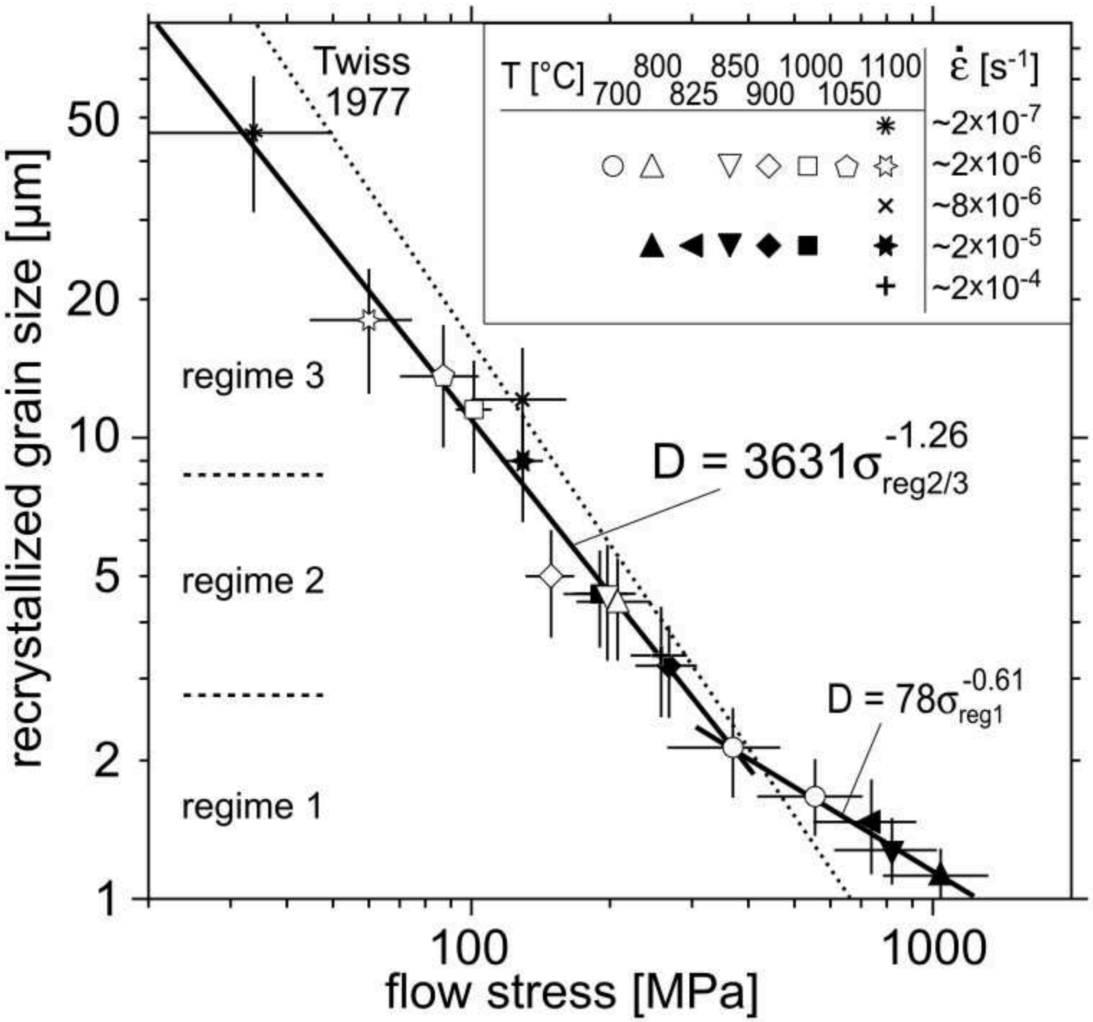
all you ever wanted to know about grain size and never dared to ask (...)

a film by Renée Heilbronner

motivation: the quartz piezometer

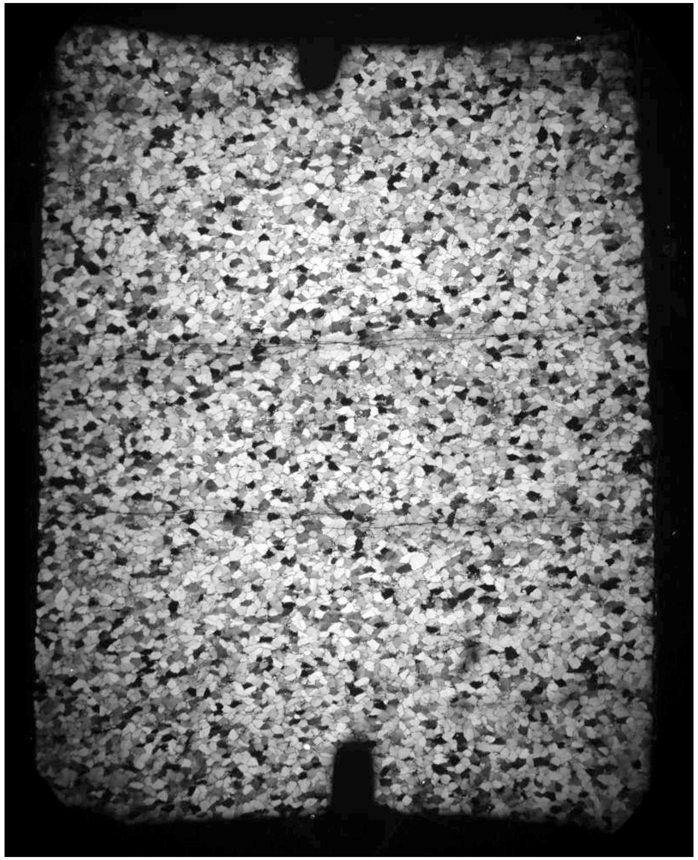


grain size as function of flow stress

experimental basis

Stipp & Tullis

(Stipp & Tullis, JGR, 2003)



coaxial

Heilbronner & Tullis

(Heilbronner & Tullis, JGR, 2006)

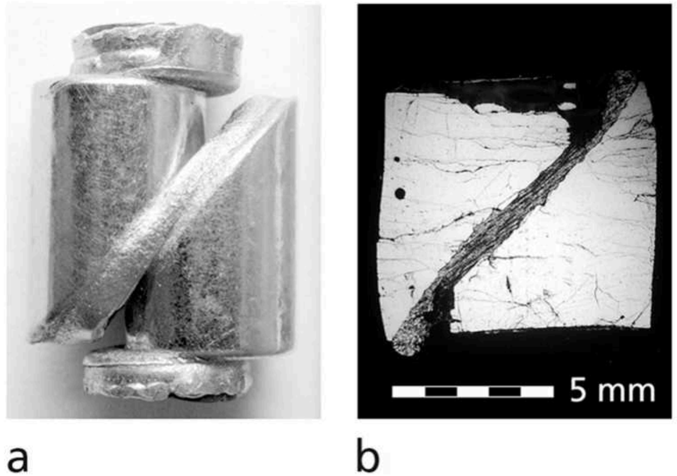
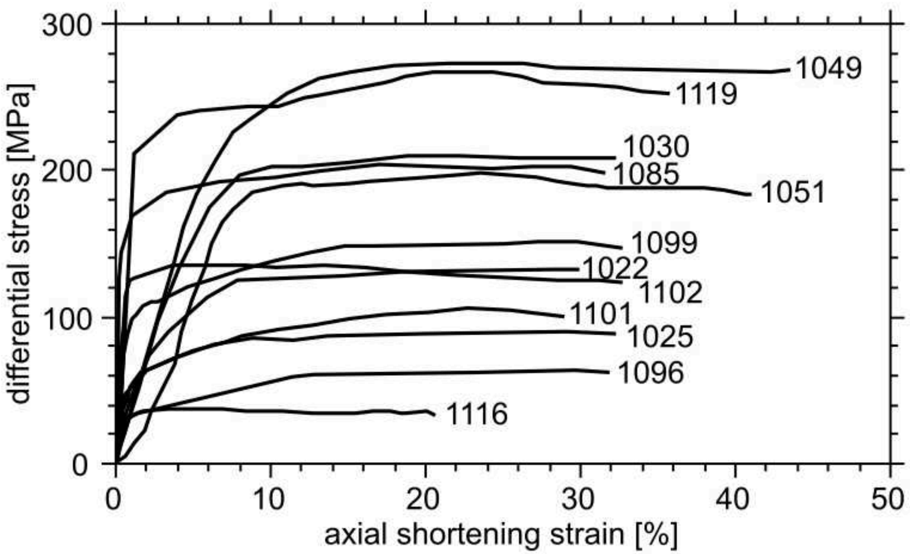
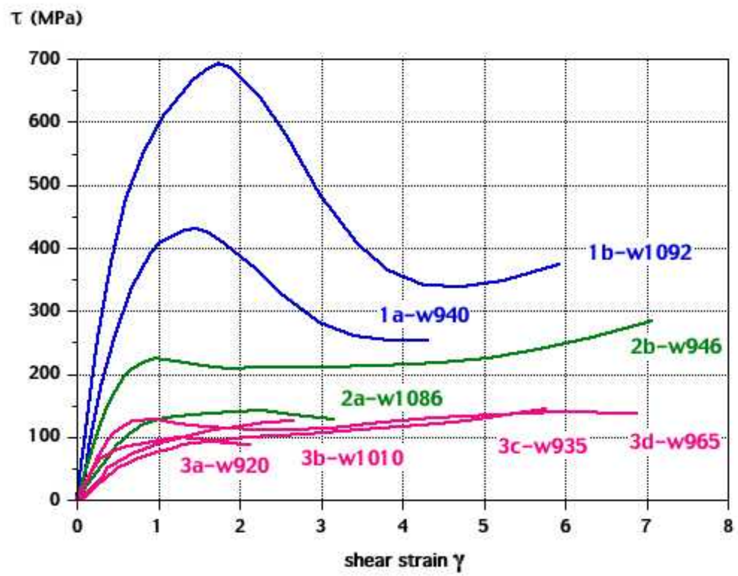


Figure 1. Geometry of experimentally sheared Black Hills quartzite samples. (a) Jacketed sample after general shear deformation: BHQ sheared between 45° precut Brazil quartz pistons (total undeformed length ≈ 15 mm, diameter = 6.3 mm), which are able to slide horizontally relative to the upper and lower ZrO_2 pistons. (b) Thin section of sheared BHQ sample and Brazil quartz pistons under circularly polarized light. Horizontal cracks in the pistons result from unloading.

shear

stress determination





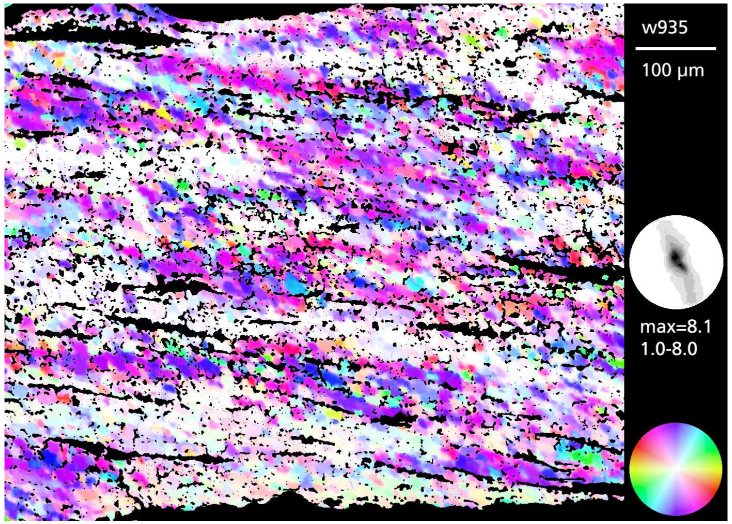
$\Delta\sigma$ versus e(%)

