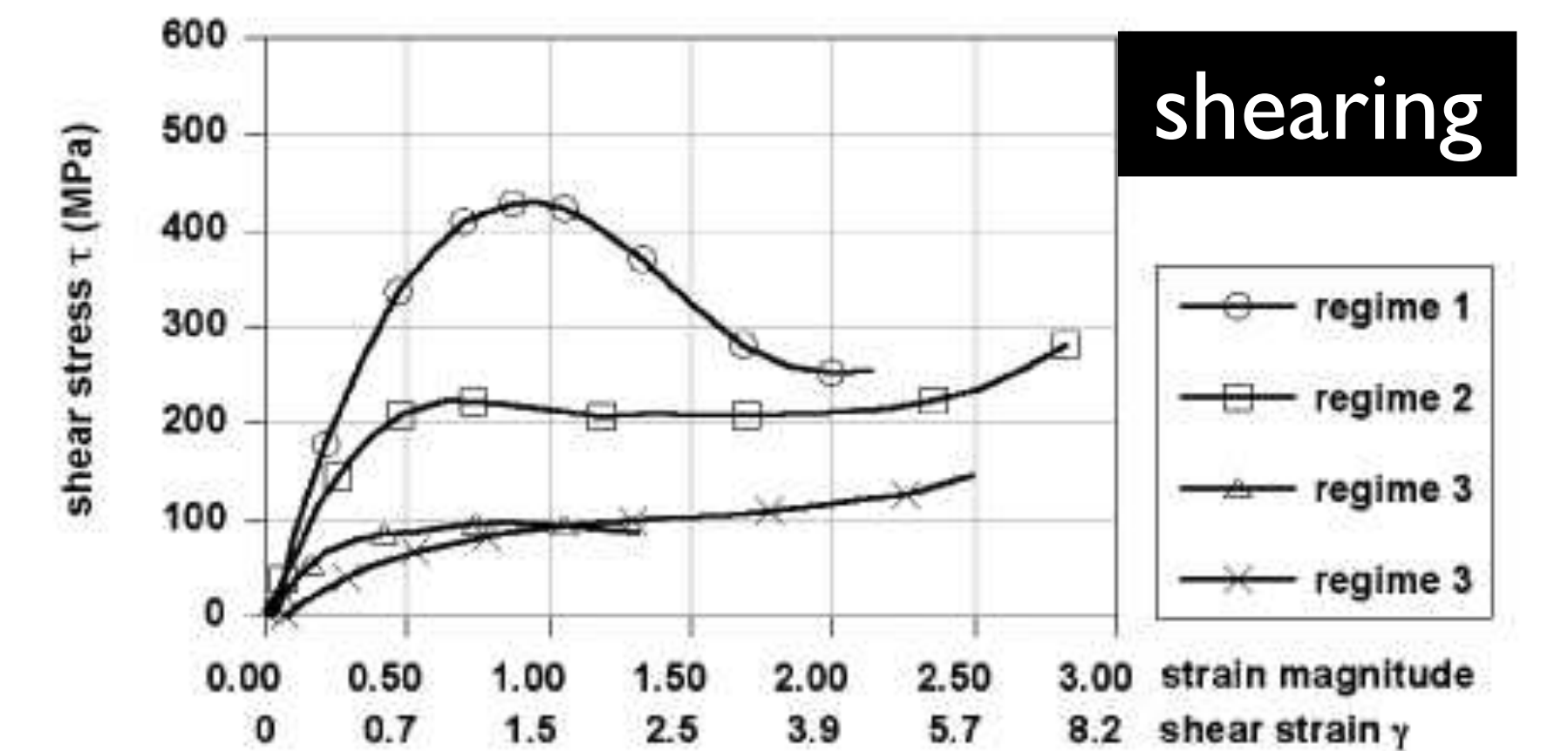
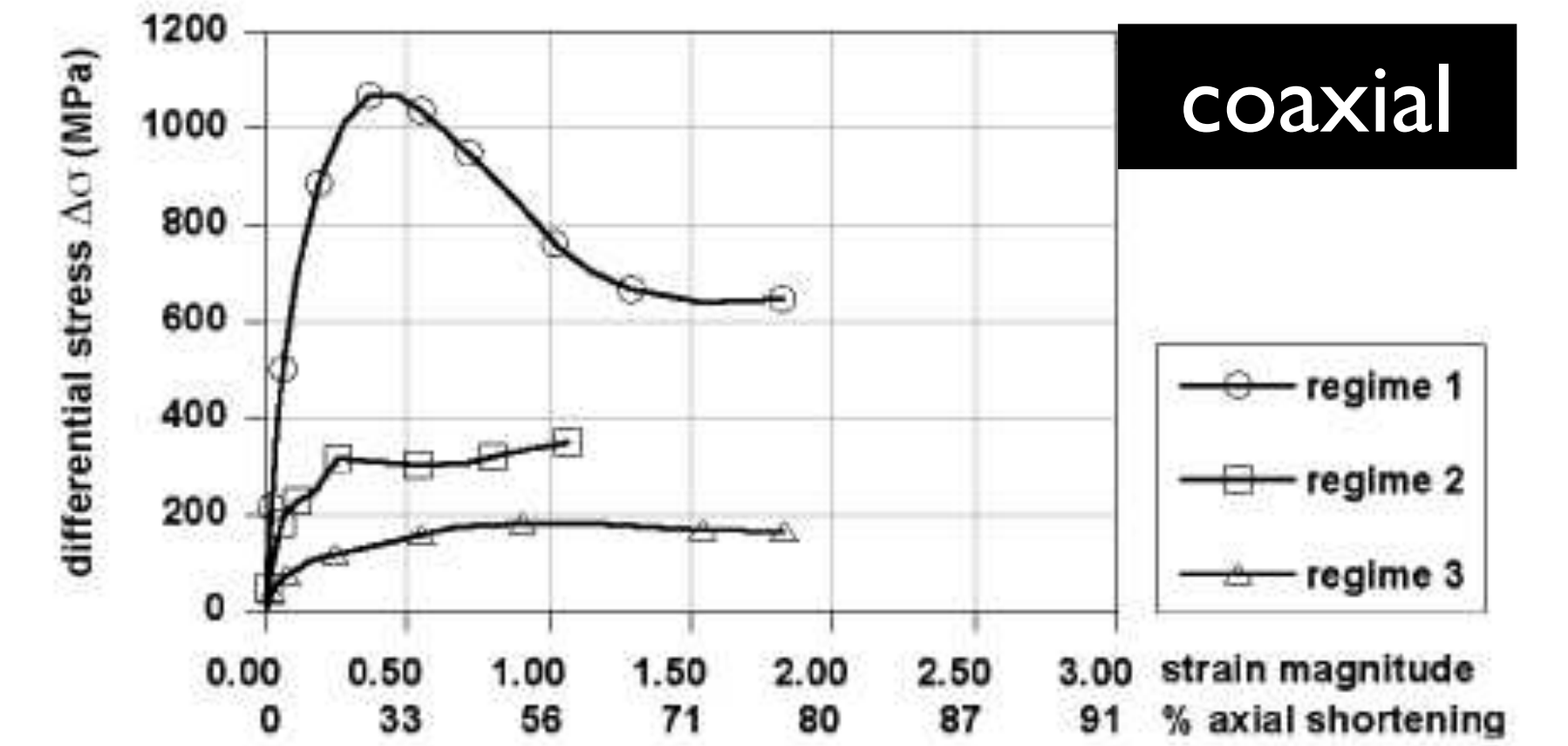
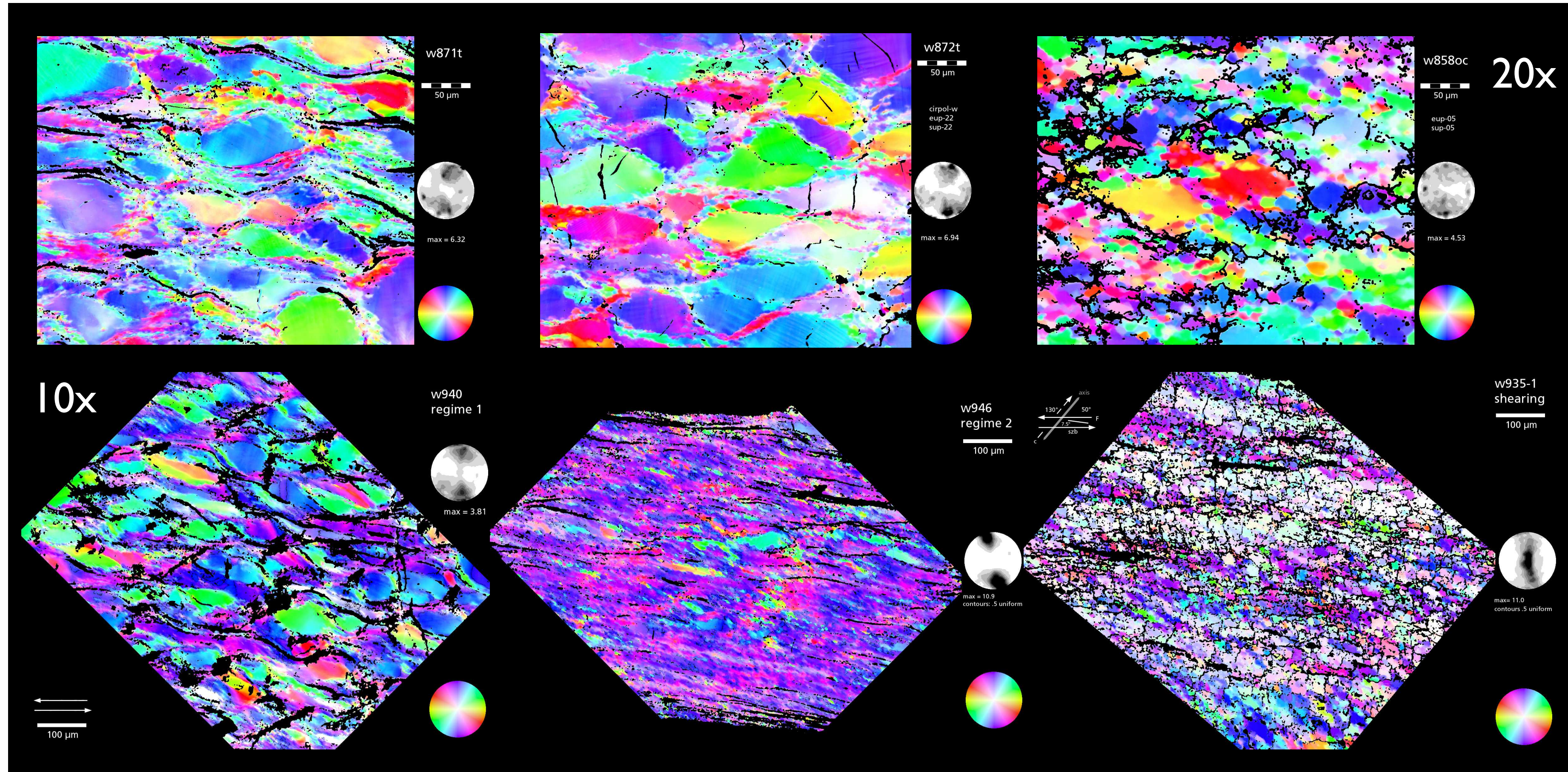
*coaxial experiments  
molten salt assembly*

*shearing experiments  
solid confining medium*

questions:

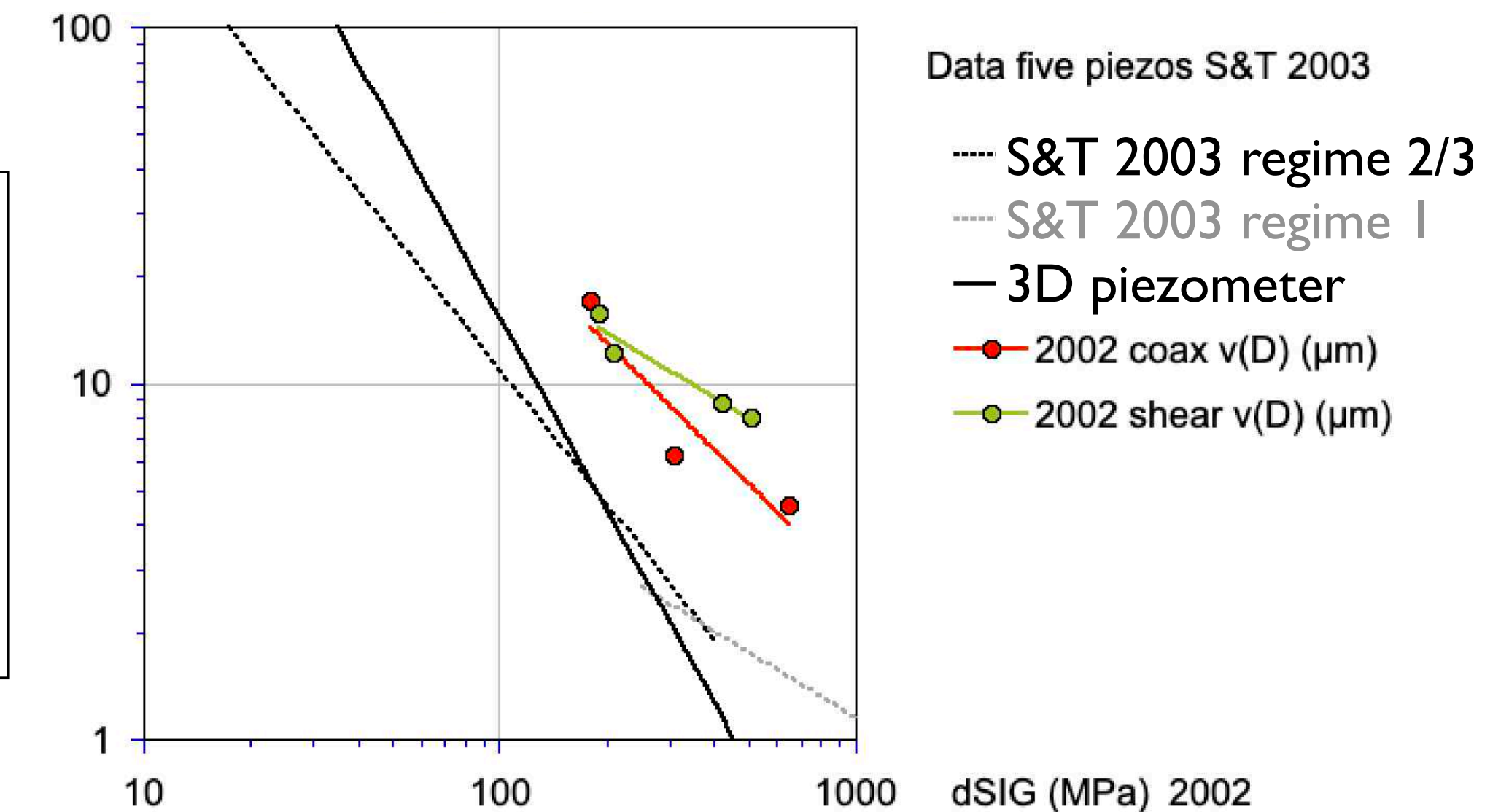
- is BHQ stronger in shear compared to coaxial deformation ?!  
i.e., piezometer not valid for shearing deformation?
- is determination of  $\Delta\sigma$  from shearing experiments incorrect?