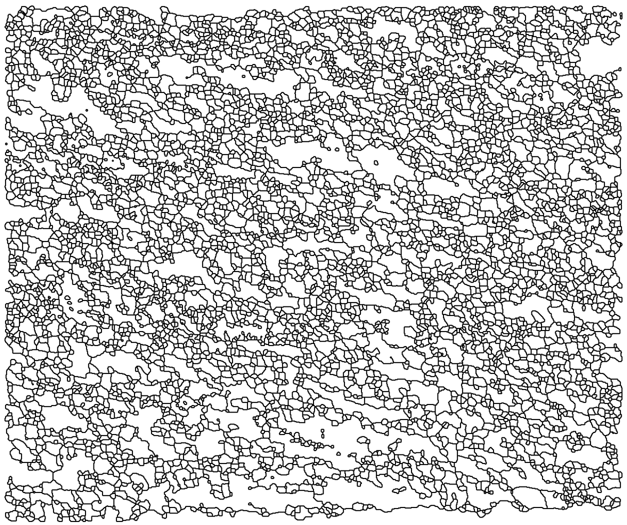
τ versus γ

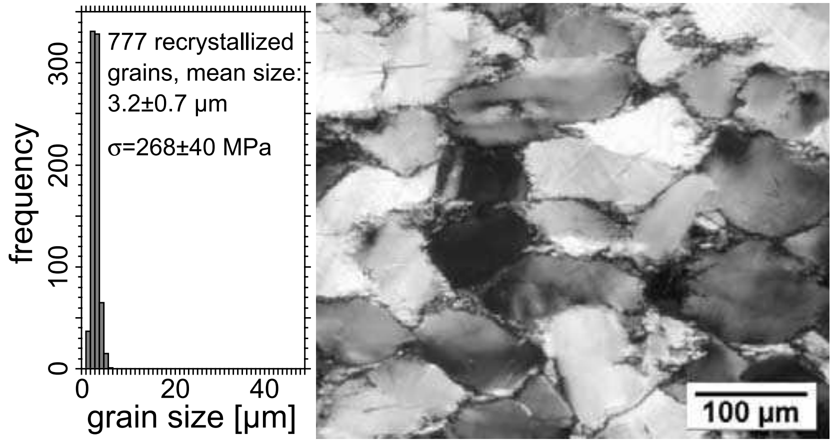
coaxial

shear

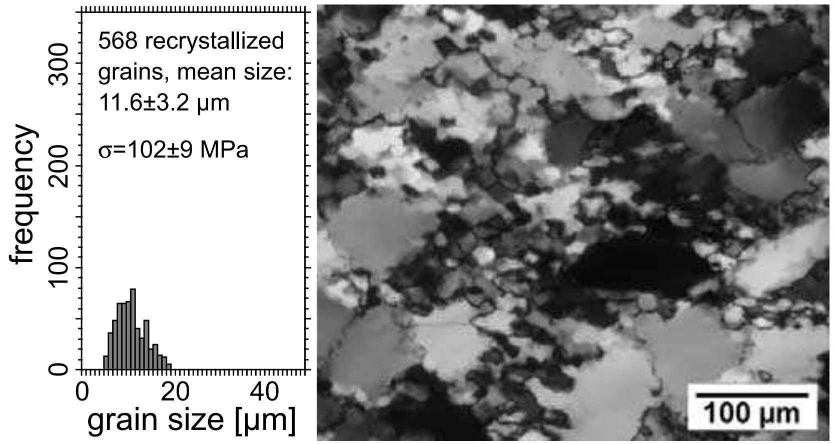
grain size determination







a) 900°C, ~ 2×10^{-5} s^{-1}, ε = 44%



b) 1000°C, ~ 2×10^{-6} s^{-1}, ε = 22%

(Stipp & Tullis, JGR, 2003)

what is the 'mean' grain size ?

RMS of $h(d_{circles})$

The recrystallized grain size piezometer for quartz

Michael Stipp and Jan Tullis
Department of Geological Sciences, Brown University, Providence, Rhode Island, USA

"Recrystallized grains were distinguished from porphyroclasts manually and on the basis of the bimodal grain size distribution which occurs in all samples except W-1066 and W-1126. The diameter of each recrystallized grain is defined as the diameter of a circle with the same area, and the average 2-dimensional recrystallized grain size for each sample was calculated as the root mean square diameter from all measured recrystallized grains in that sample"

what is the 'mean' grain size ?

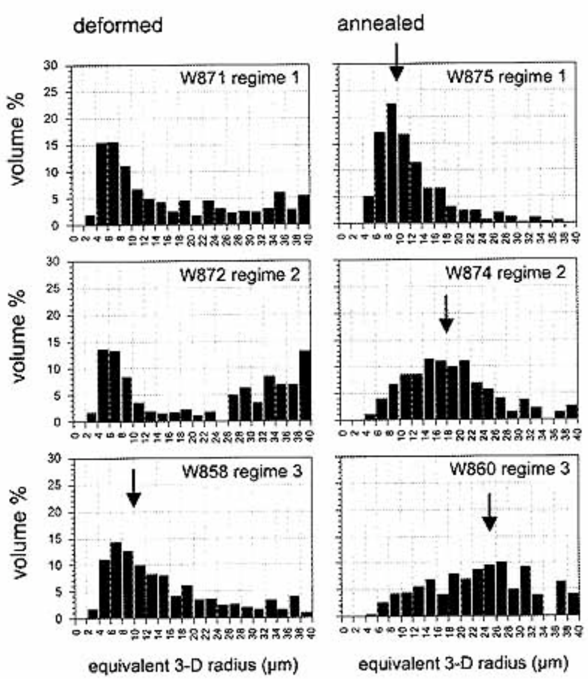
The effect of static annealing on microstructures and crystallographic preferred orientations of quartzites experimentally deformed in axial compression and shear

RENÉE HEILBRONNER[1] & JAN TULLIS[2]

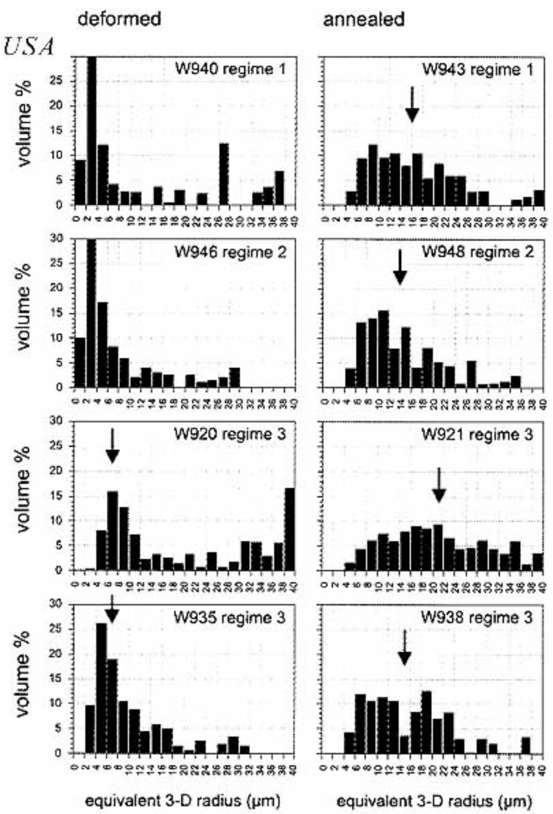
[1] *Department of Earth Sciences, Basel University, Bernoullistrasse 32, CH-4056 Basel, Switzerland*
(e-mail: Renee.Heilbronner@unibas.ch)
[2] *Department of Geological Sciences, Brown University, Providence RI 02912, USA*

mode of vol%($R_{spheres}$)

(need 2D-3D conversion)



5. Grain size distributions of axially deformed samples, before annealing (left column) and after annealing [right] column), plotted as volume % versus radius of equivalent sphere. 2D grain boundary maps were [prepa]red from misorientation images (magnification ×5 for deformed samples, ×5 for annealed samples): from the distributions of cross sectional areas were determined: from these the 3D grain size distributions were [calcu]lated. Note: the maximum radius included is 40 μm, corresponding to the largest remaining porphyroclast. [Arro]ws indicate mode of recrystallized grain size.

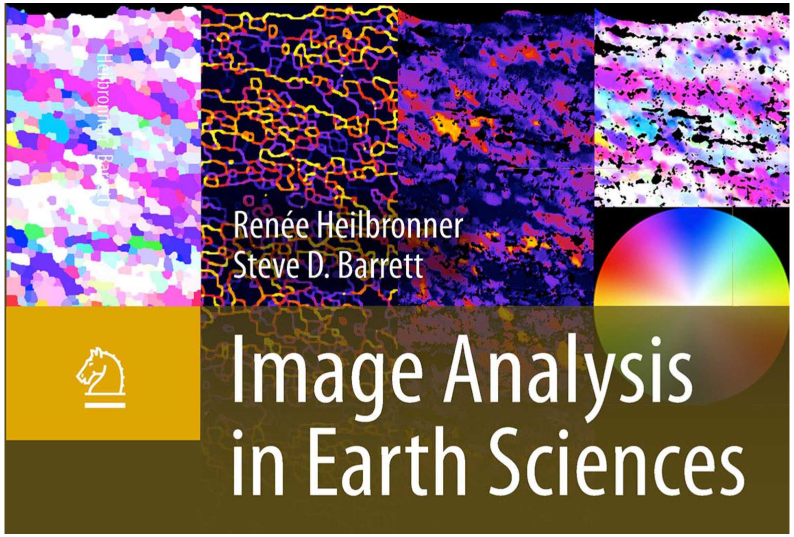


6. Grain size distributions of samples deformed by shearing, before annealing (left column) and after [ann]ealing (right column), plotted as volume % versus radius of equivalent sphere. 2D grain boundary maps [wer]e prepared from misorientation images (magnification ×10 for deformed samples, ×5 for annealed [sam]ples). For explanation see Figure 5. Arrows indicate mode of recrystallized grain size.

... which reminds me ...

for segmentation,
for 2D-3D conversion,
... and many other useful techniques...

see: Heilbronner & Barrett, Springer (2014)



copies still available at reduced rate at Margrete's office

why go back ?

re-measure CIP grain size using EBSD:
(see if CIP measurements are OK, especially fine-grained)

think about grain size

and then:

1. check Stipp & Tullis piezometer using EBSD
2. check if piezometer is indeed different for different regimes
3. check if piezometer is same for coaxial and shear
4. check if piezometer is texture dependent

EBSD

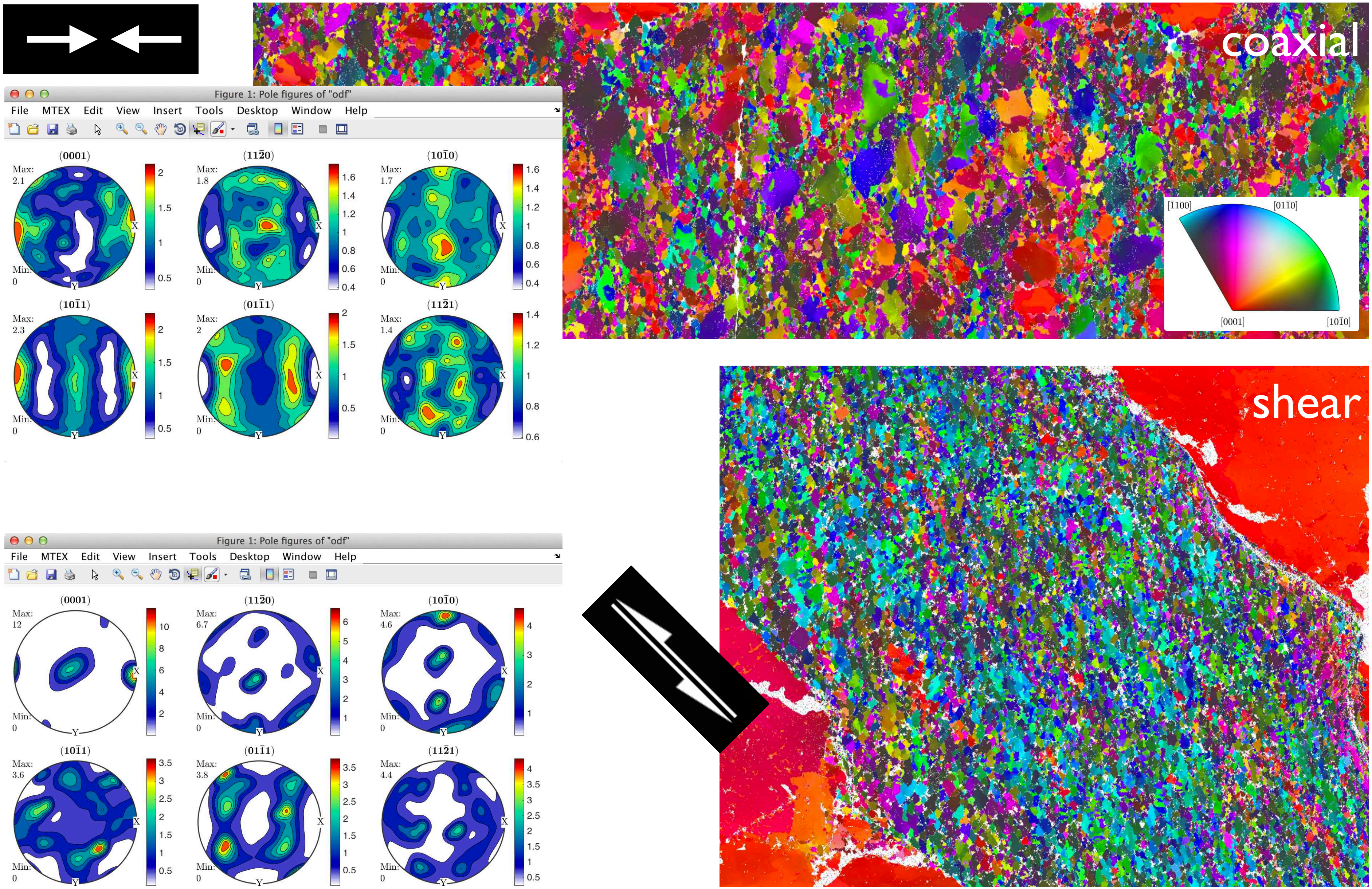
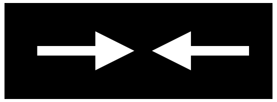
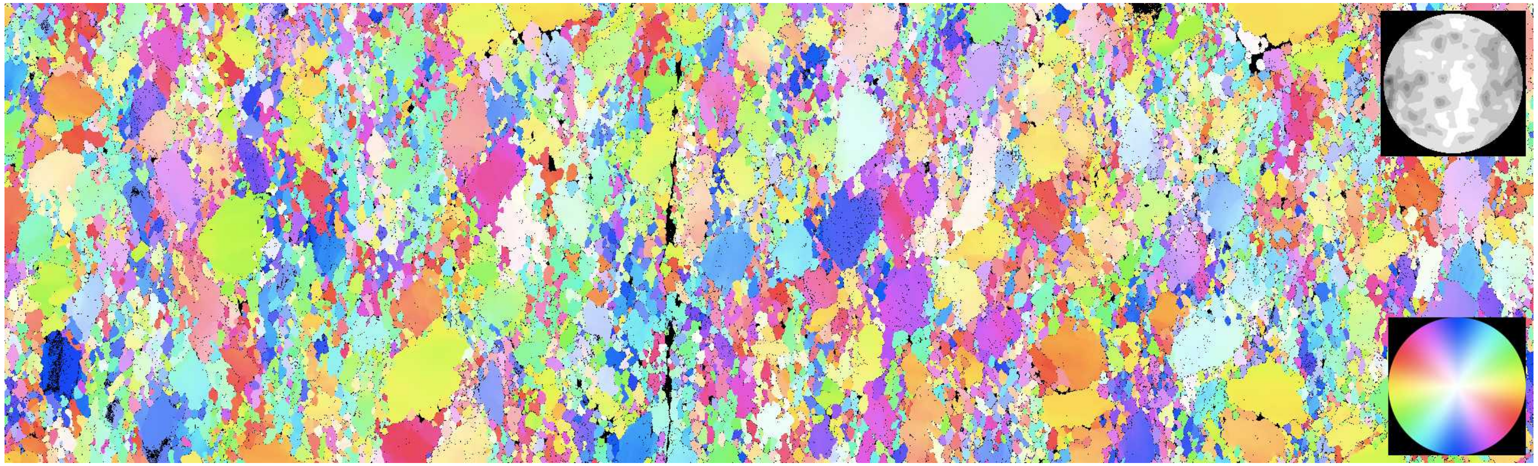


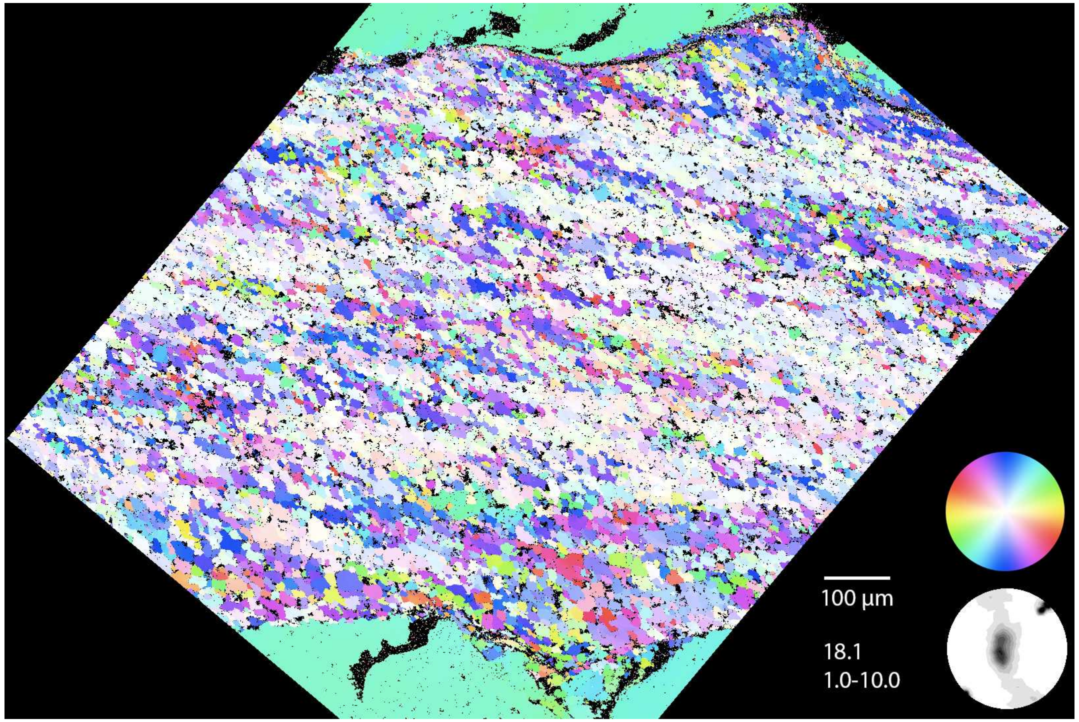
Figure 1: Pole figures of "odf"
File MTEX Edit View Insert Tools Desktop Window Help
(0001)
(11$\bar{2}$0)
(10$\bar{1}$0)
(10$\bar{1}$1)
(01$\bar{1}$1)
(11$\bar{2}$1)
coaxial
[$\bar{1}$100]
[01$\bar{1}$0]
[0001]
[10$\bar{1}$0]
Figure 1: Pole figures of "odf"
File MTEX Edit View Insert Tools Desktop Window Help
(0001)
(11$\bar{2}$0)
(10$\bar{1}$0)
(10$\bar{1}$1)
(01$\bar{1}$1)
(11$\bar{2}$1)
shear

convert to CIP



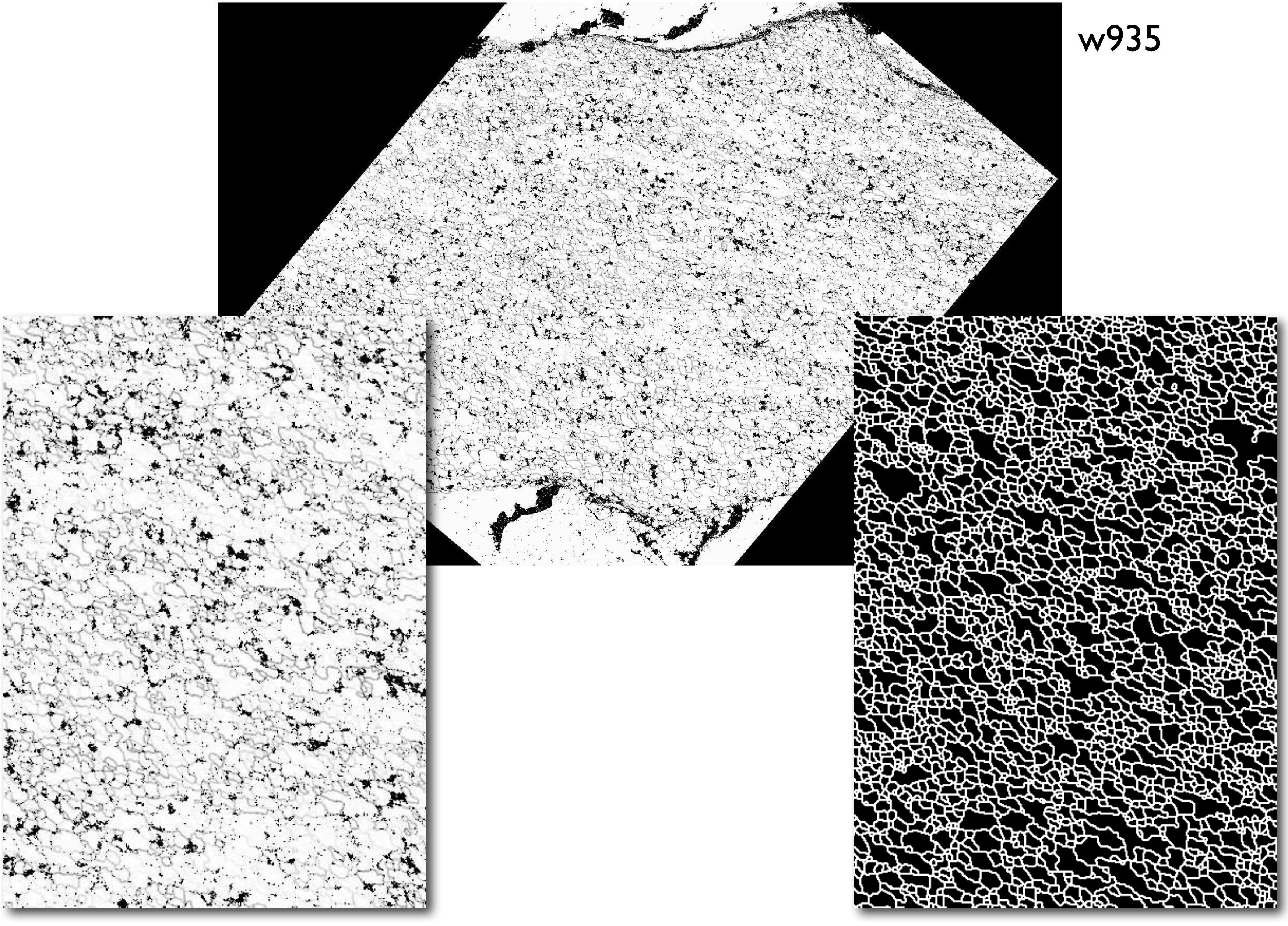


w1029



w935

segmentation



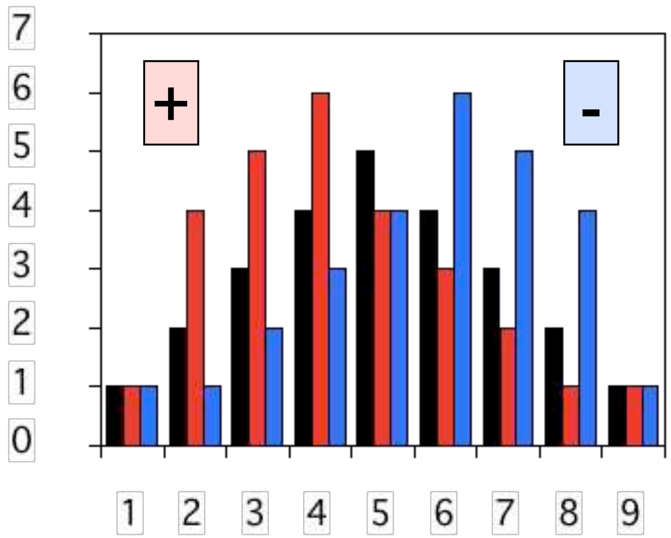
w935

finding the right mean...

arithmetic mean $\bar{X} = 1/n \cdot \sum x_i$

root-mean-square $RMS = \sqrt{(1/n \cdot \sum x_i^2)} \approx$ area average

Mode = most frequent value



	symm.	+ skew	- skew
Mean	5.00	4.33	5.67
Mode	5.00	4.00	6.00
RMS	5.39	4.75	5.99
Skewness	0.00	0.53	-0.53
RMS/$\bar{X}$	**108%**	**110%**	**106%**

$RMS > \bar{X}$

finding the right mode:
for noisy data, use empirical relationship:
difference (Mean - Mode) = 3 · difference (Mean - Median)

Mean of grouped data

$$\bar{x} = \frac{\sum f\,x}{\sum f}.$$

Mode of grouped data (not noisy)

IV Mode

For frequency distribution, it is the value of the variable corresponding to the maximum frequency.

For example consider the frequency distribution as :

x :	20	25	30	35	40
f :	17	19	27	20	5

Here the maximum frequency is 27 and the corresponding value of the variable is 30. So mode is 30.