# what we could have noted in 2002 ...

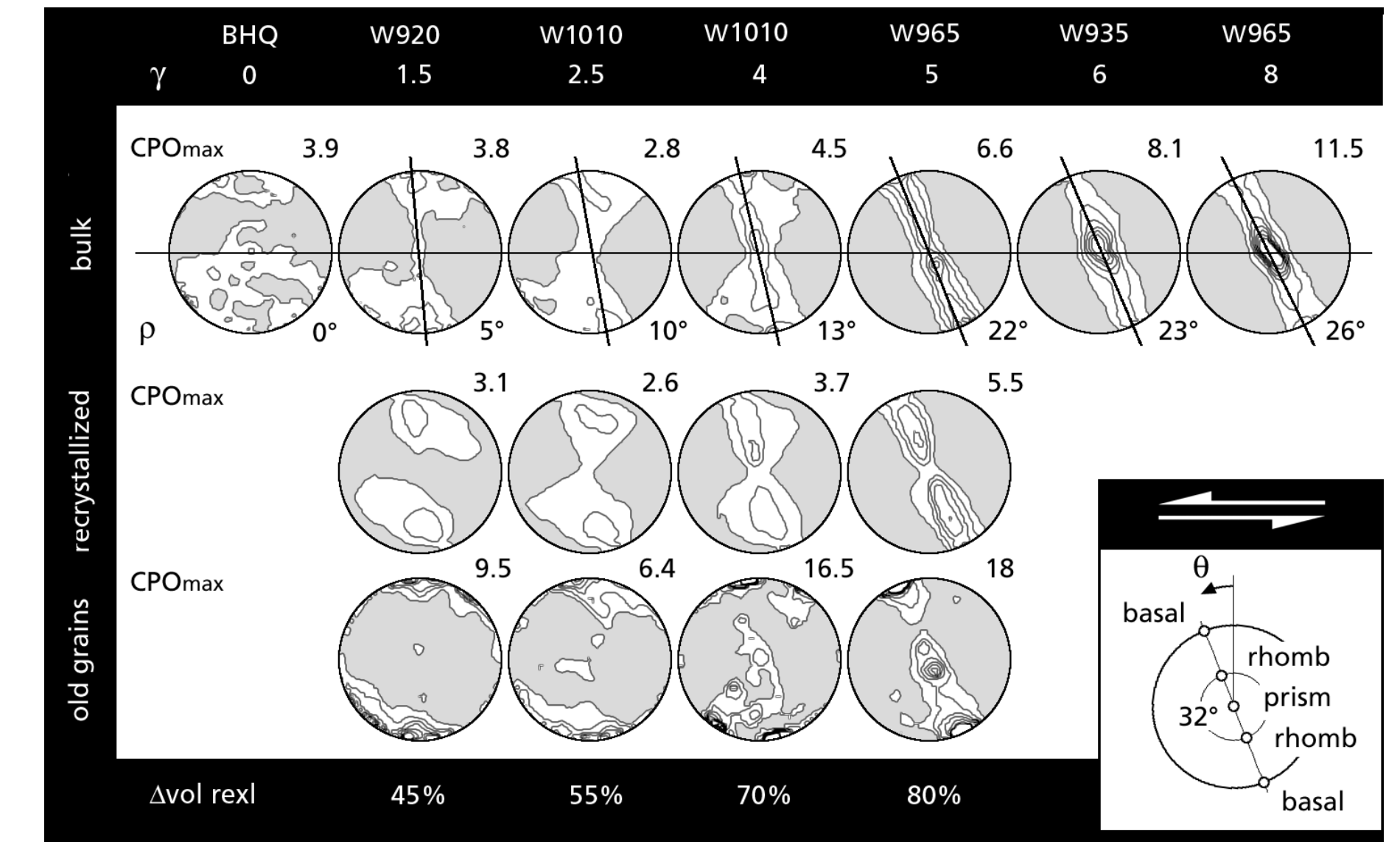
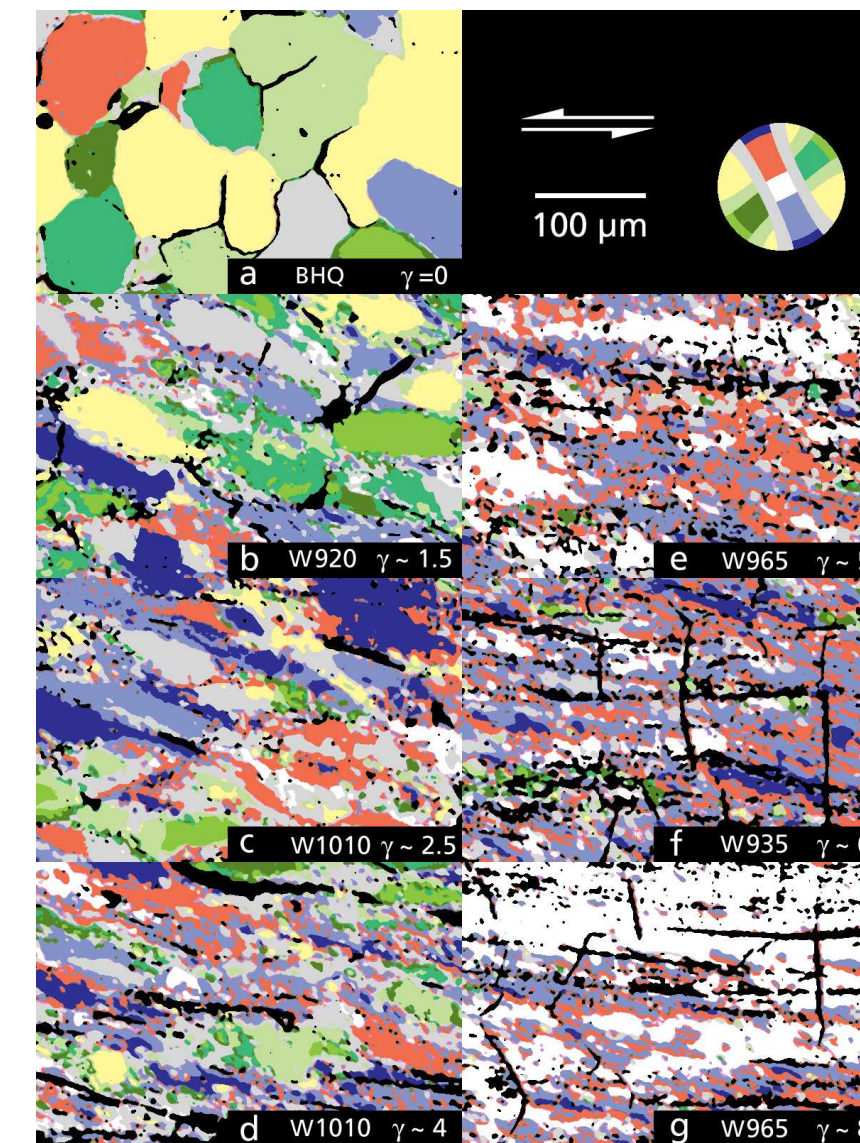
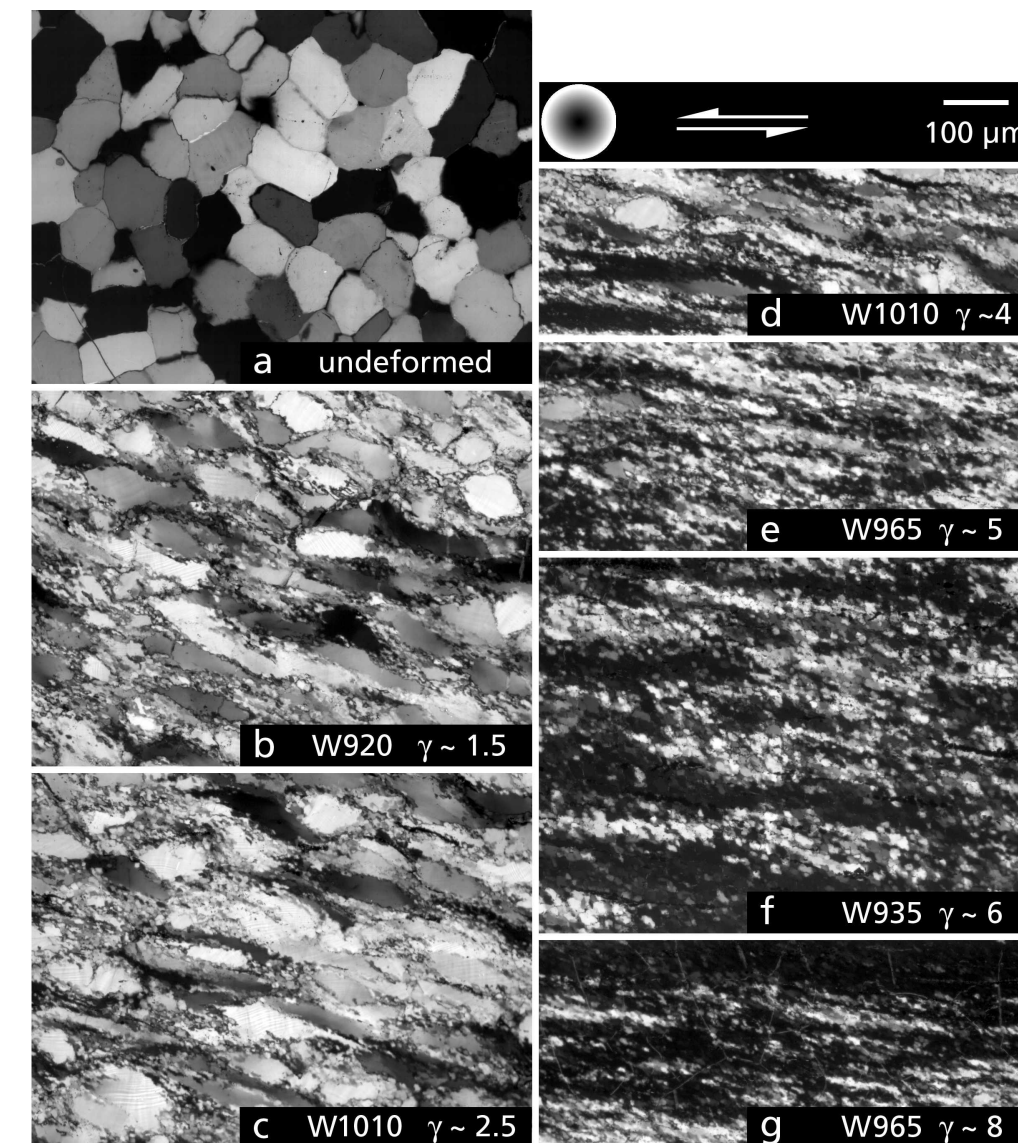
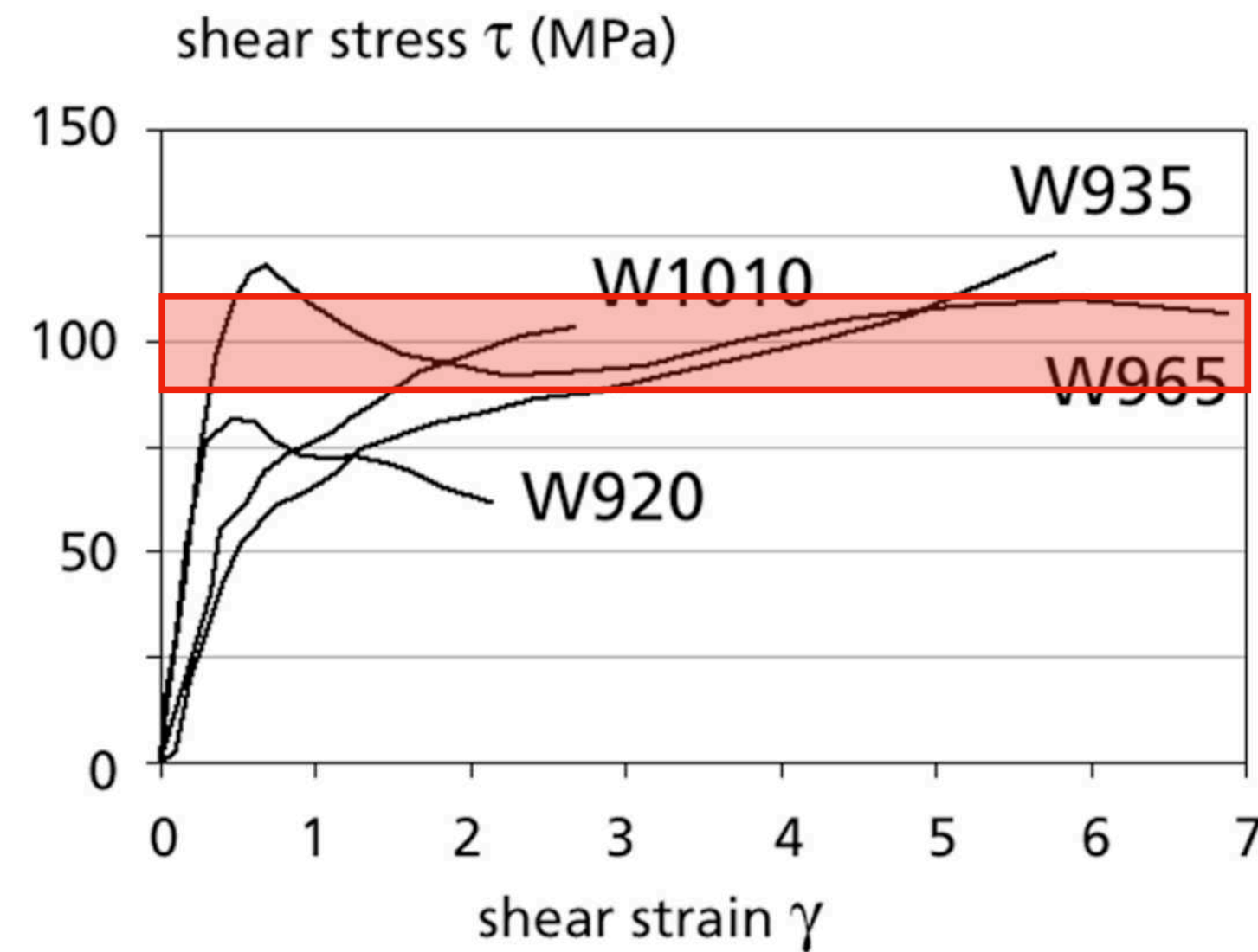


should have seen:  
 $\tau$  ( $\Delta\sigma/2$ ) of shearing experiments  
 $\approx \Delta\sigma$  of coaxial experiments  
 should have guessed:  
 shearing exp. reg.3  $\rightarrow$  I show lower  
 $\Delta\sigma$ -gradient than coaxial exp.

regime	coaxial $\Delta\sigma$ (MPa)	shearing $\Delta\sigma$ (MPa)
1	650	510
2	310	420
3	180	210



# what we could have learned in 2006 ...



recrystallized grain size  
on average is  $\sim 17\mu m$

~~corresponding shear stress~~  
71 MPa

$$\Delta\sigma_{piezo} = 71 \text{ MPa}$$

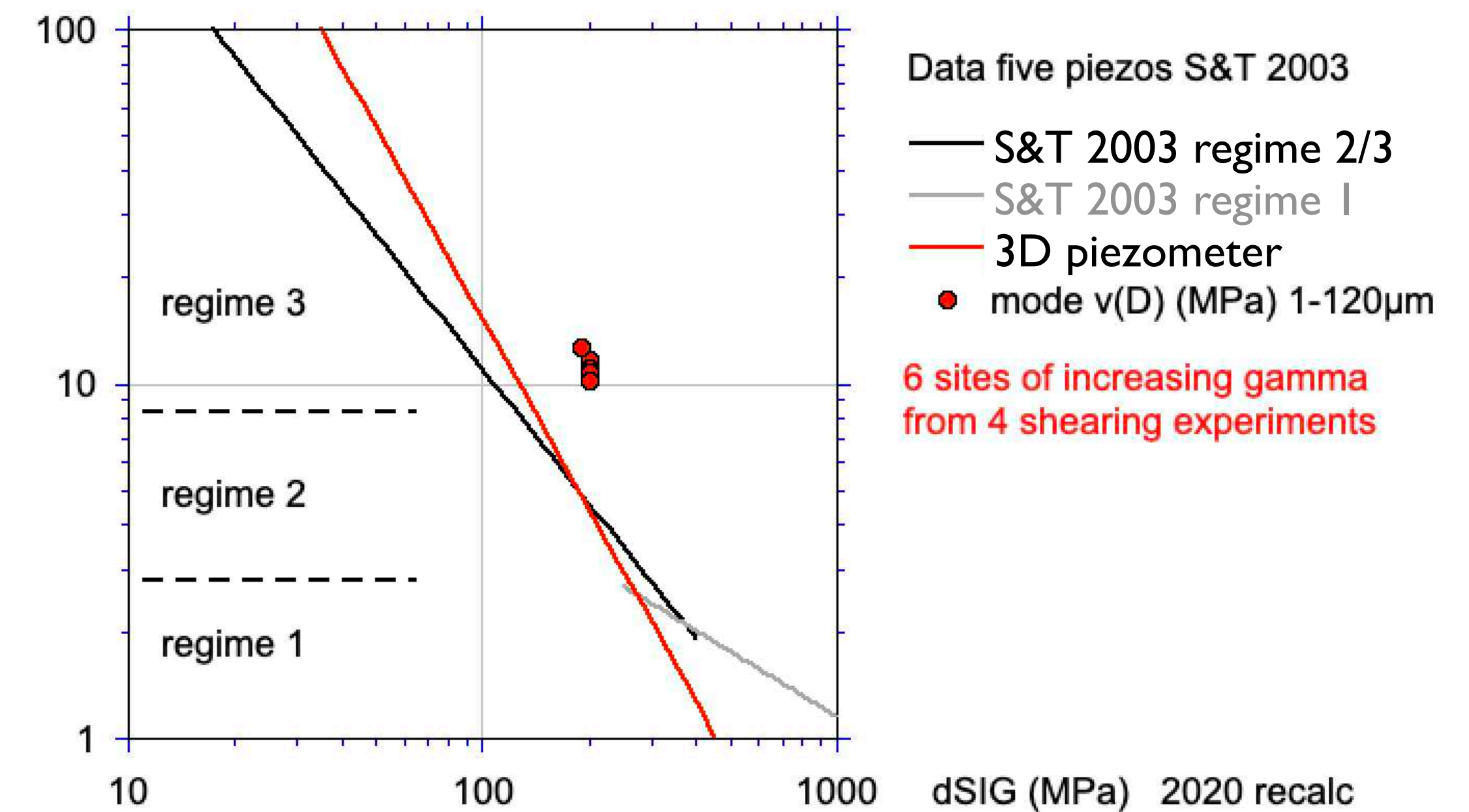
$$\Rightarrow \tau_{piezo} = 35 \text{ MPa}$$

average measured shear stress was 100 MPa

$$\Delta\sigma_{meas} \approx 200 \text{ MPa}$$

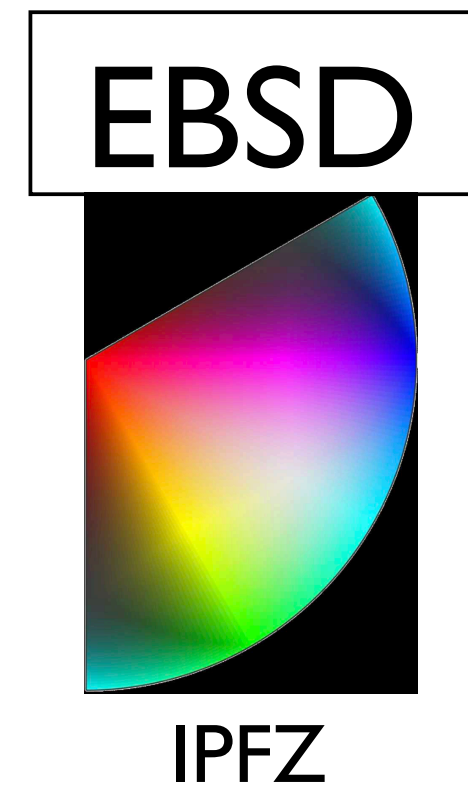
$$\Leftarrow \tau_{meas} \approx 100 \text{ MPa}$$

- the difference between stress values is  
not "reasonable"

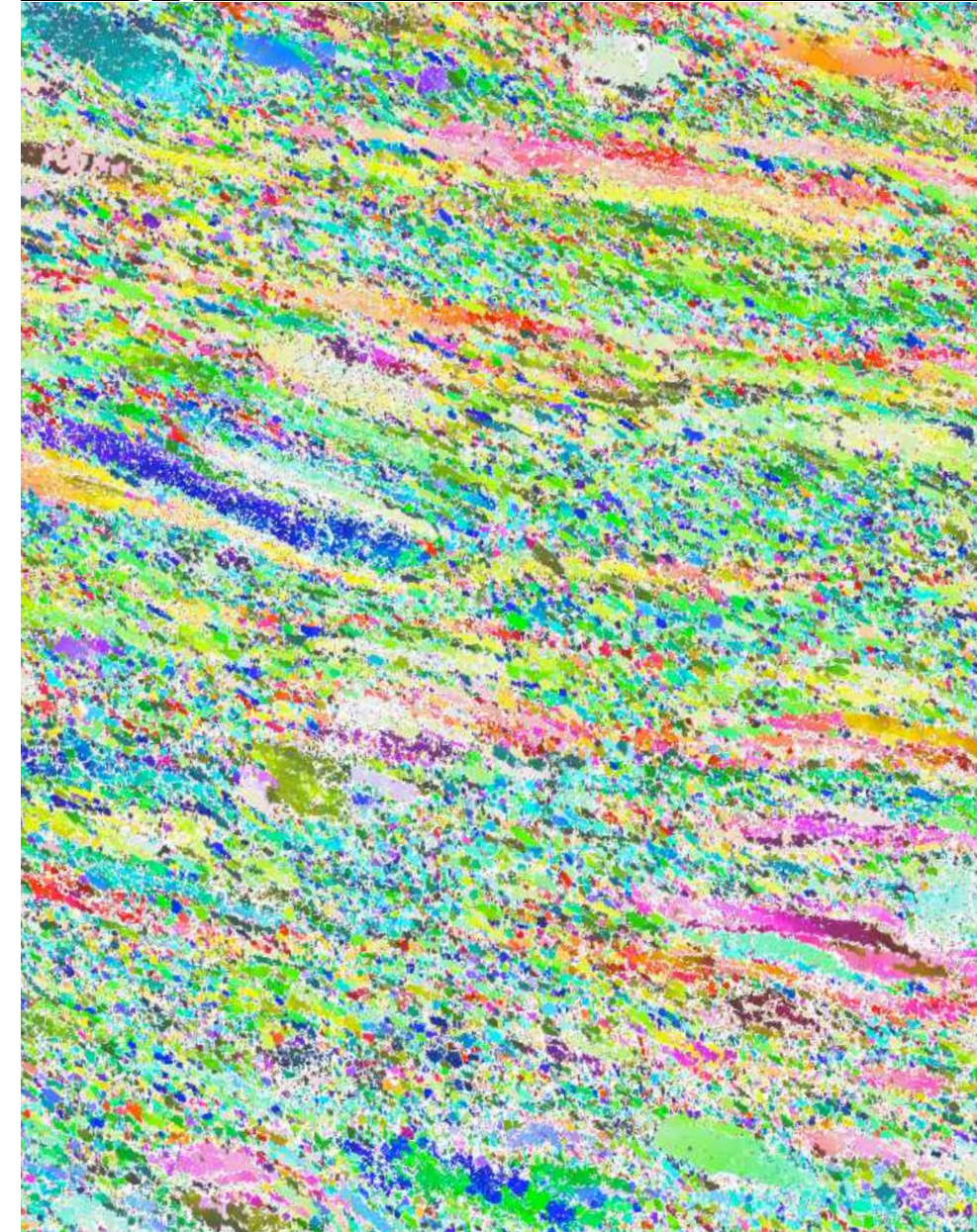


- solid medium shear samples are significantly  
stronger than molten salt coaxial samples !!