For grouped data with class,

$$\text{Mode} = L + \frac{h(f_1 - f_0)}{2f_1 - f_0 - f_2} \qquad \ldots(vii)$$

where L = lower limit of the class containing the mode

h = width of the modal class

f_1 = frequency of the model class

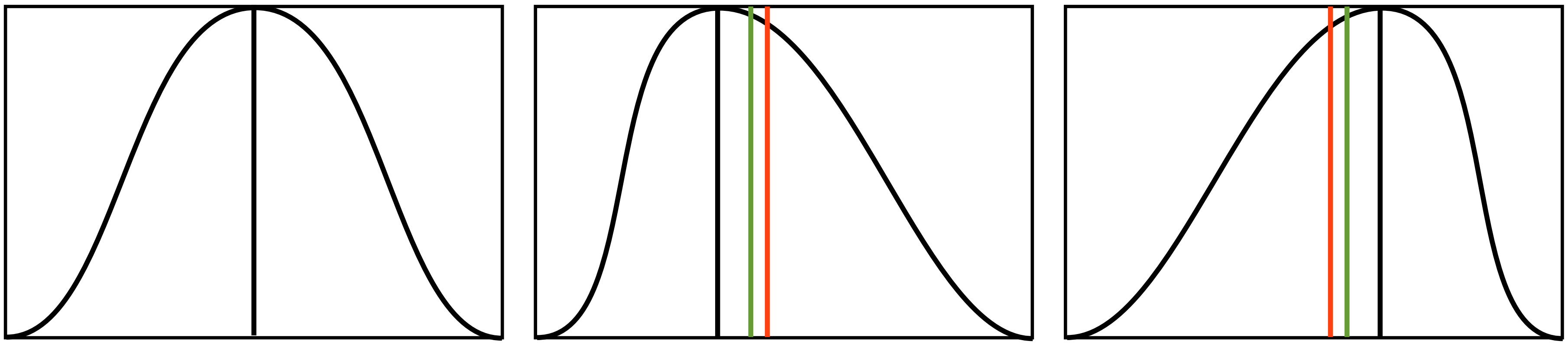
f_0 = frequency of the preceding of the modal class

f_2 = frequency of the succeeding of the modal class

In some situation, $2f_1 - f_0 - f_2 = 0$, in such a case, the value of the mode can be taken as

$$\text{Mode} = L + \frac{h(f_1 - f_0)}{|f_1 - f_0| + |f_1 - f_2|}$$

Mean - Median - Mode (modal value)



Empirical relationship:
Difference between the Mean and Median is
~1/3 of the difference between the Mean and Mode

Mode = Mean - 3 [Mean - Median]
Mode = 3 Median - 2 Mean

Use this relation for noisy data

st.dev. and RMS of grouped data

IV. Standard deviation (s.d.)

For grouped data, if x_i = class mark, $N = \Sigma f_i$ then

$$\text{s.d.} = \sigma = \sqrt{\frac{\sum f_i (x_i - \overline{x})^2}{N}} \quad \text{or,} \quad \sqrt{\frac{\sum f_i x_i^2}{N} - (\overline{x})^2}$$

For the ungrouped data, $x_1, x_2, \ldots\ldots, x_n$,

$$\text{s.d.} = \sigma = \sqrt{\frac{\sum x_i^2}{n} - \left(\frac{\sum x_i}{n}\right)^2}$$

The square of the s.d. is known as variance. Both are independent of change of origin.

V. Root mean square deviation (rms)

$$\text{rms} = \sqrt{\frac{\sum f_i (x_i - A)^2}{N}} \quad \text{for grouped data.}$$

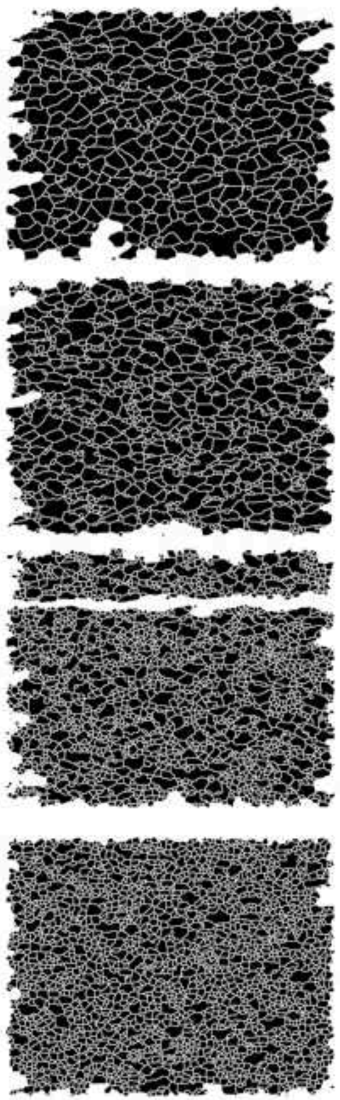
where A is any arbitrary number. But rms is least when $A = \overline{x}$.

Textbook Of Engineering Mathematics
Debashis Dutta

use 3D mode(s) (= my mission on earth...)

CQ87 regime 3 coaxial

increasing recrystallization

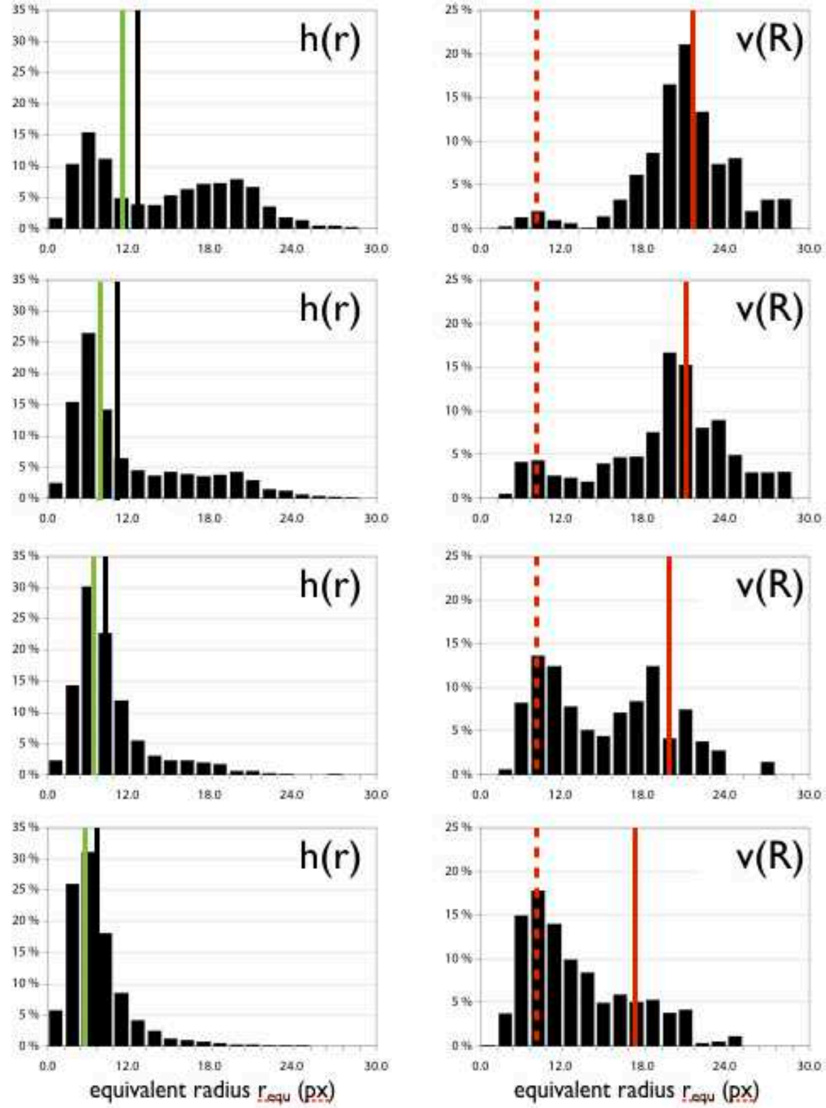


mean r_{equ} = 10.5px
RMS = 12.4px
RMS/mean = 118%

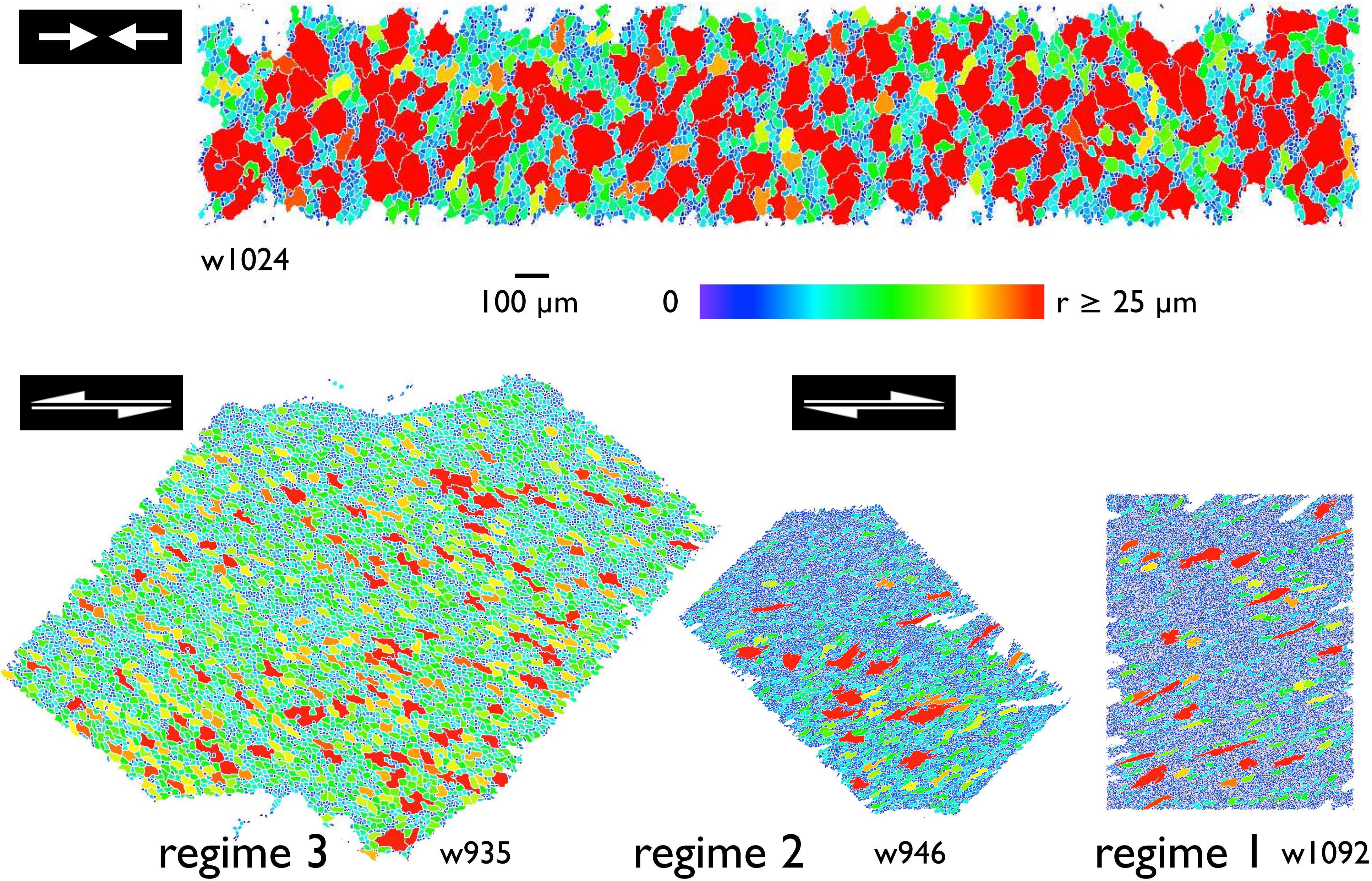
mean r_{equ} = 7.6px
RMS = 9.5px
RMS/mean = 125%