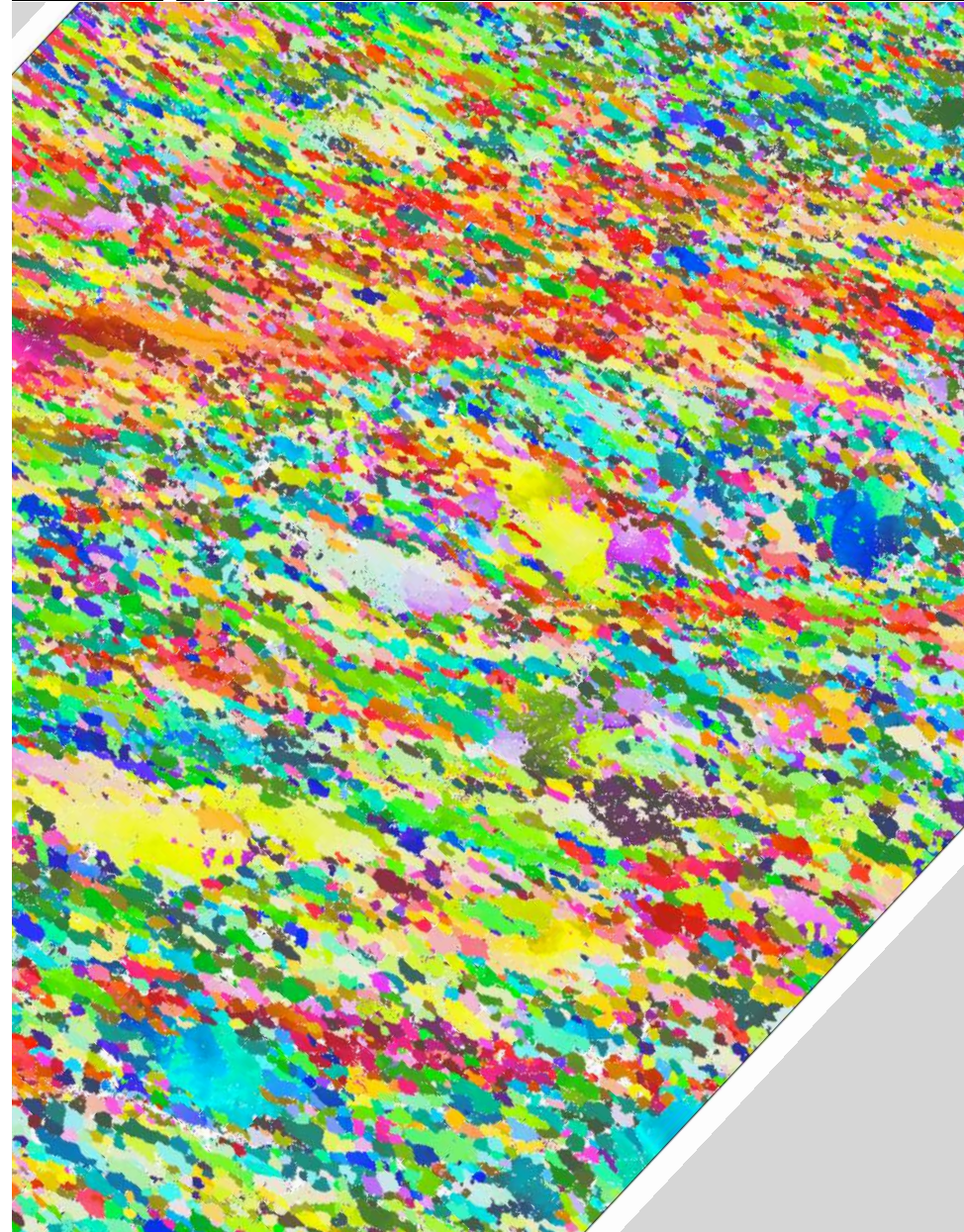
# 2017 old sample at high resolution



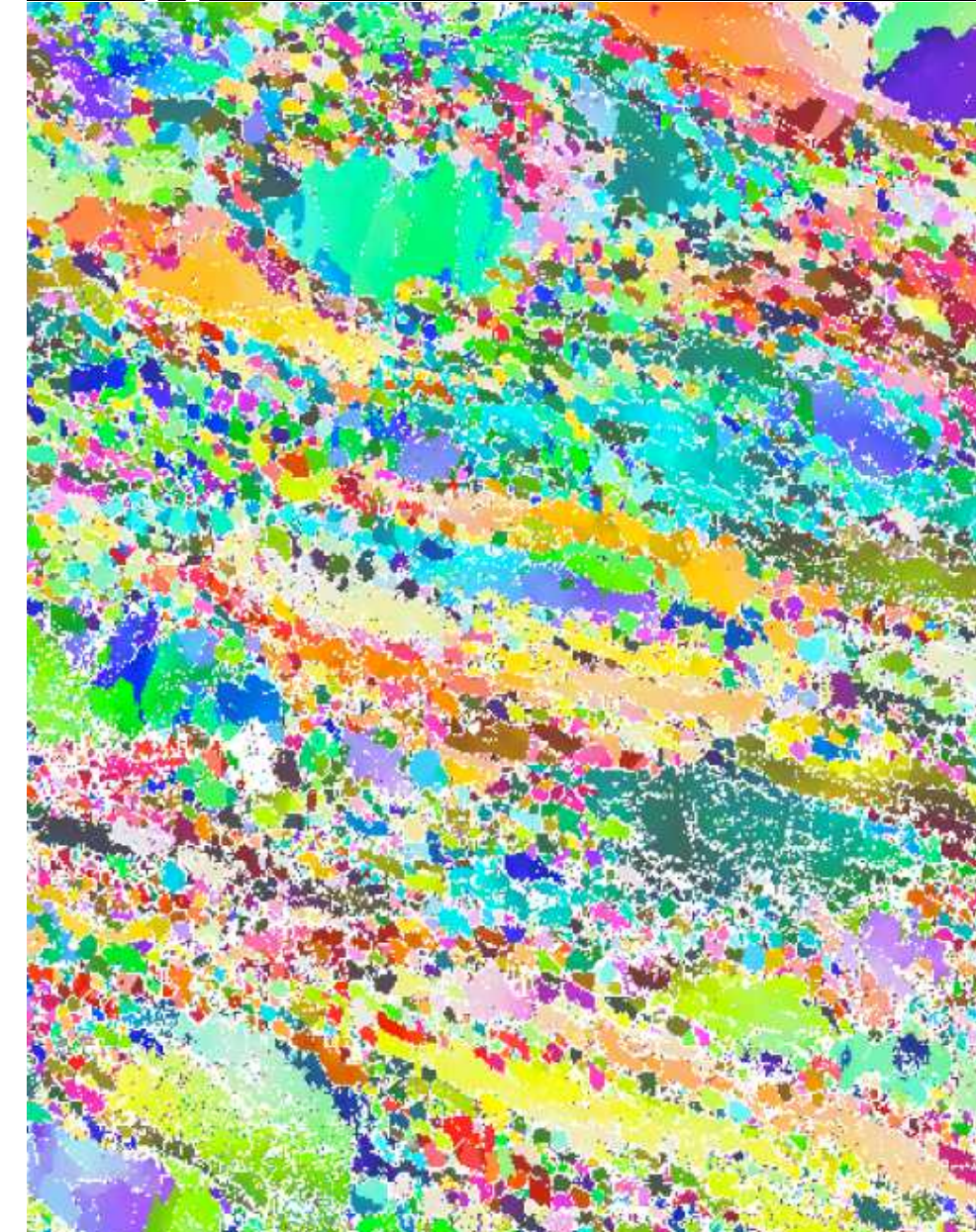
regime 1 - w1092



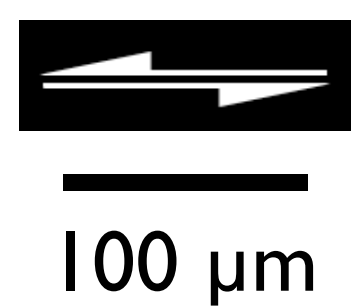
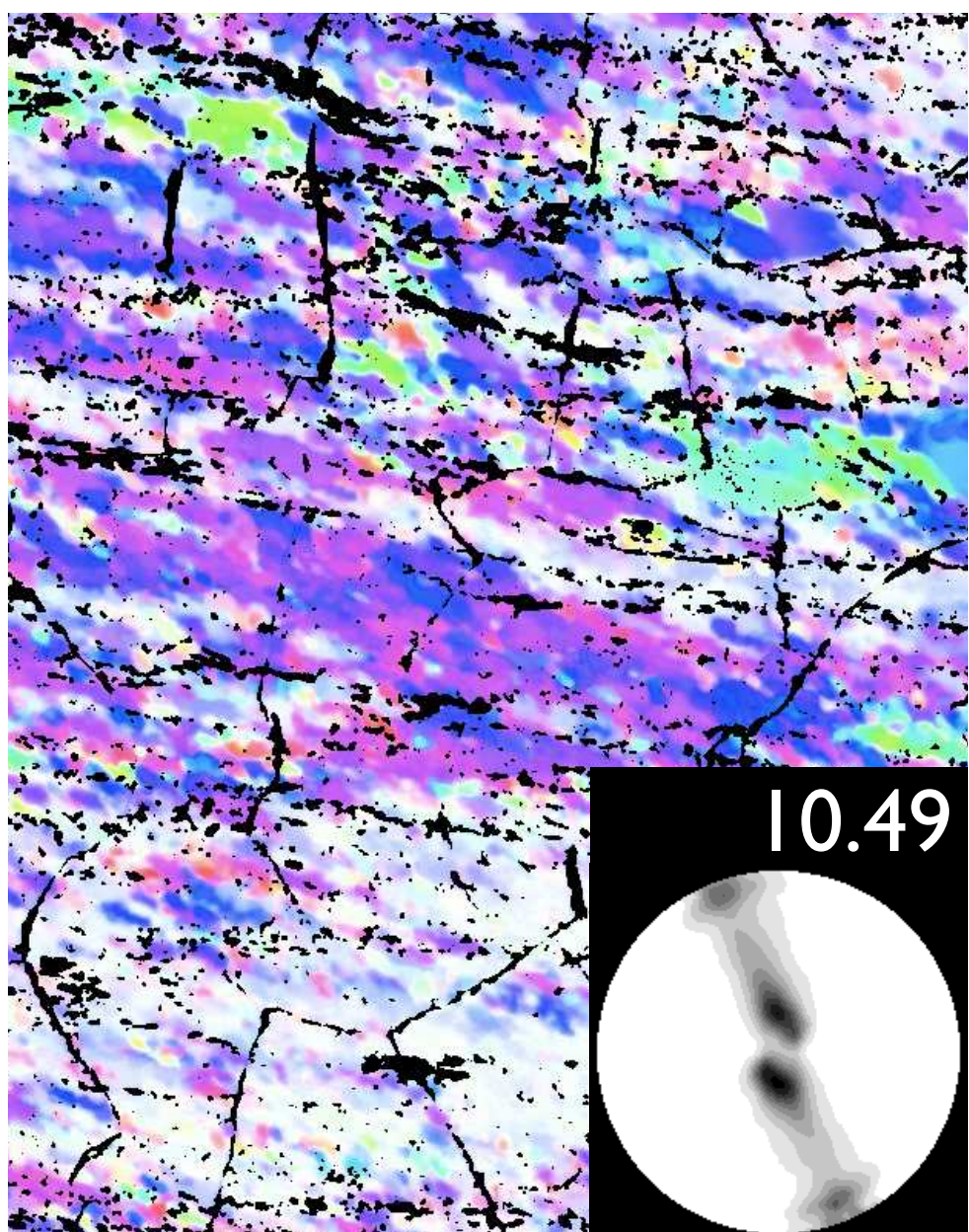
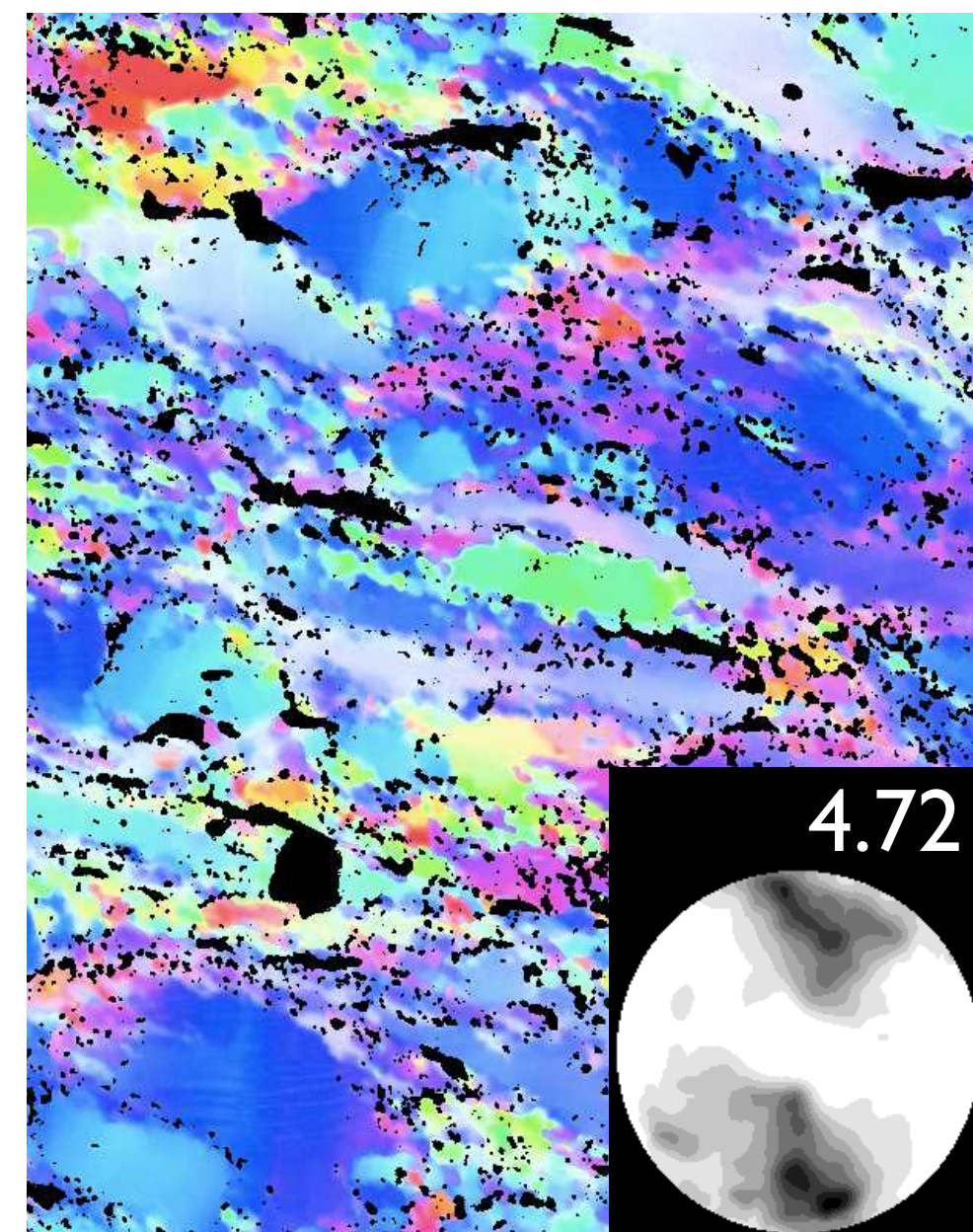
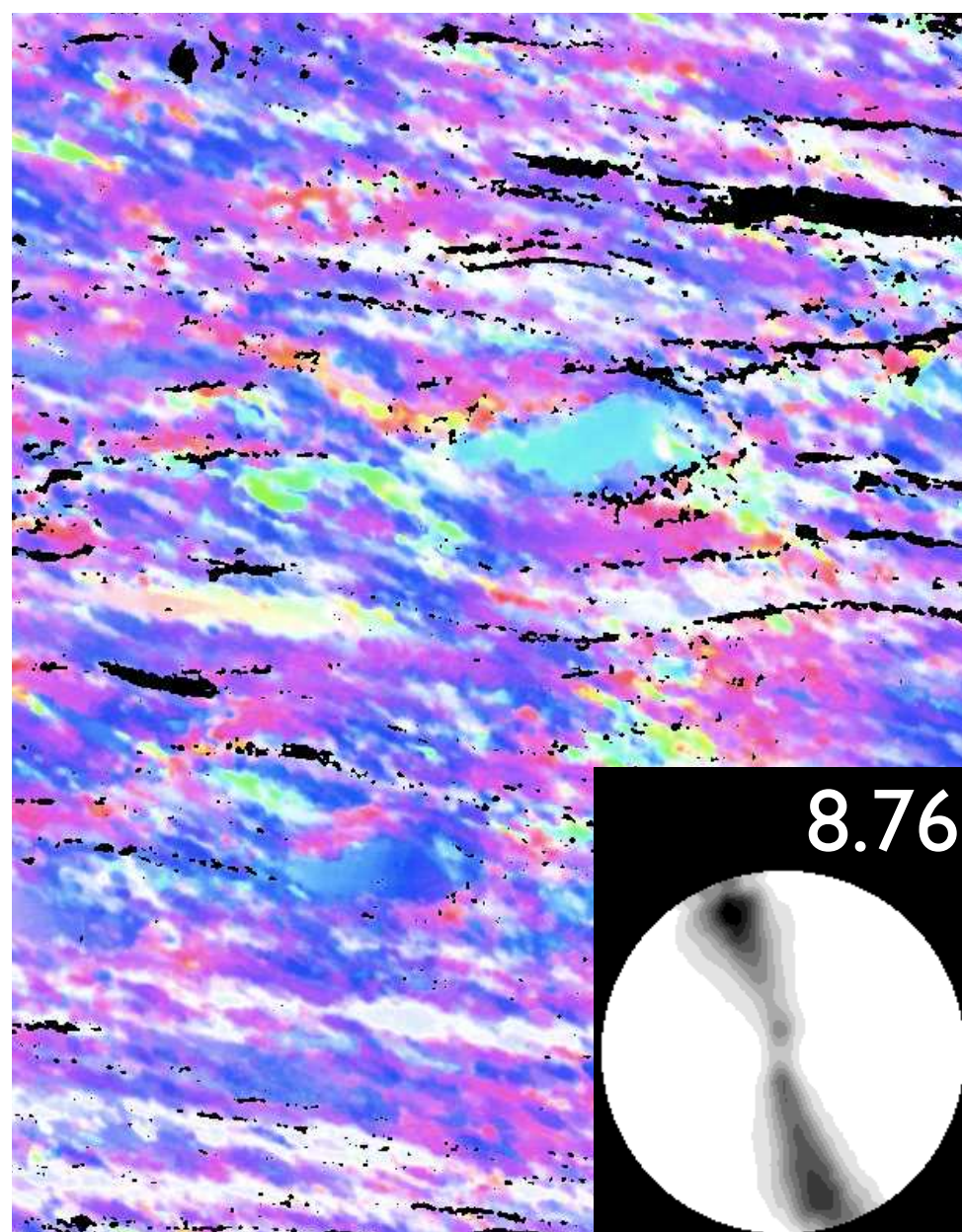
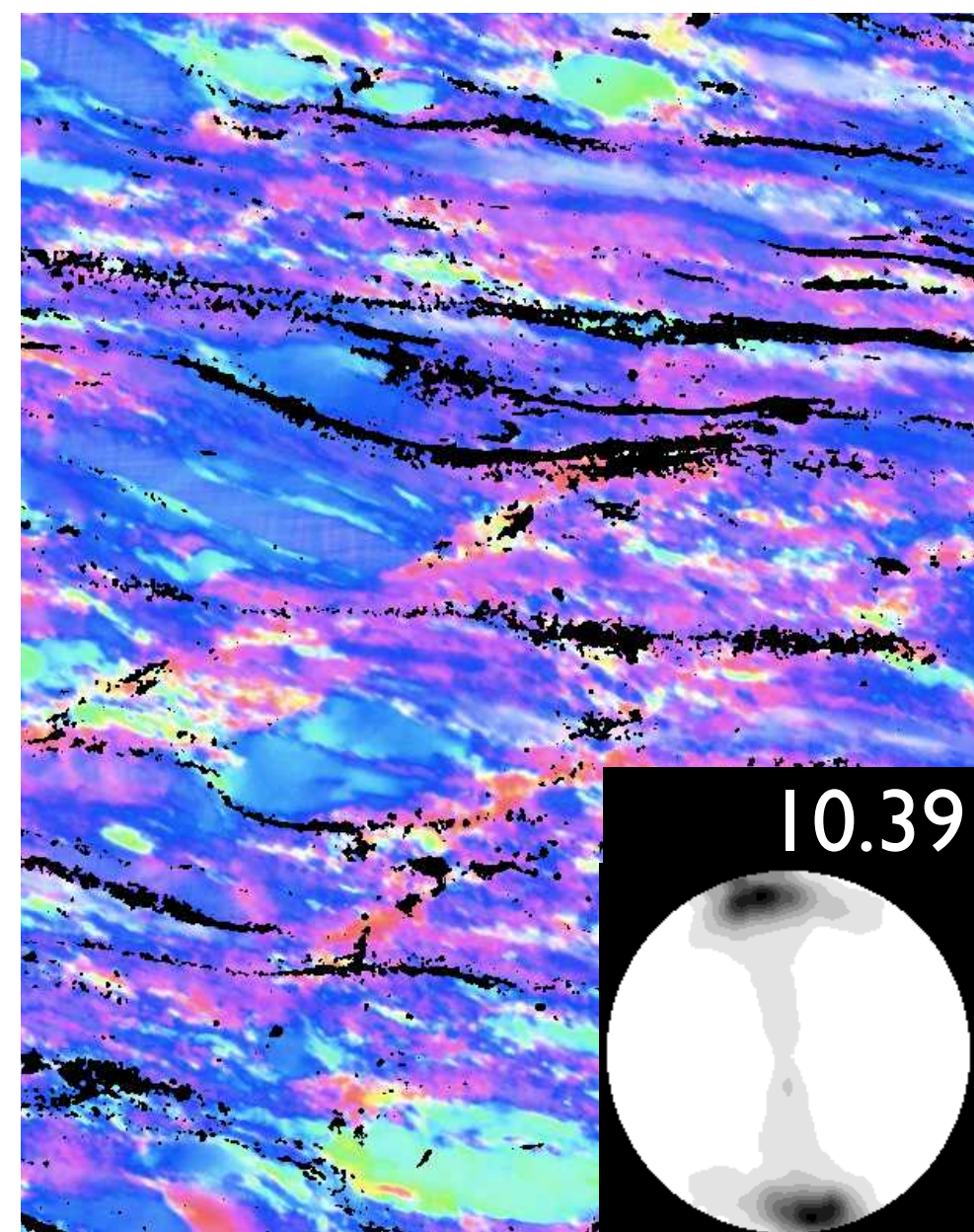
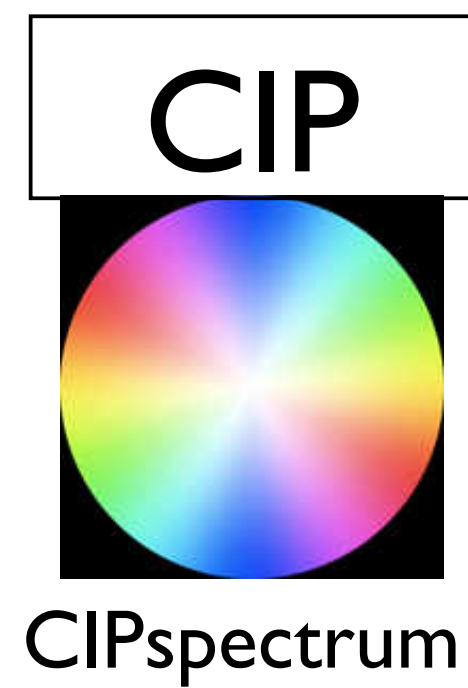
regime 2 - w946