mean r_{equ} = 5.7px
RMS = 6.7px
RMS/mean = 118%

mean r_{equ} = 4.5px
RMS = 5.2px
RMS/mean = 117%

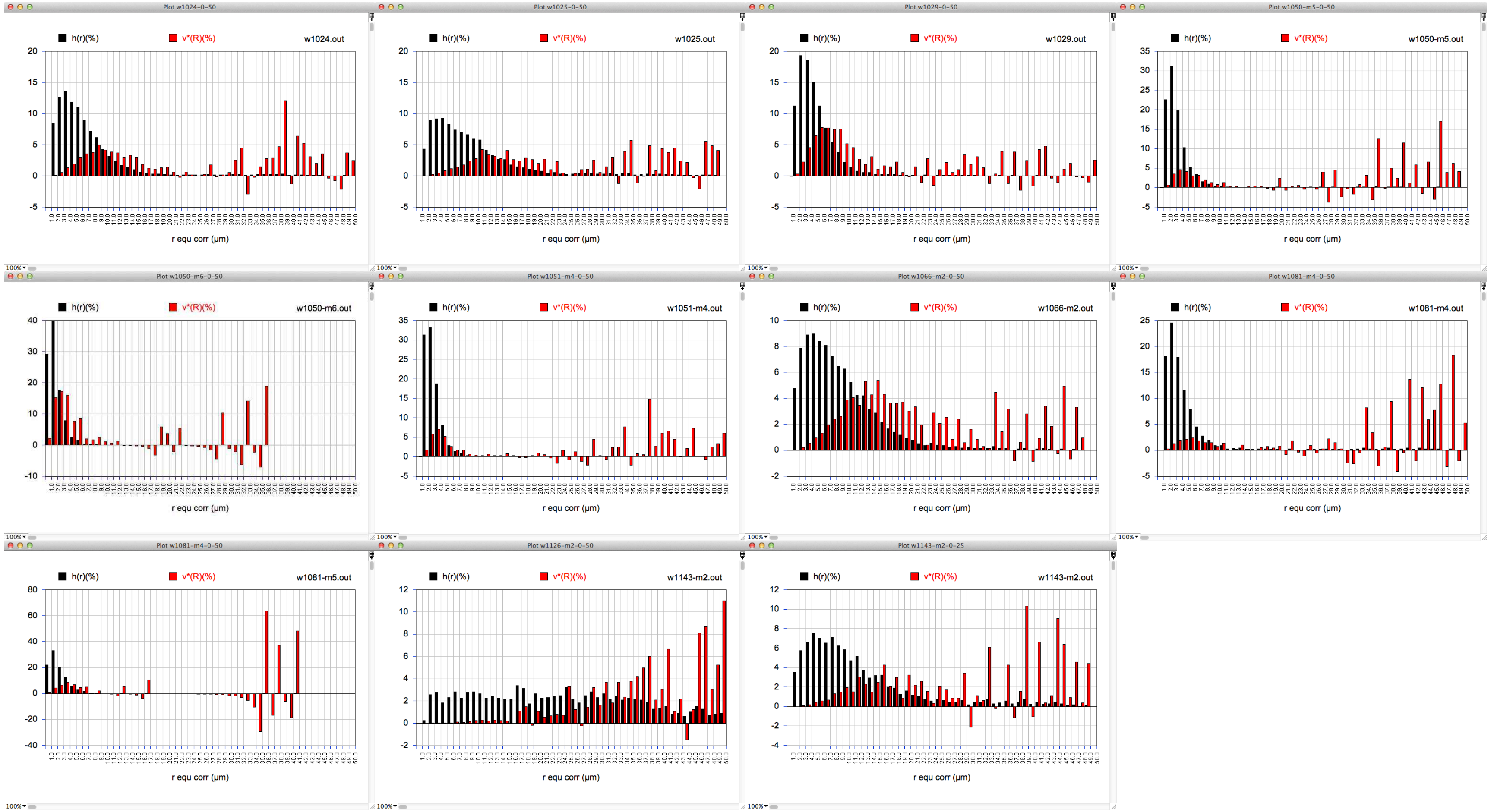


grain size mapping

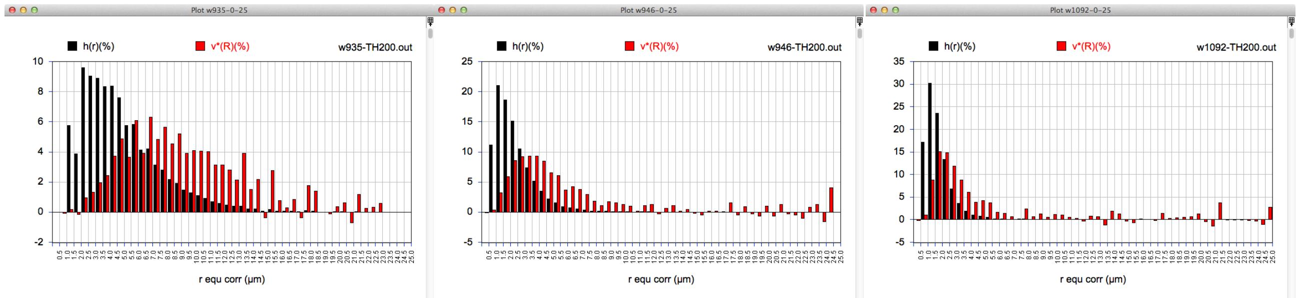


2D to 3D

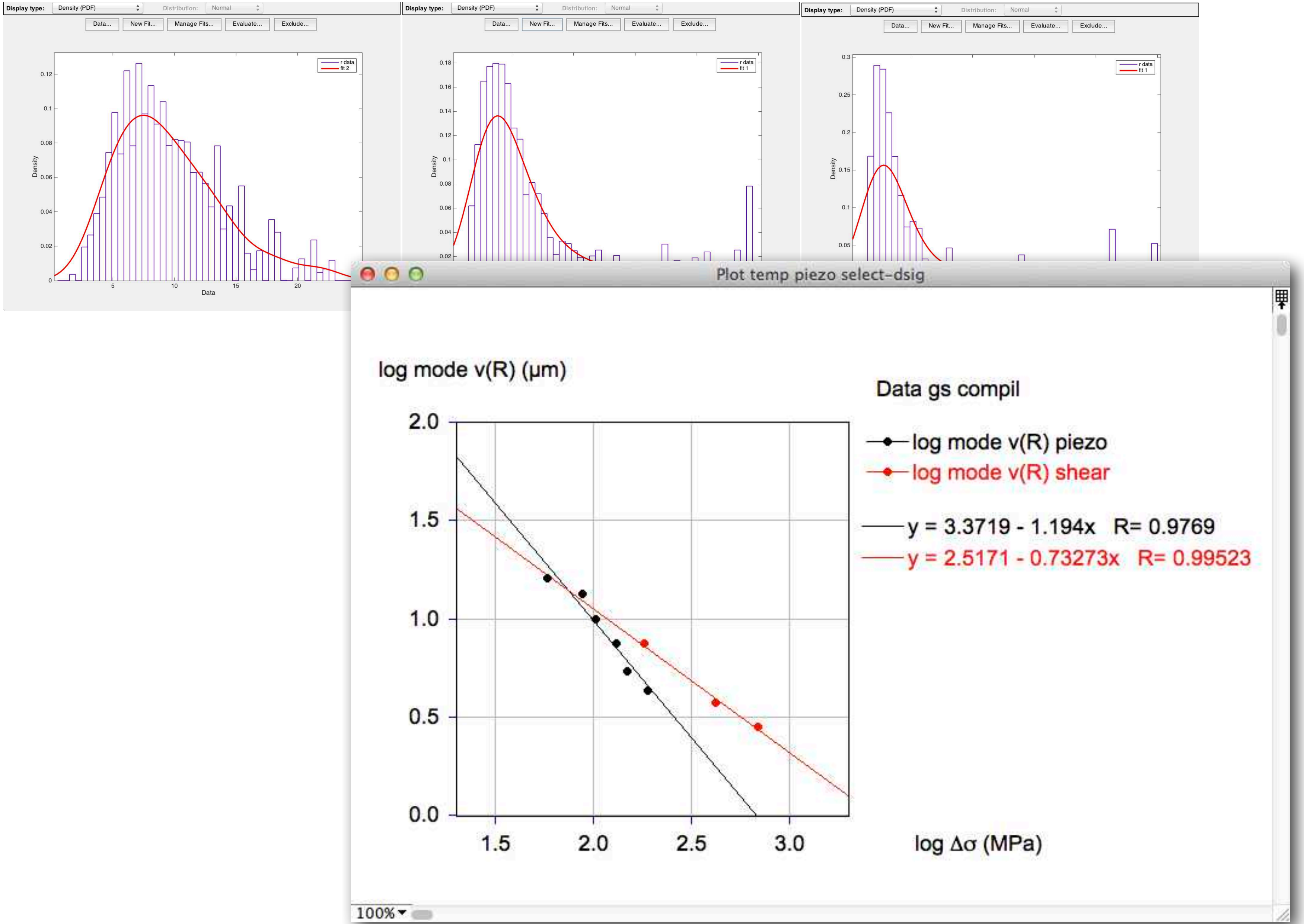
coaxial



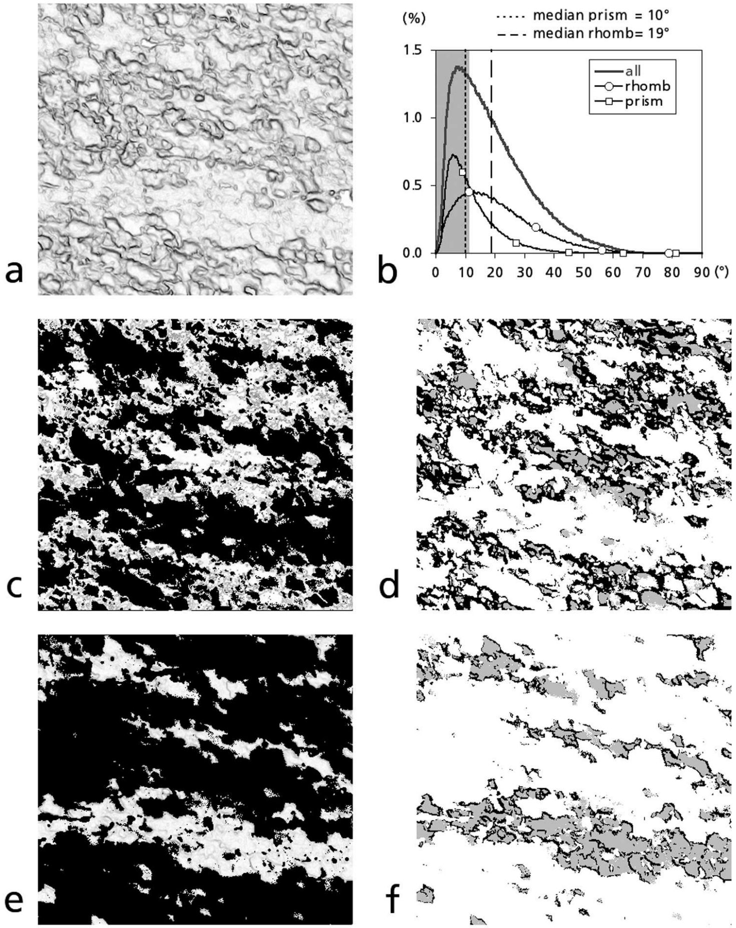
shear

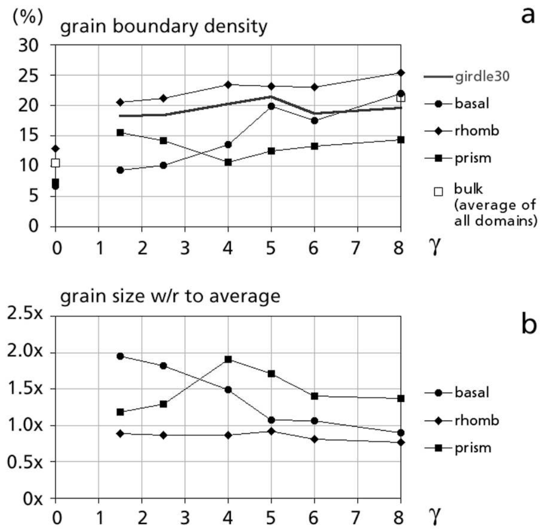


finding the right modes ... and plot!



are grains of the Y domain larger ?