regime 3 - w1010

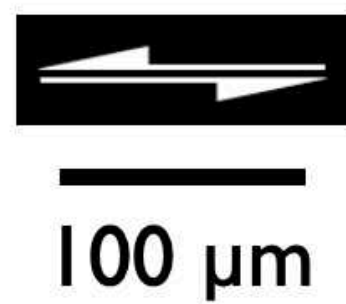
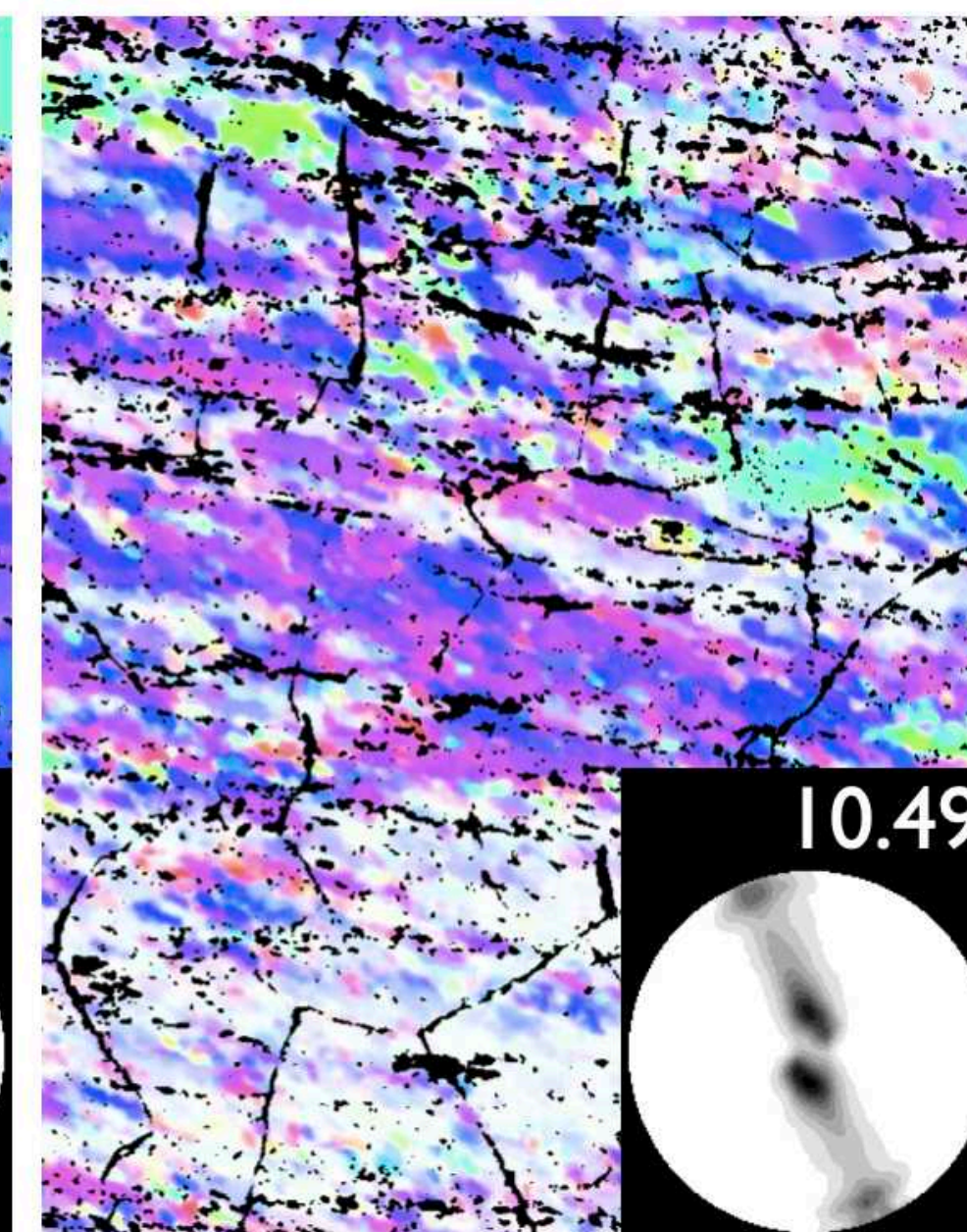
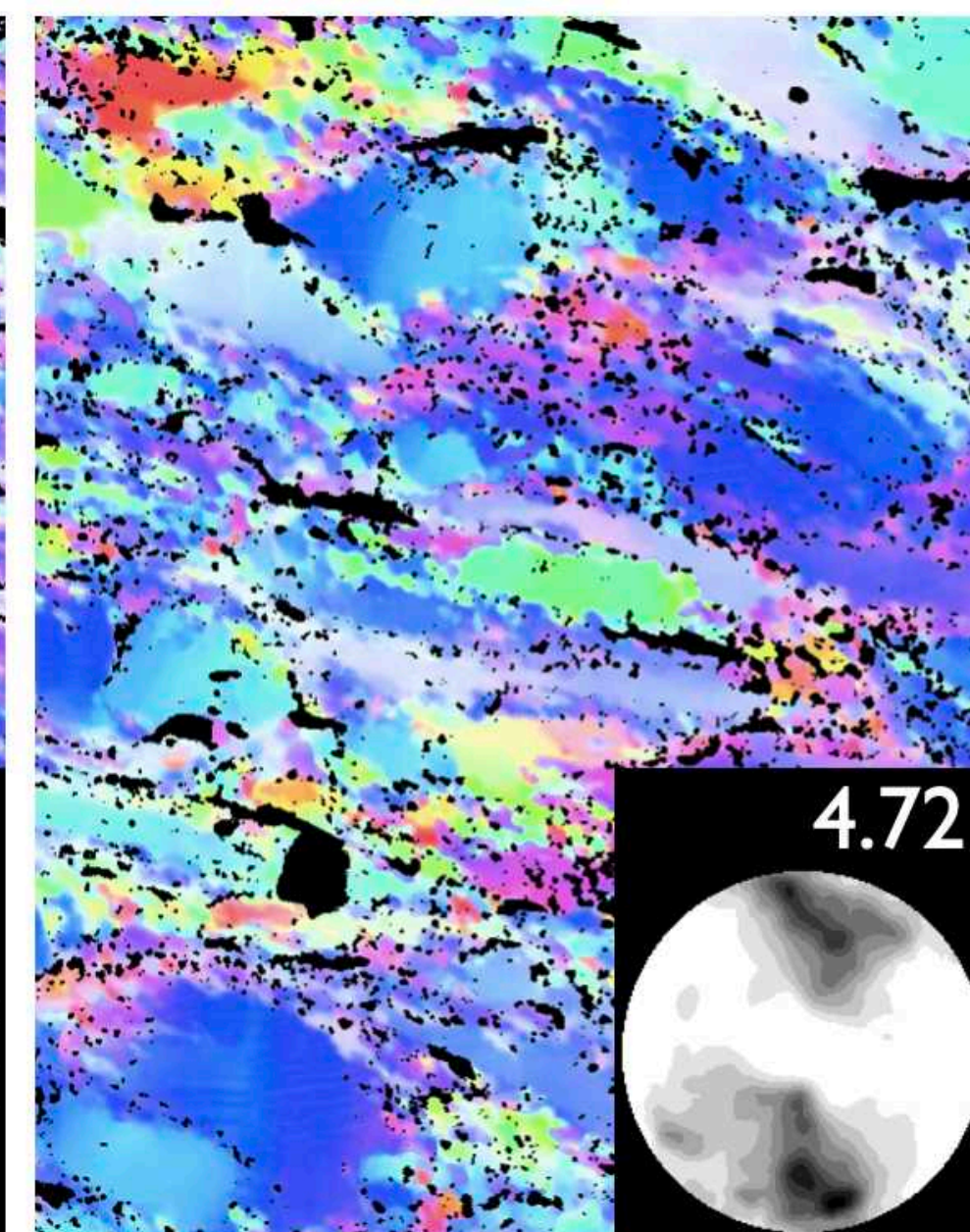
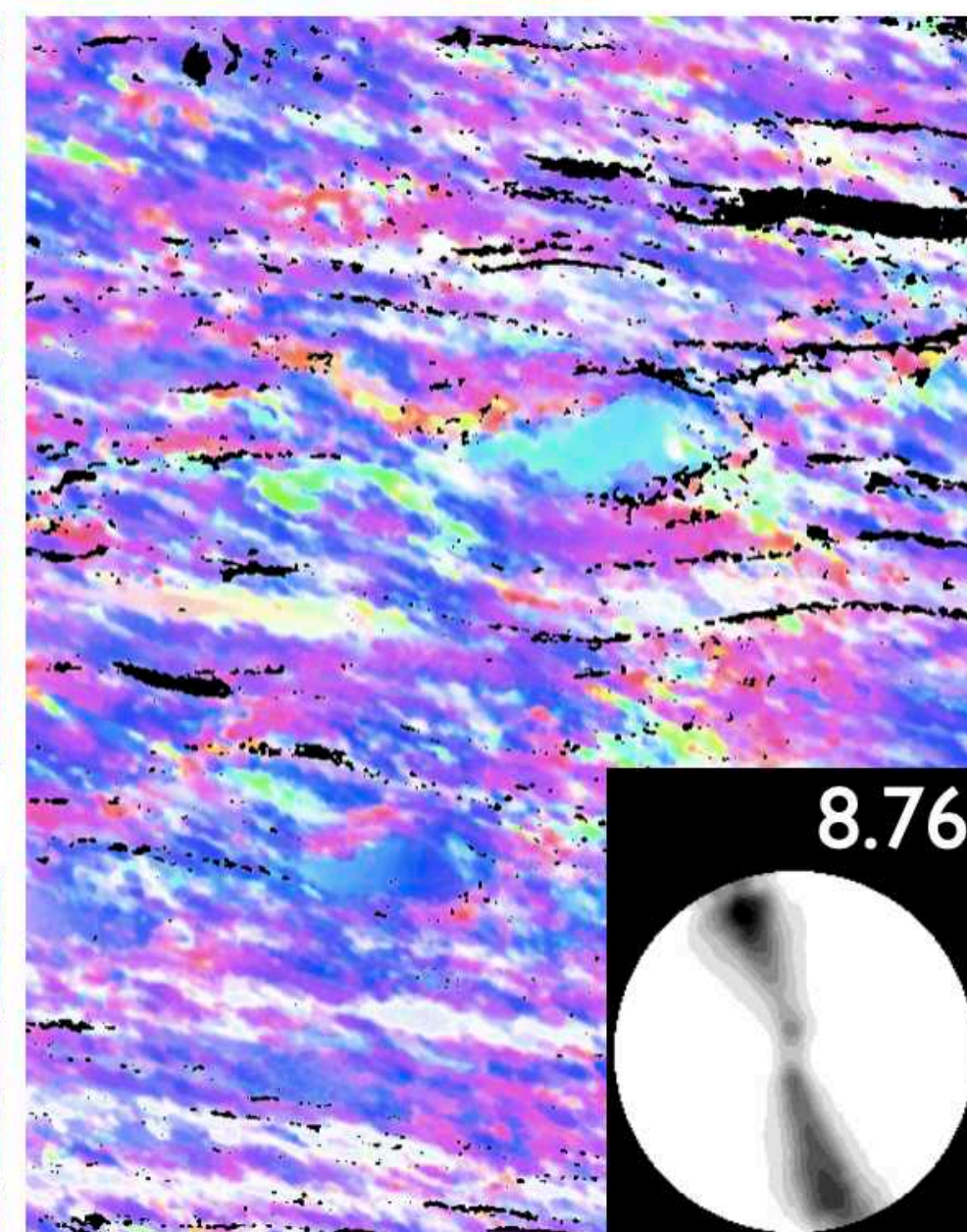
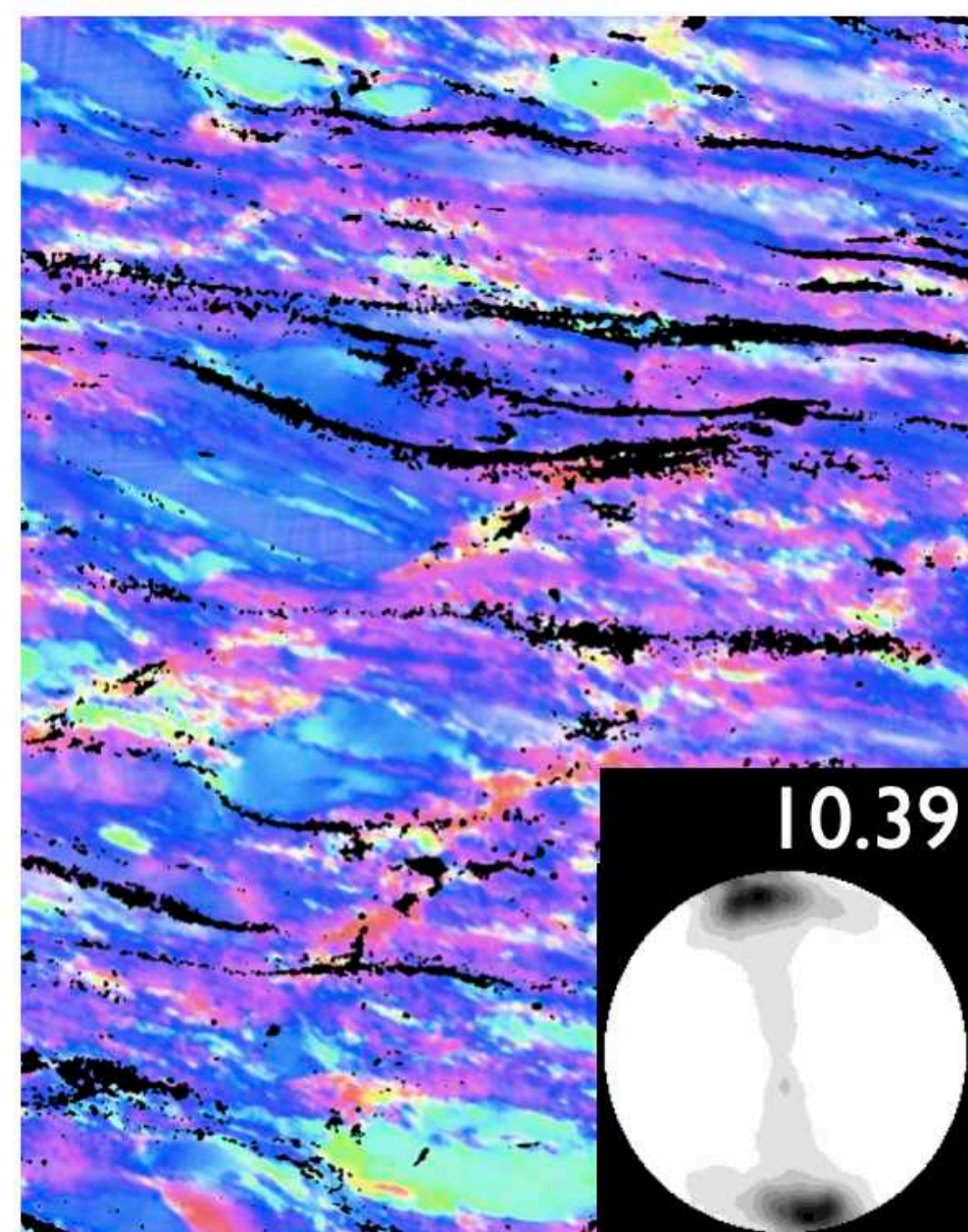