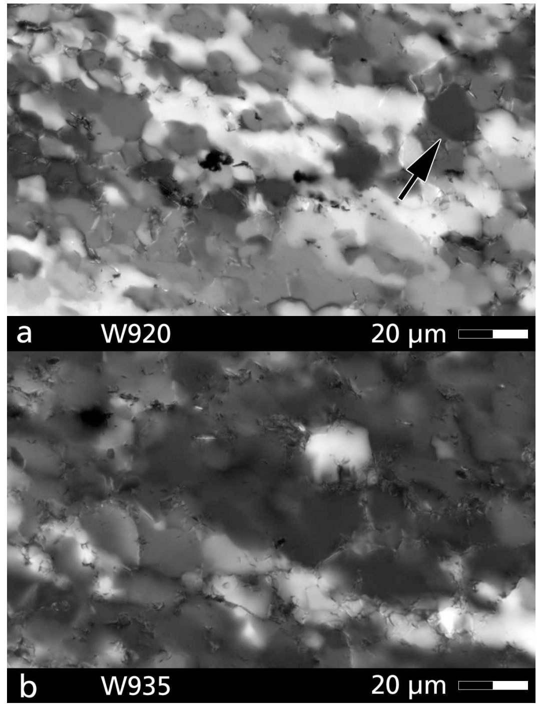
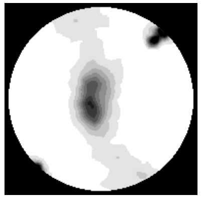
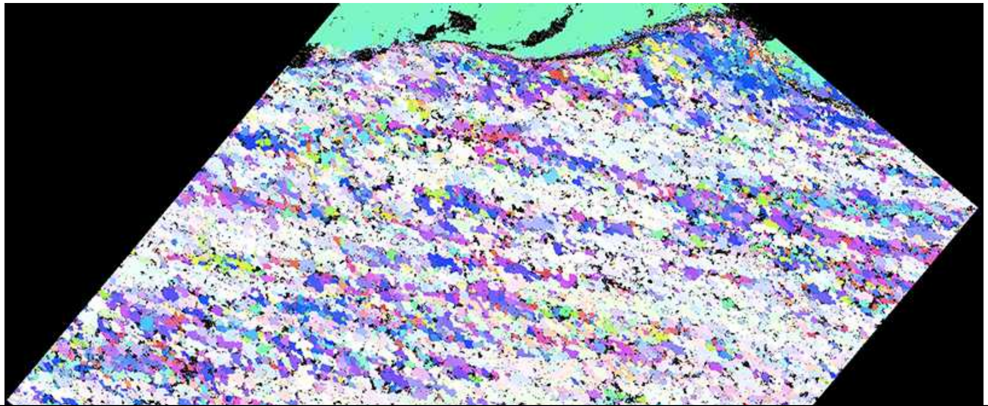


Figure 13. Optical micrographs (using circular polarization) illustrating the difference in recrystallized grain size between the prism, the rhomb, and other domains. Details of samples with low and high volume percent recrystallization are shown. (a) W920 with $\gamma \sim 1.5$. (b) Prism domain of W935 with $\gamma \sim 6$. Grains of the prism domain appear black; grains of the rhomb domain are gray and grains of the basal domain light. Note larger size of prism grains (arrow in Figure 13a) compared to grains of other orientations.

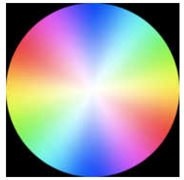
(Figure 10a), the recrystallized grain size of the rhomb domain is approximately 12 μm and that of the prism domain is approximately 19 μm, corresponding to shear stresses of 93 and 64 MPa, respectively. The gradual

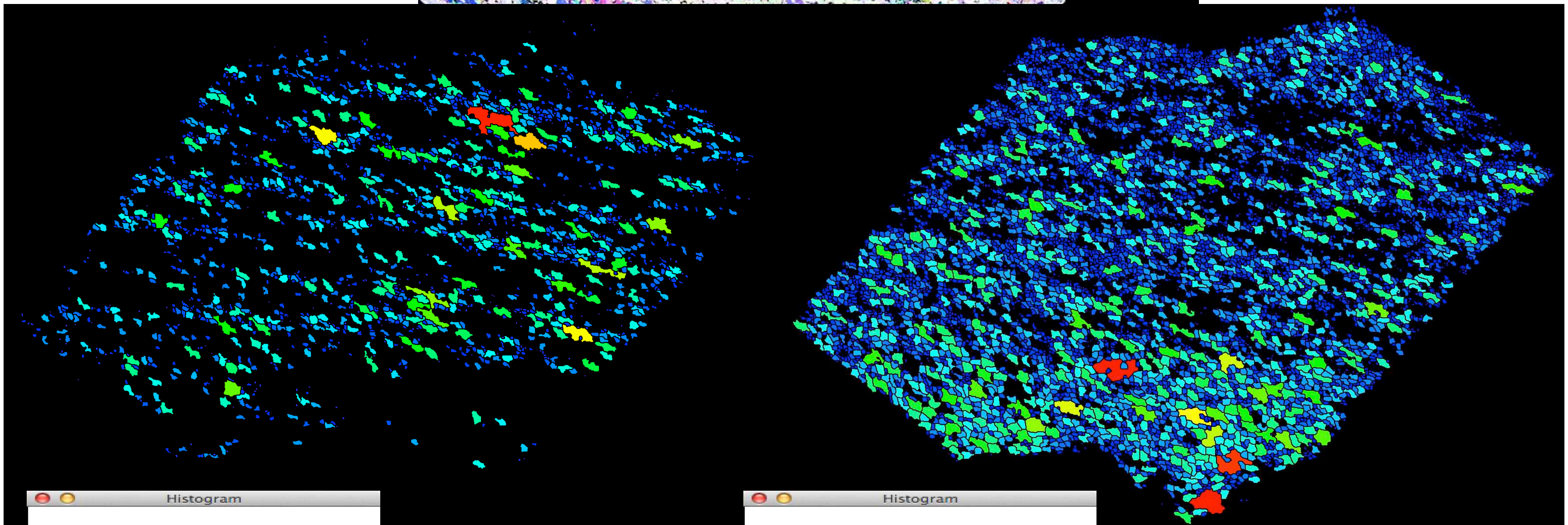
texture domain

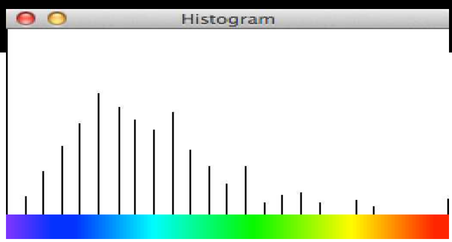




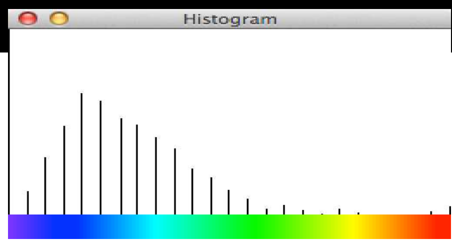
COI







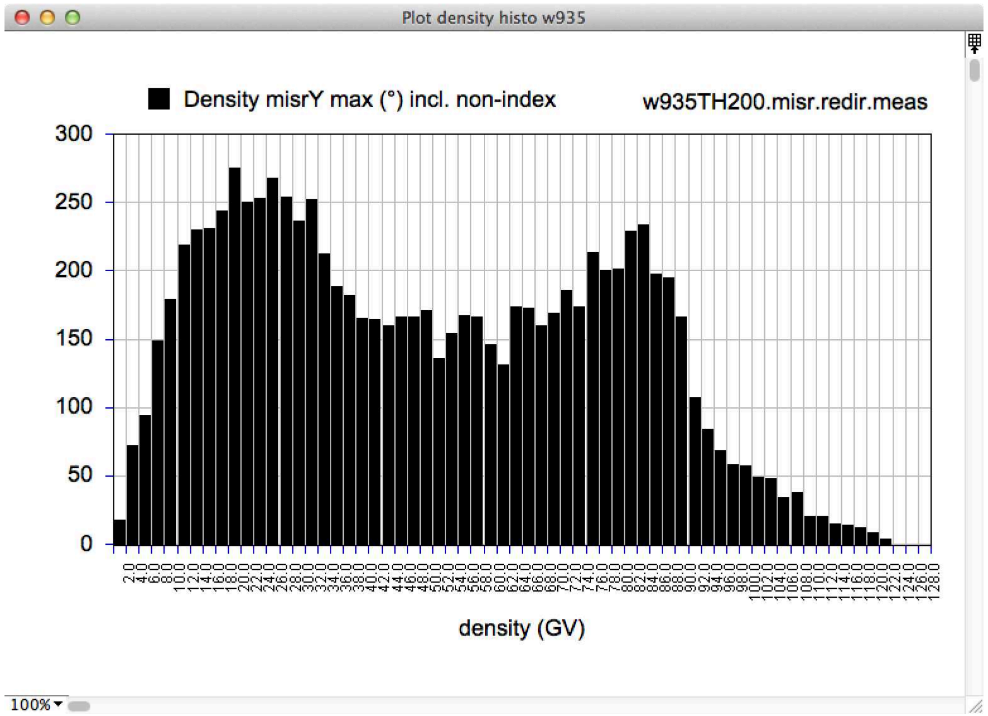
Y-domaine

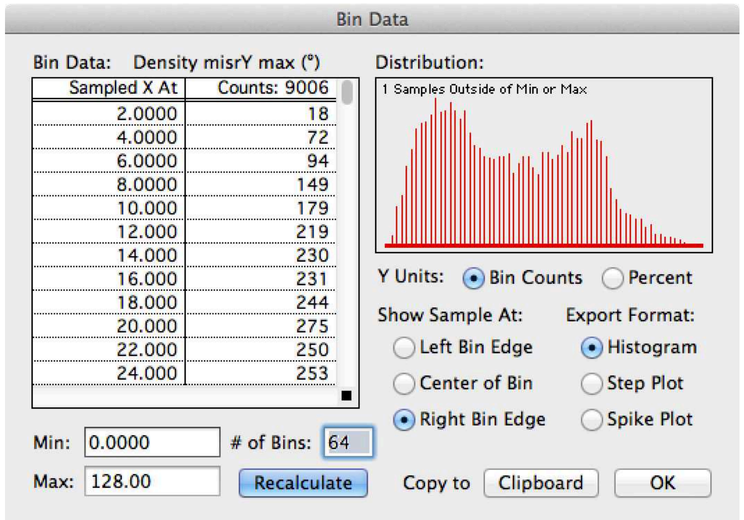


all others

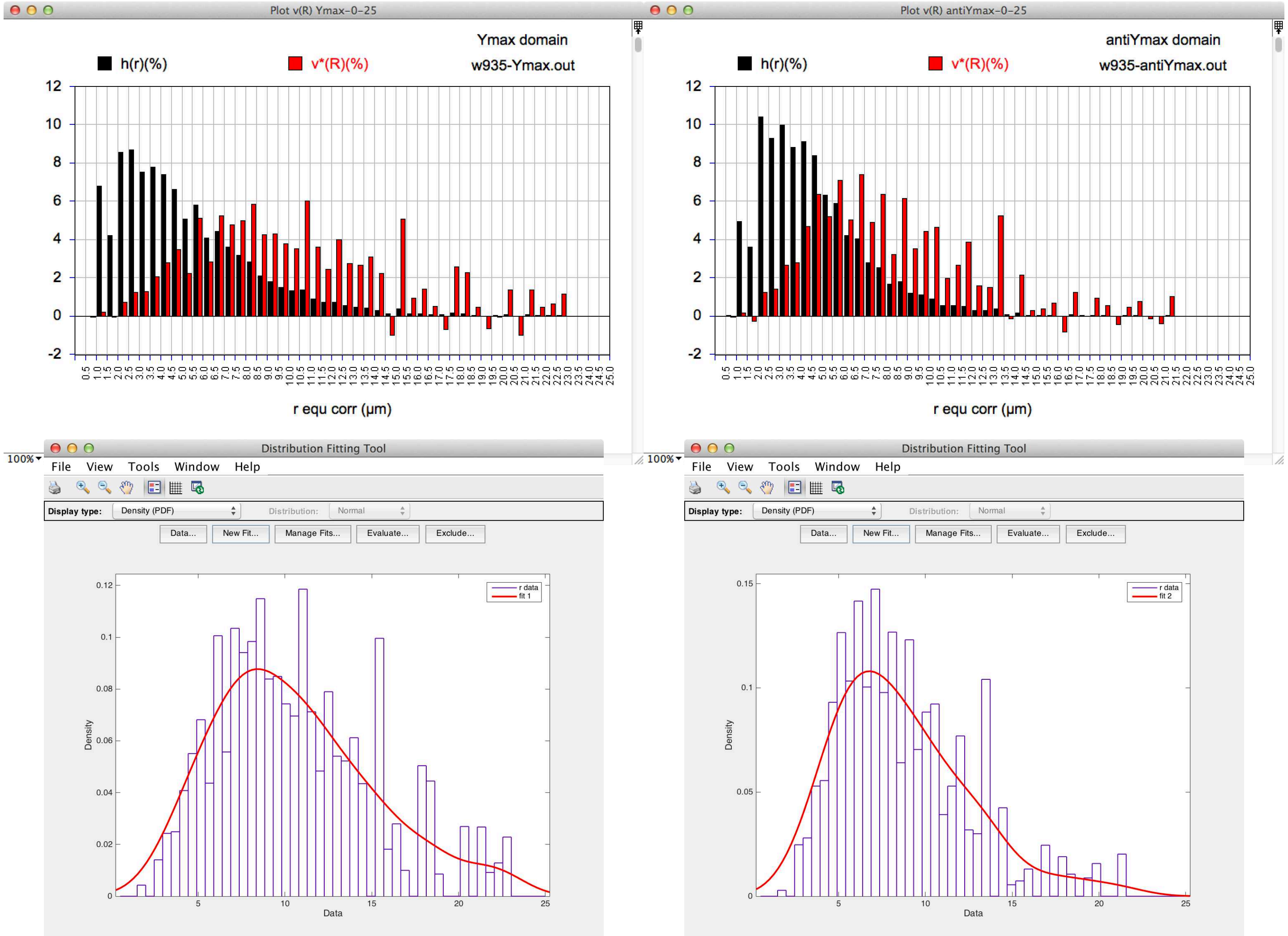
finding the cutoff

using density of w935.MISr1_052_169-th15 which is misor about Ymax
histogram shows 2 maxes Y max at ~22 GV
choose cutoff at 40 GV - by looking at histo
median = 46.461 GV