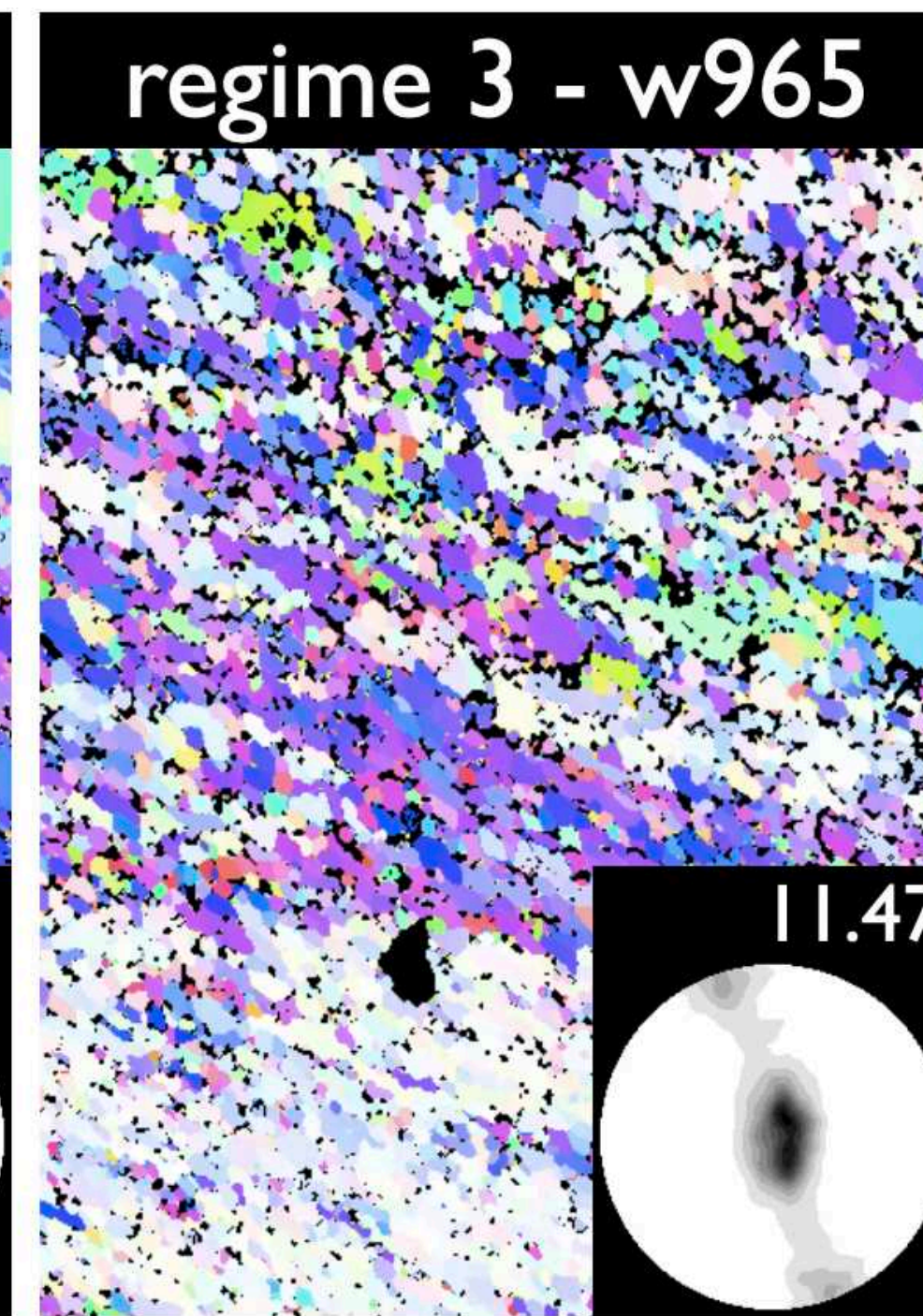
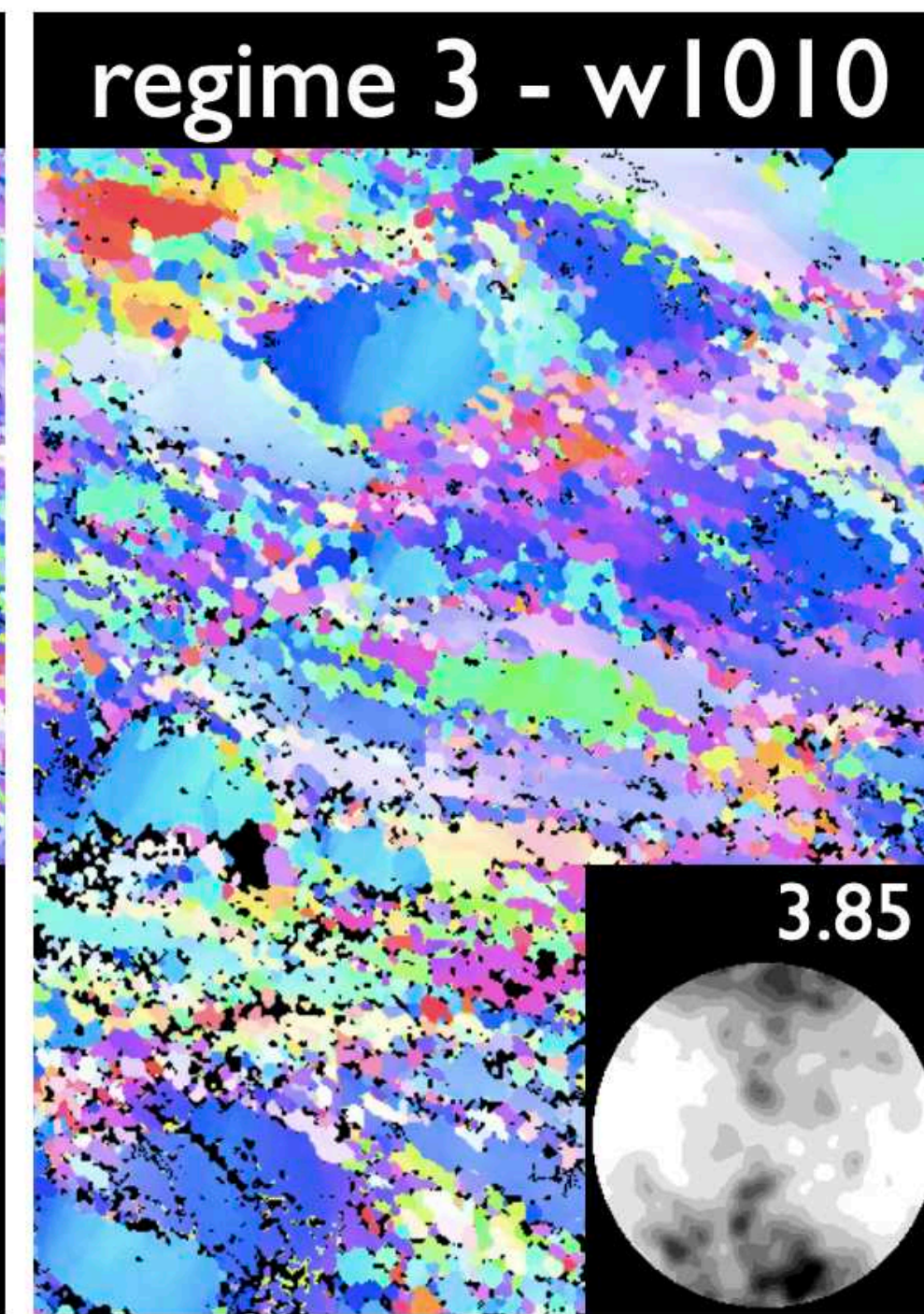
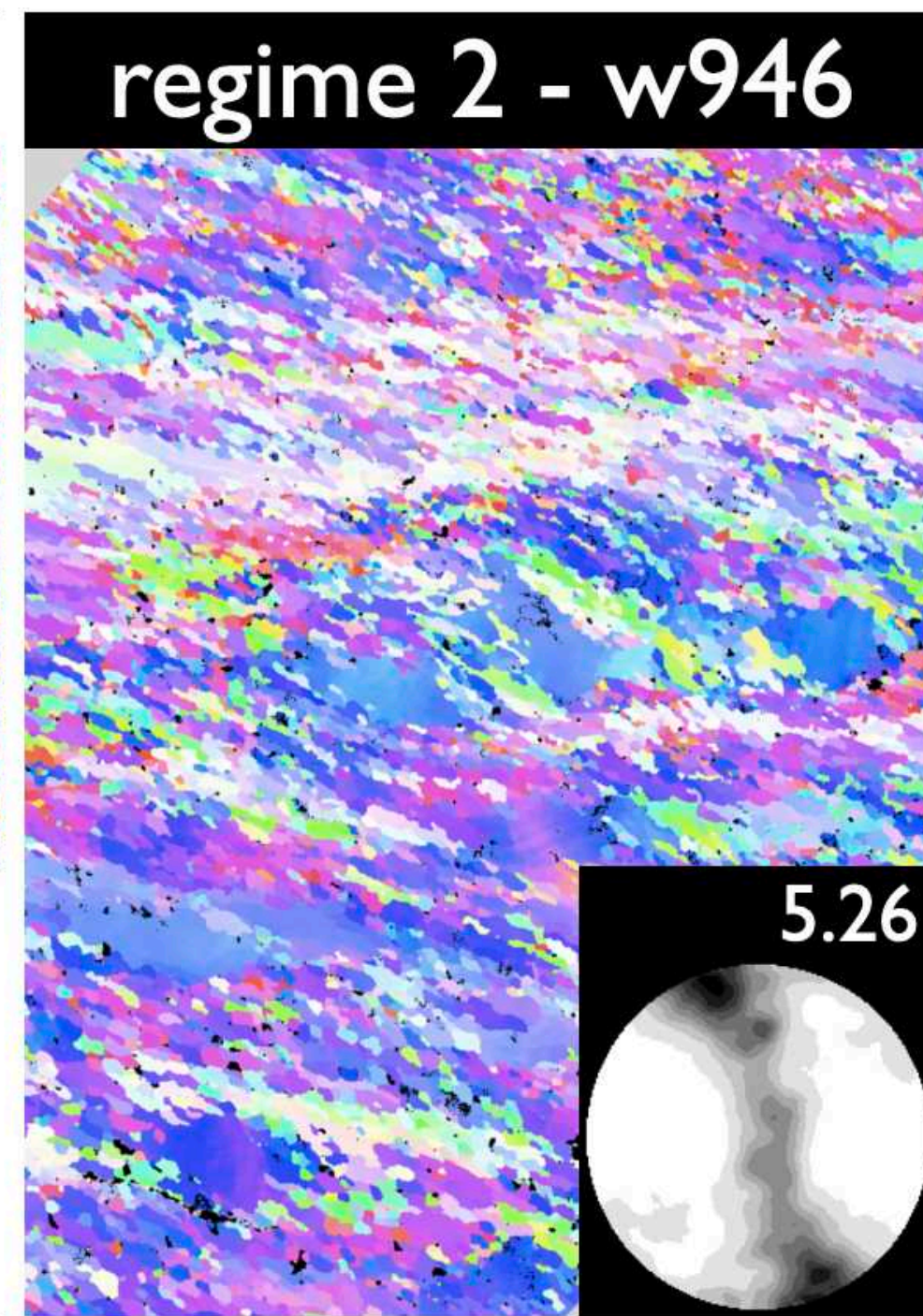
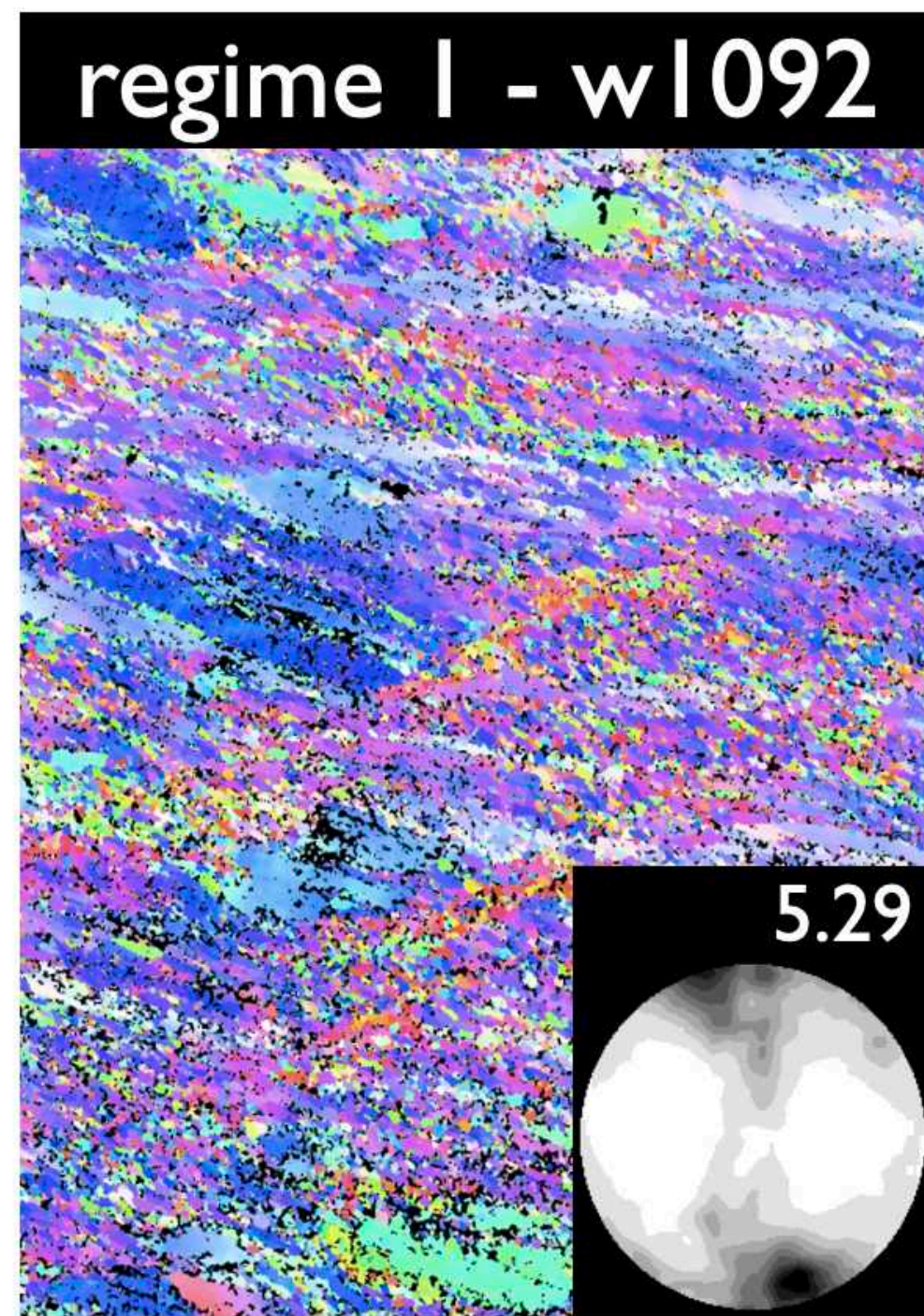
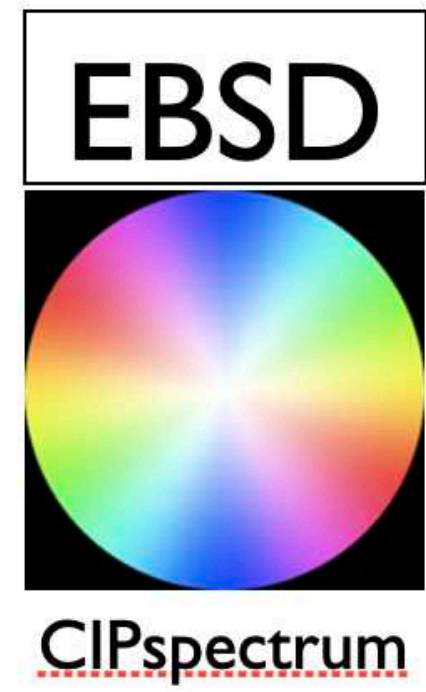


regime 3 - w965

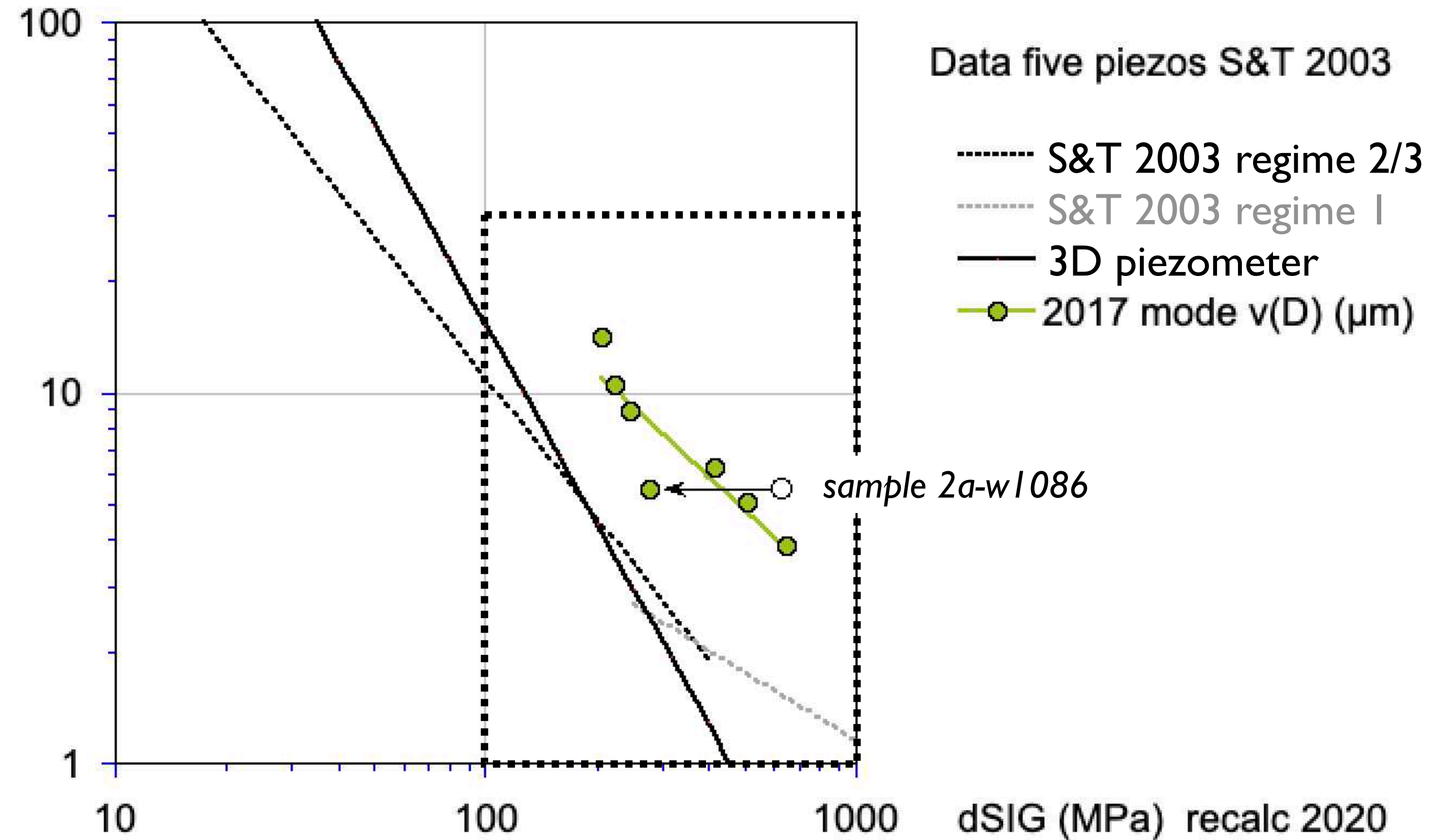
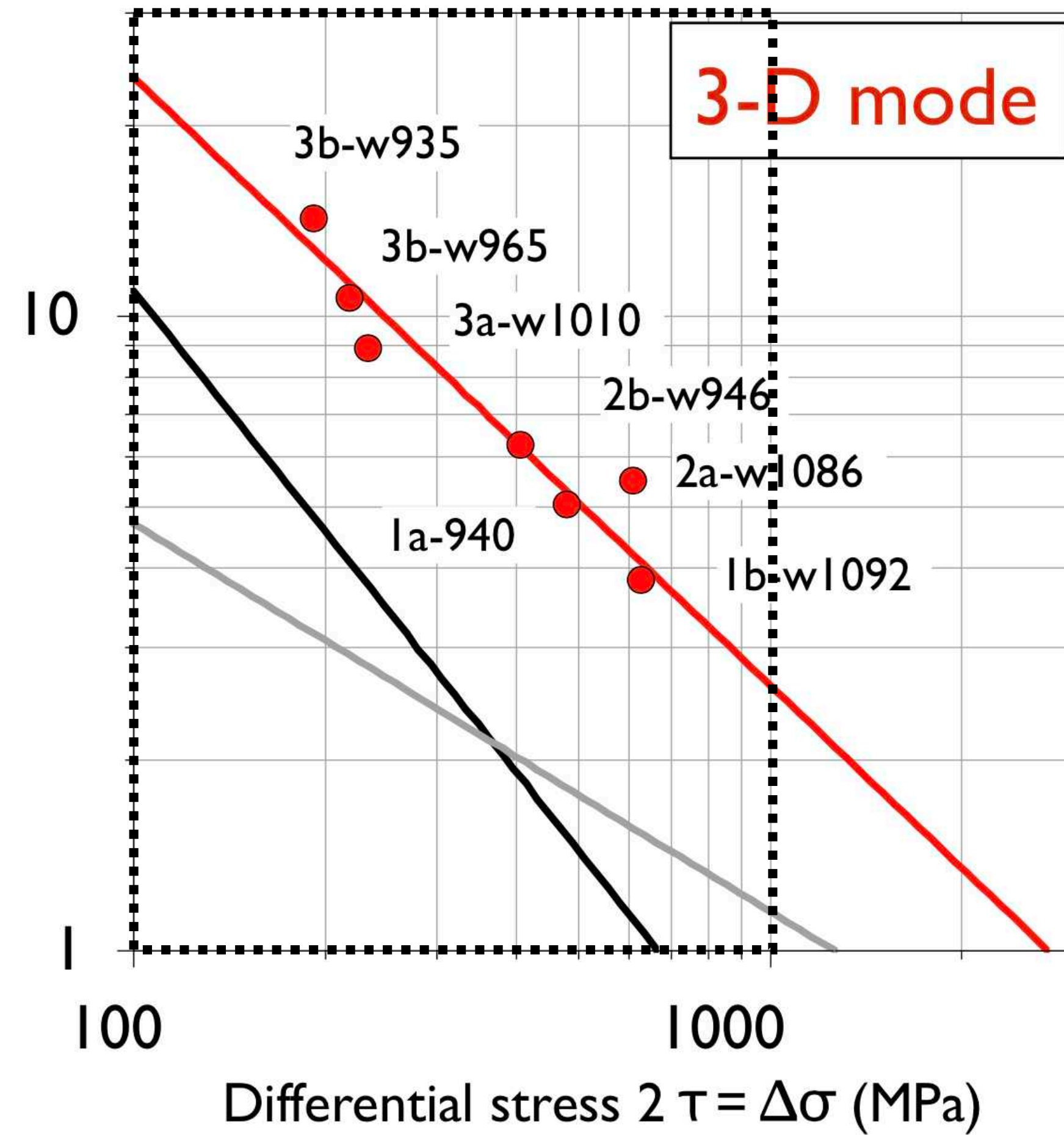




# CIP maps from EBSD



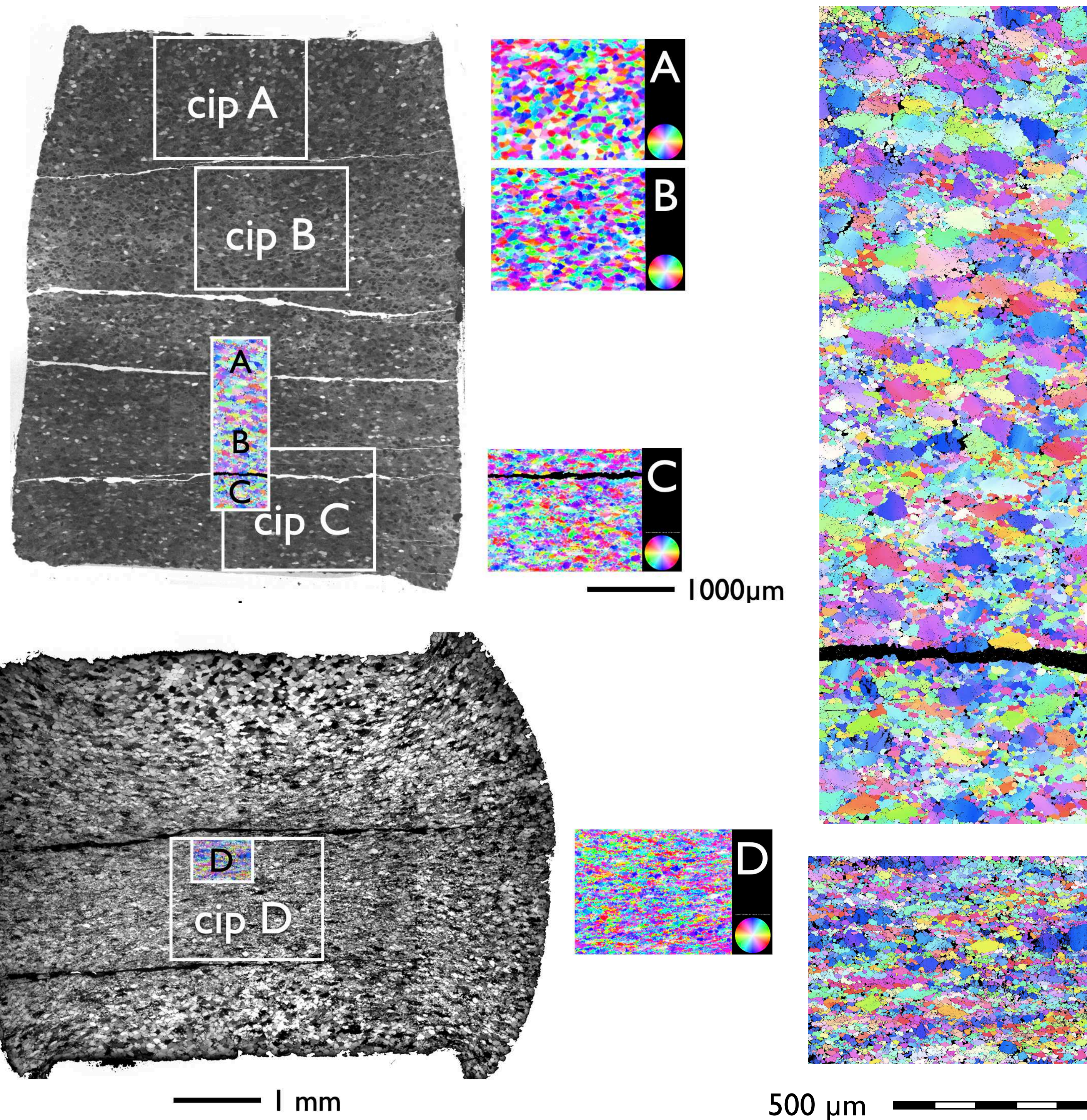
# run records redone – $\Delta\sigma$ recalculated



2020 recalculated  $\Delta\sigma$  yield similar result  
except for sample 2a-w1086 (wrong run record)

should we worry about spatial  
resolution?