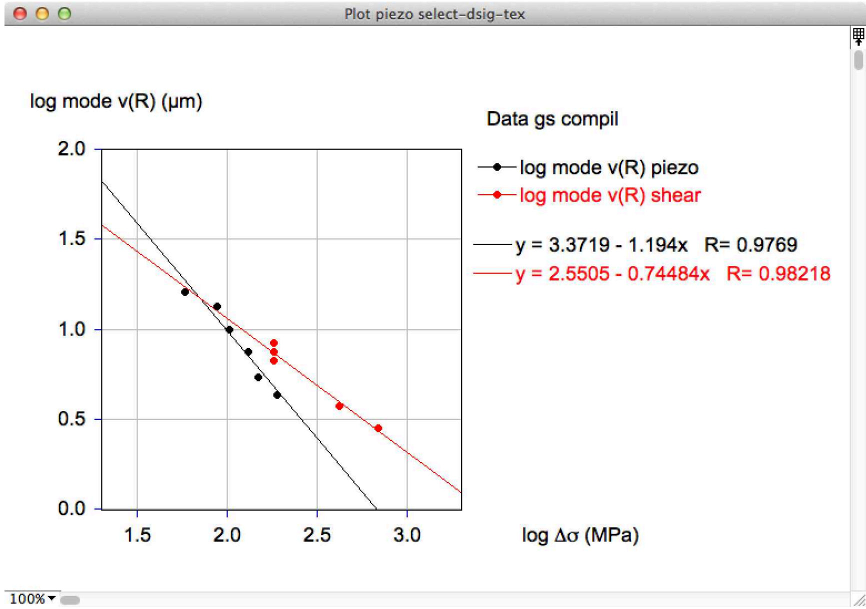


finding the modes

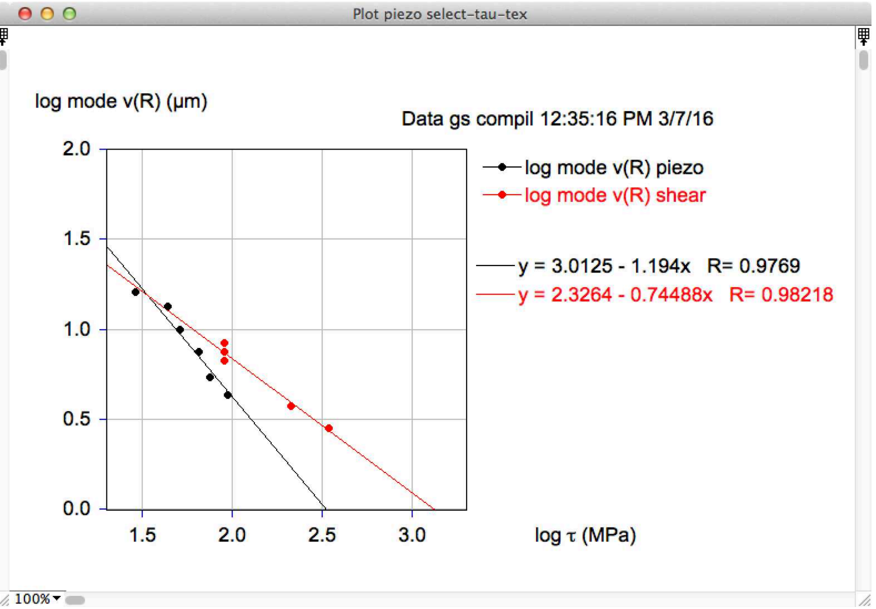


compile the data

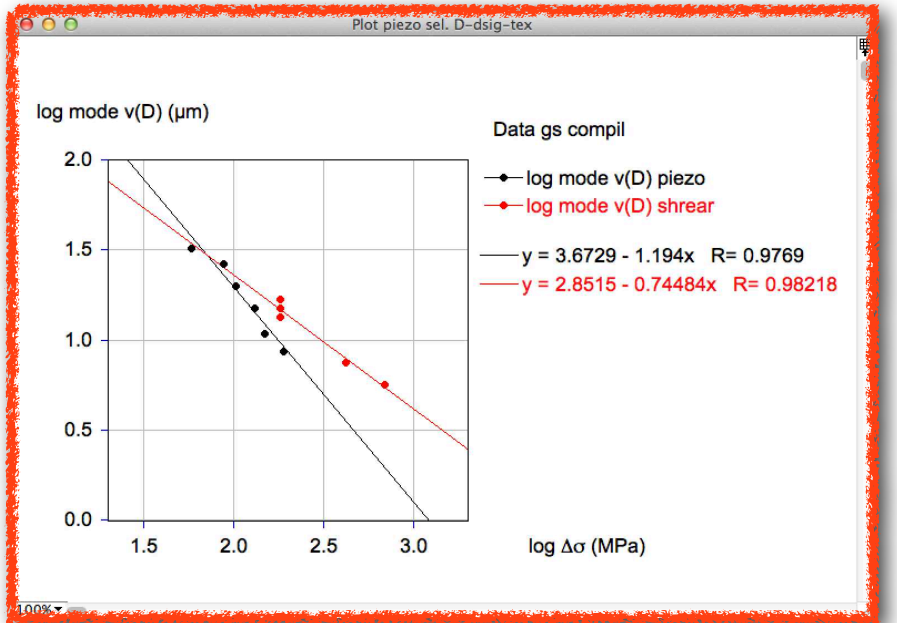
file	bdwidth	mean	median	mode	
1024	3	11.2799	10.9417	9.9697	
1025	3	14.3326	14.2531	13.2929	
1029	3	9.8560	8.9081	7.4949	
1050-m5	3	7.9871	6.6192	5.4242	
1051-m5	3	7.2336	5.6598	4.3131	
1081-m4	3	10.5499	8.8464	6.2525	
(same)	2	10.5499	8.4985	5.8990	
1081-m5	3	7.8843	6.5423	4.7071	
1126-m2	3	11.2210	11.4041	11.8788	(truncated to 0-15)
1143-m2	3	15.9396	16.0590	16.1919	
w935	1.5	9.5255	8.8836	7.4747	
w946	1.5	6.7962	4.9580	3.7222	
w1092	1.5	5.9887	3.7802	2.8333	
w935 Ymax	1.5	10.5231	9.8970	8.3990	
w935 antiYmax	1.5	8.6637	8.0686	6.6667	



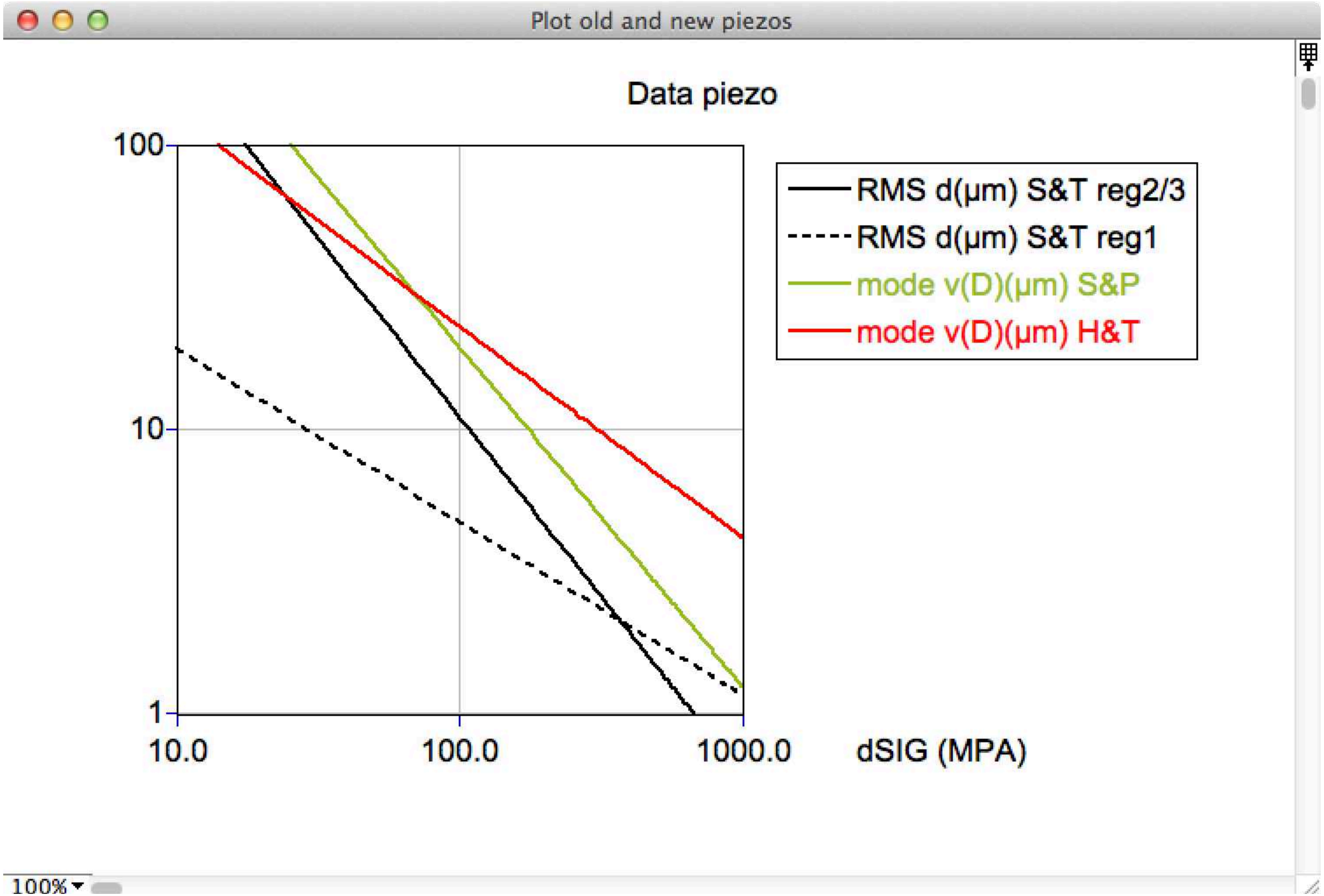
Plot piezo select-dsig-tex
log mode v(R) (µm)
Data gs compil
2.0
1.5
1.0
0.5
0.0
1.5
2.0
2.5
3.0
log mode v(R) piezo
log mode v(R) shear
y = 3.3719 - 1.194x R= 0.9769
y = 2.5505 - 0.74484x R= 0.98218
log Δσ (MPa)
100%



Plot piezo select-tau-tex
log mode v(R) (µm)
Data gs compil 12:35:16 PM 3/7/16
2.0
1.5
1.0
0.5
0.0
1.5
2.0
2.5
3.0
log mode v(R) piezo
log mode v(R) shear
y = 3.0125 - 1.194x R= 0.9769
y = 2.3264 - 0.74488x R= 0.98218
log τ (MPa)
100%



Plot piezo sel. D-dsig-tex
log mode v(D) (µm)
Data gs compil
2.0
1.5
1.0
0.5
0.0
1.5
2.0
2.5
3.0
log mode v(D) piezo
log mode v(D) shrear
y = 3.6729 - 1.194x R= 0.9769
y = 2.8515 - 0.74484x R= 0.98218
log Δσ (MPa)
100%



Plot old and new piezos
Data piezo
100
10
1
10.0
100.0
1000.0
dSIG (MPA)
RMS d(µm) S&T reg2/3
RMS d(µm) S&T reg1
mode v(D)(µm) S&P
mode v(D)(µm) H&T
100%

was it worth it ?