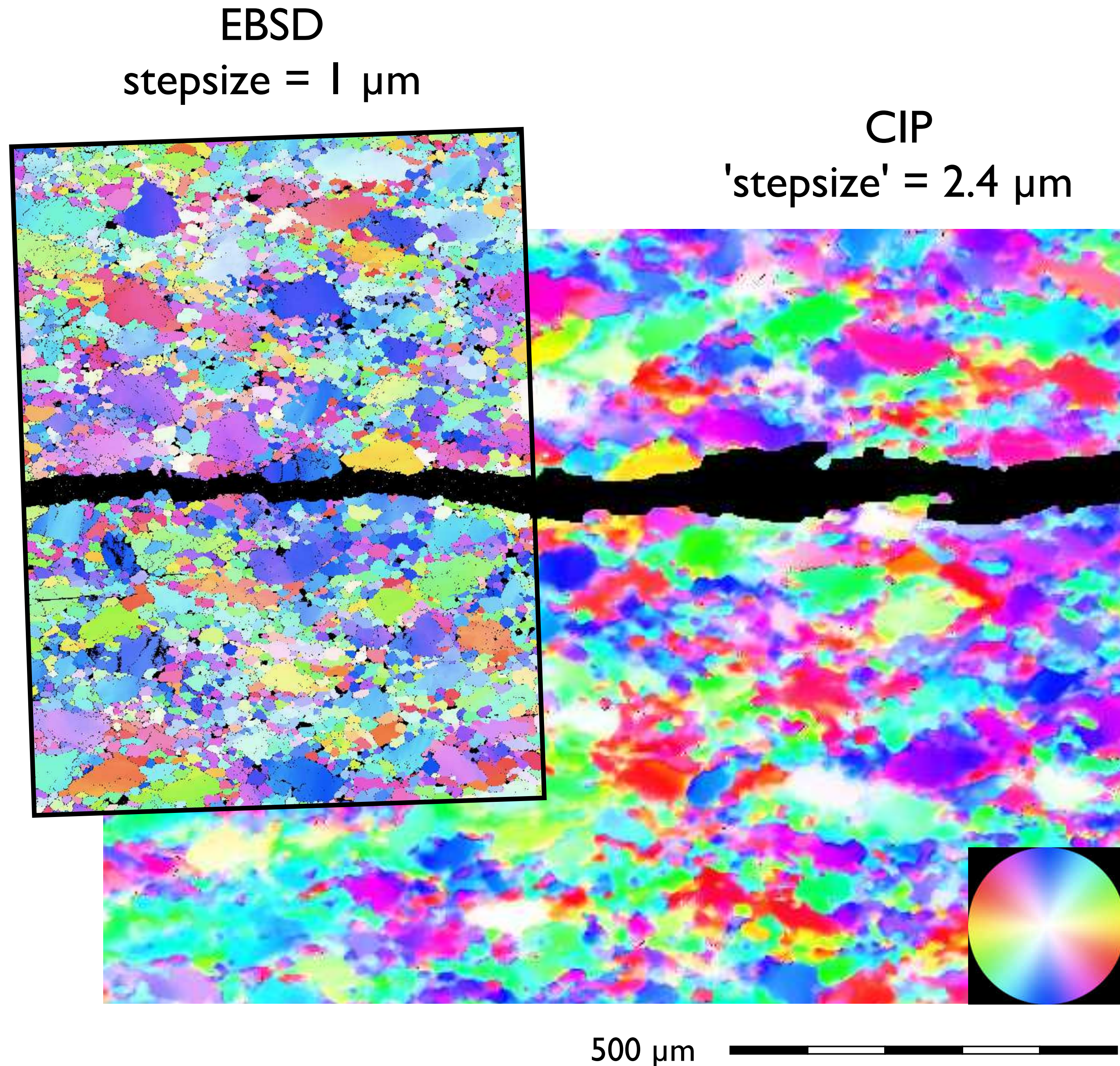
# sometimes lucky EBSD map of previous CIP



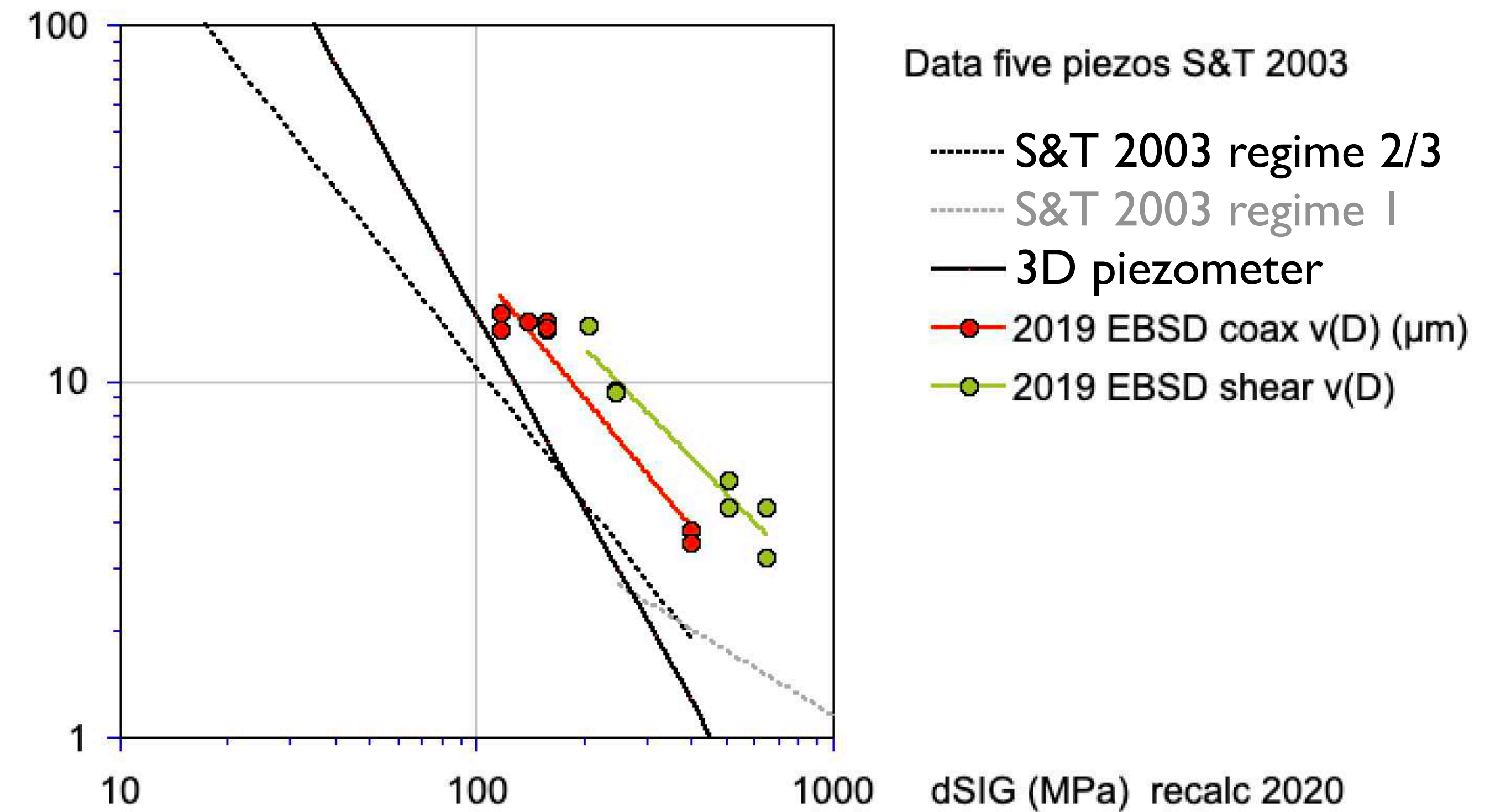
method	stepsize (µm)	3D grain size (µm) site C	3D grain size (µm) siteD
CIP	2.64	18	19
EBSD	0.5	16	15

high resolution decreases grain size  
 ⇒ steeper piezometer

# coaxial and shear at EBSD resolution



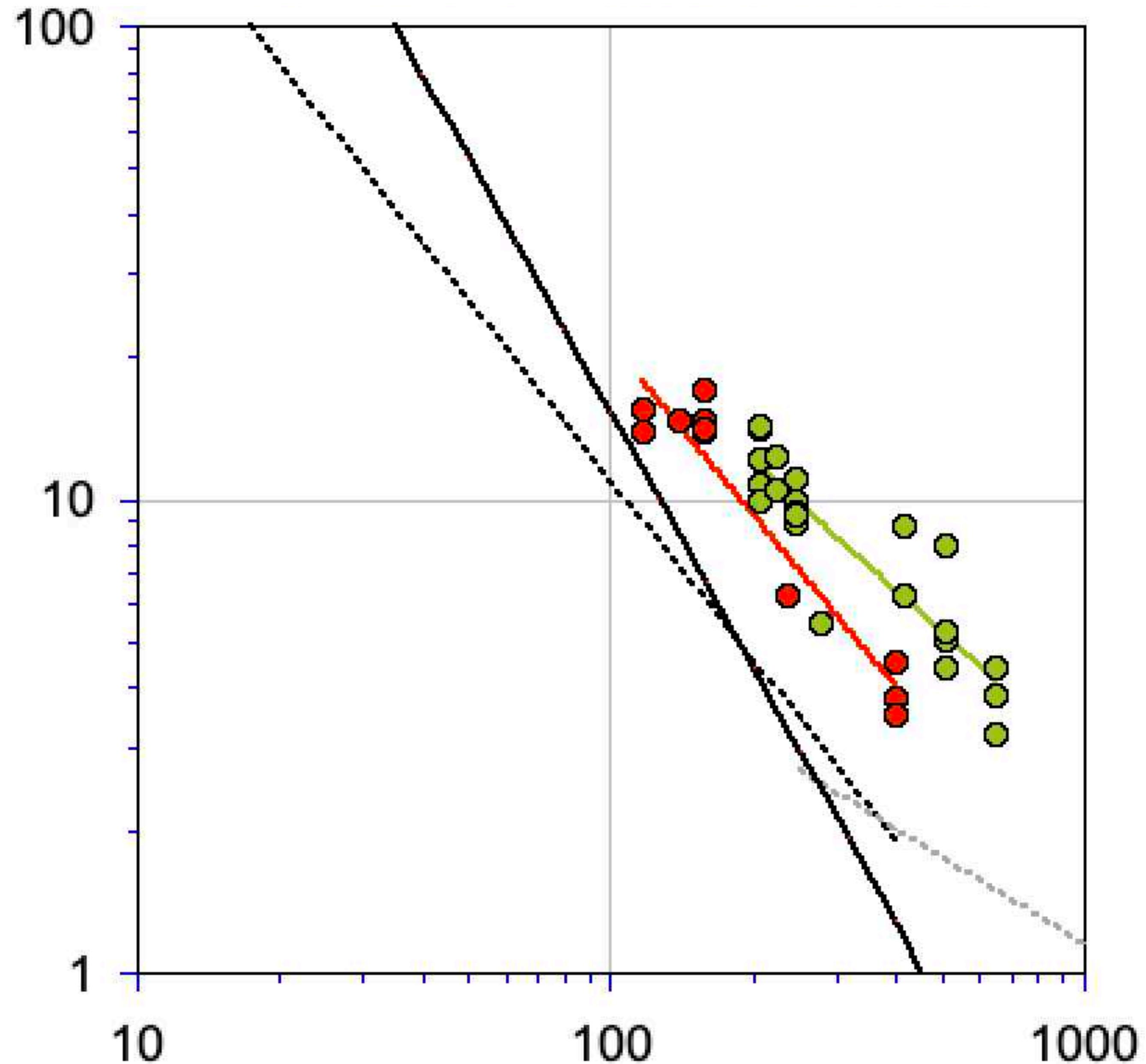
## high resolution data only



high resolution: two piezometers  
one each (coaxial and shearing) for all regimes

# putting it alltogether

all best data



$$d = 3631 \cdot \Delta\sigma^{-1.26}$$

$$d = 78 \cdot \Delta\sigma^{-0.61}$$

$$D = 58060 \cdot \Delta\sigma^{1.79}$$

$$D_{\text{axial}} = 5529 \cdot \Delta\sigma^{-1.21}$$

$$D_{\text{shear}} = 1435 \cdot \Delta\sigma^{-0.90}$$

axial (solid medium)  $\approx$  2x S&D (MSC) piezometer  
shear (solid medium)  $\approx$  2x axial piezometer

dSIG (MPa) recalc 2020

**so what does it mean, Holger ?**



... often enough I was more fascinated by the mere fact that I could quantify microstructures, shapes, distributions, correlations... and grain size, ....

... and often it was Holger who pointed out to me why any of that may actually be interesting





we tried early to get our daughter interested in rocks – no luck !



maybe a better chance with the next generation ?