1. check Stipp & Tullis piezometer using EBSD
measured same h(d) - modes of v(D) $\approx 2 \cdot \mathrm{RMS}(d)$

2. check if piezometer is indeed different for different regimes
cannot say yet - not enough data re-done for regime 1
for shear: maybe all the same

3. check if piezometer is same for coaxial and shear
no the same

4. check if piezometer is texture dependent
yes it is !
:-)

part 2

2. olivine - pyroxene (= work in progress)

motivation:
torsion experiments to find flow law for mantle material

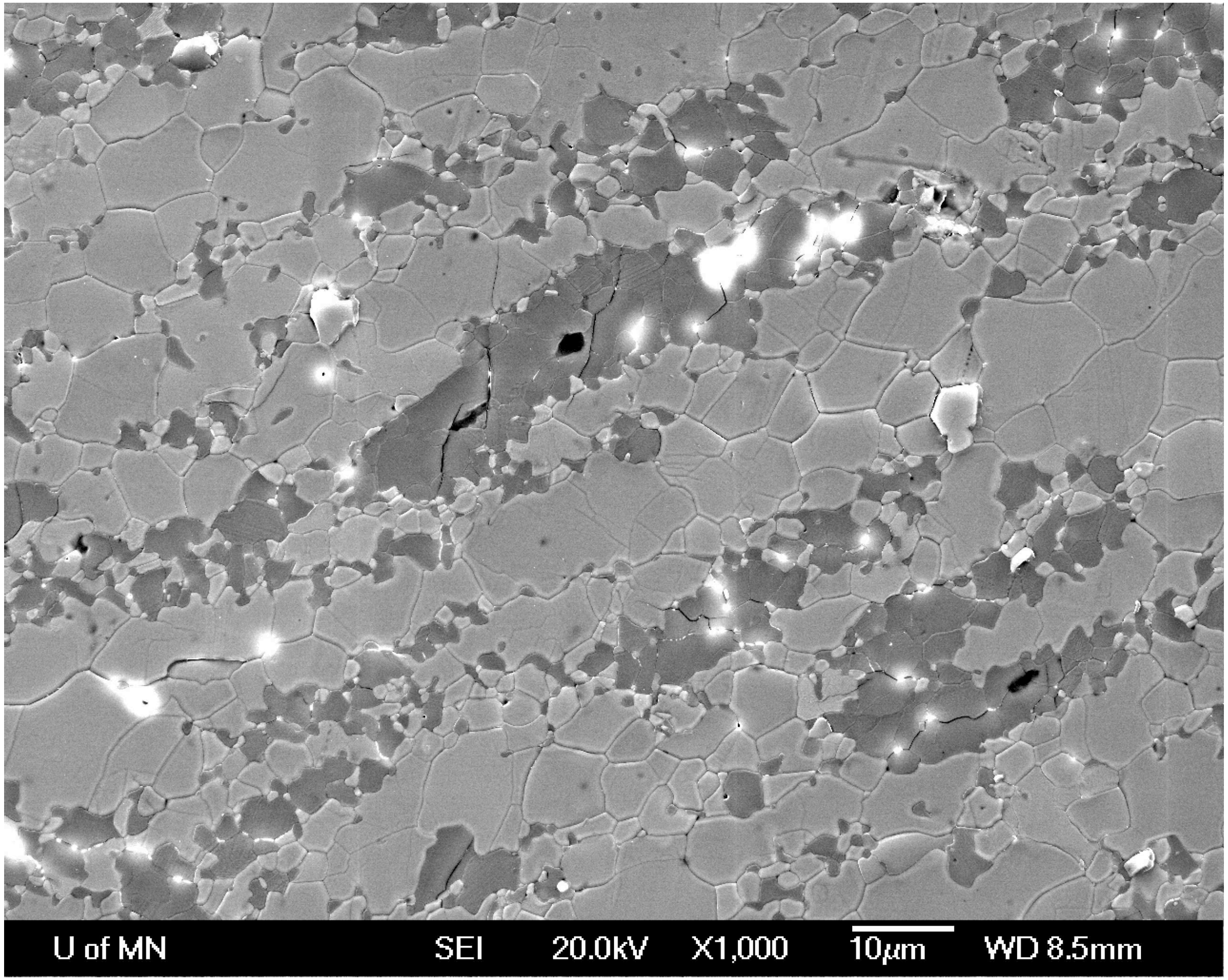
first finds:
dislocation creep and diffusion creep

aim of microstructure analysis:
step 1: find grain size(s) of olivine and pyroxene
step 2: find shape(s)
step 3: find spatial relations

... think about results

... see forthcoming paper by Miki Tasaka

torsion experiments on Ol - Opx



Miki Tasaka
David Kohlstedt
Mark Zimmermann

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70:30 mixture
olivine-orthopyroxene

Paterson apparatus
T = 1200°C
p_c = 300MPa
$\dot{\gamma} = 1.6 \cdot 10^{-4}\ s^{-1}$
$\gamma = 1.9$

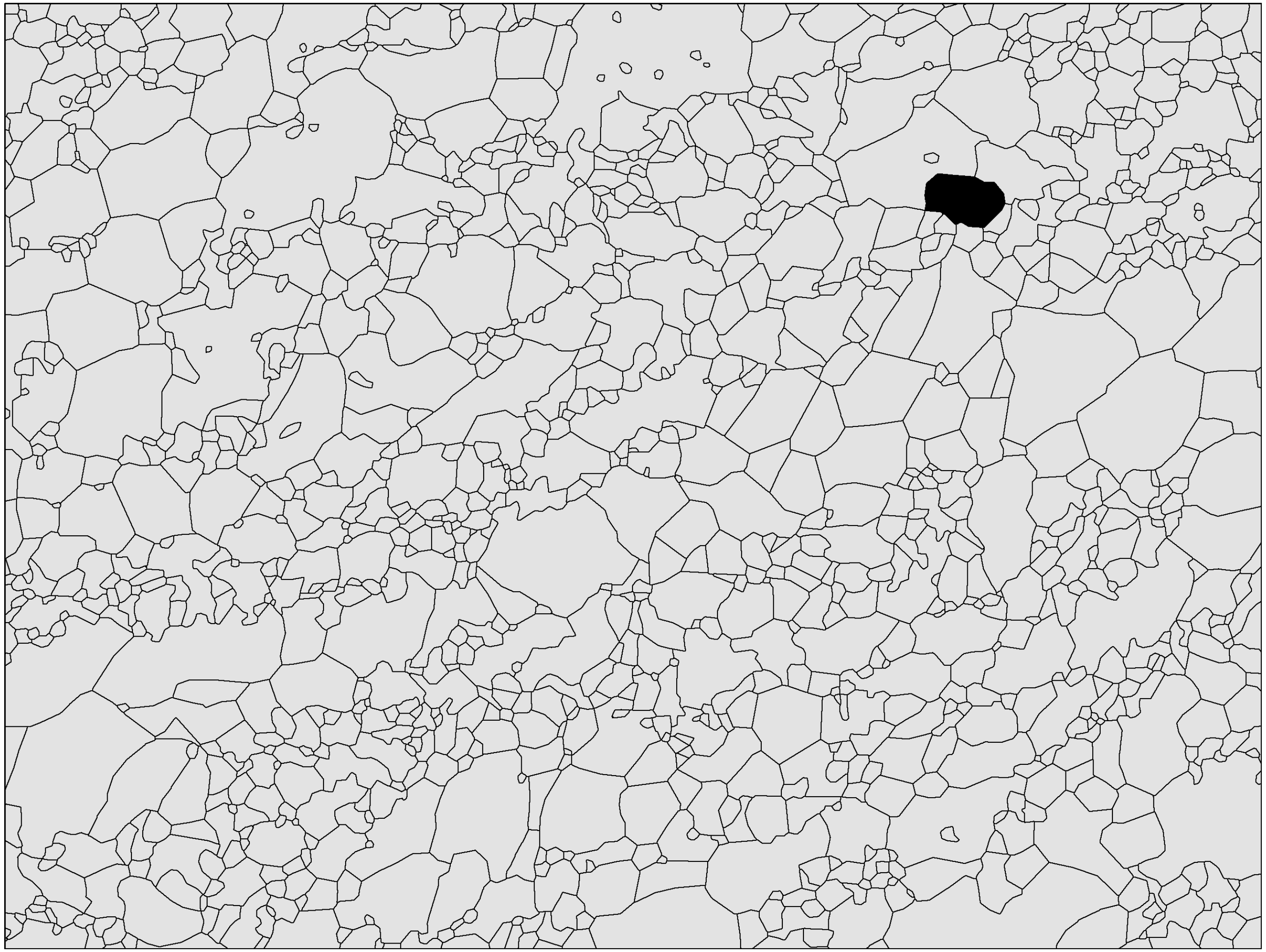
torsion experiments on Ol - Opx

segmentation

olivine orthopyroxene all grains

⇒ *have to be careful with segmentation*

torsion experiments on Ol - Opx



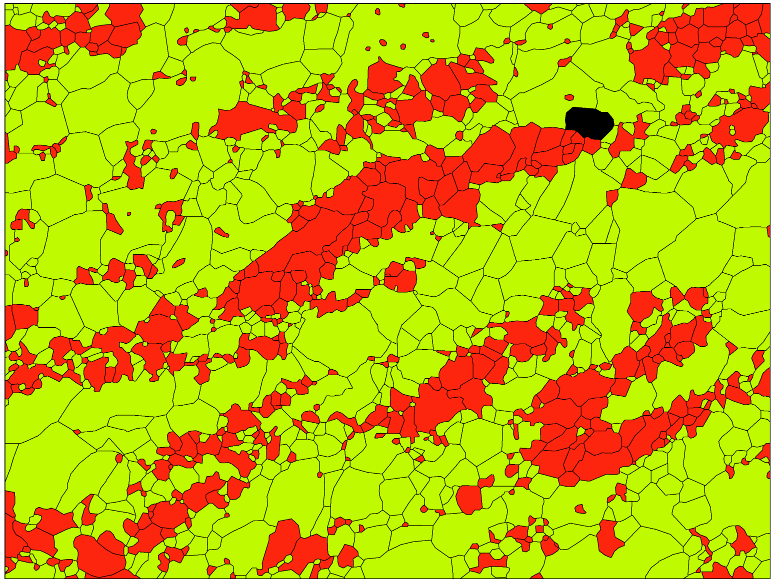
segmentation
grain boundary map

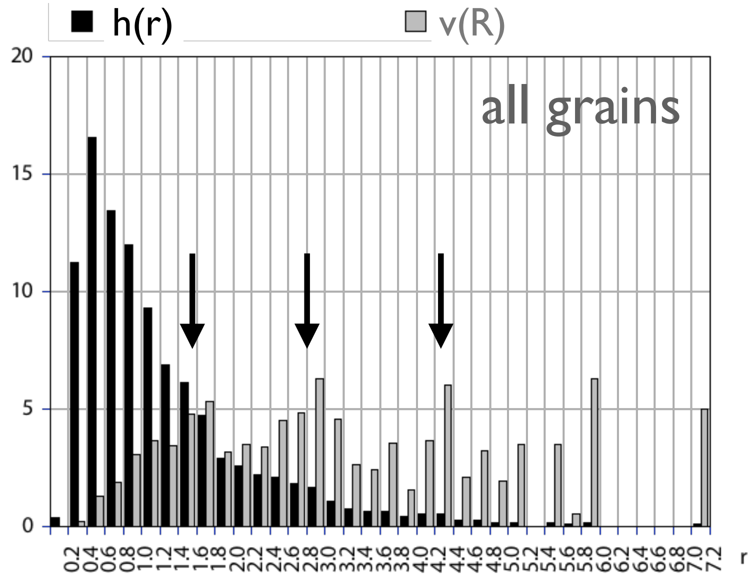
all grains

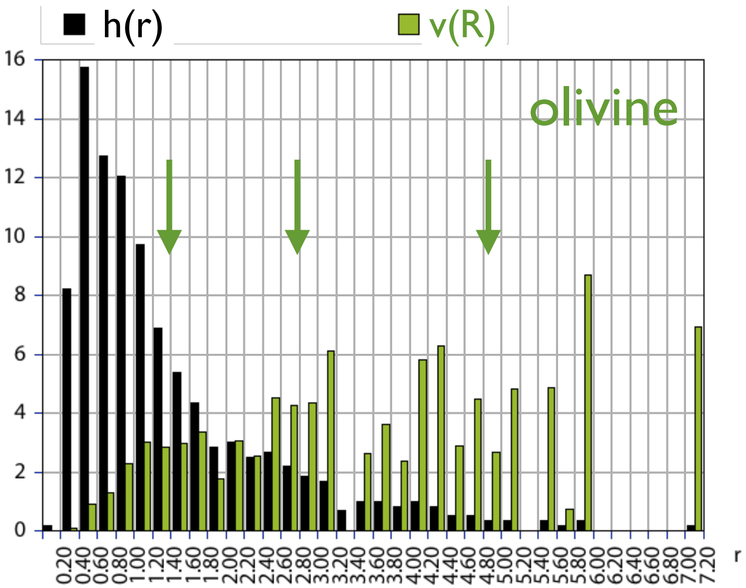
torsion experiments on Ol - Opx

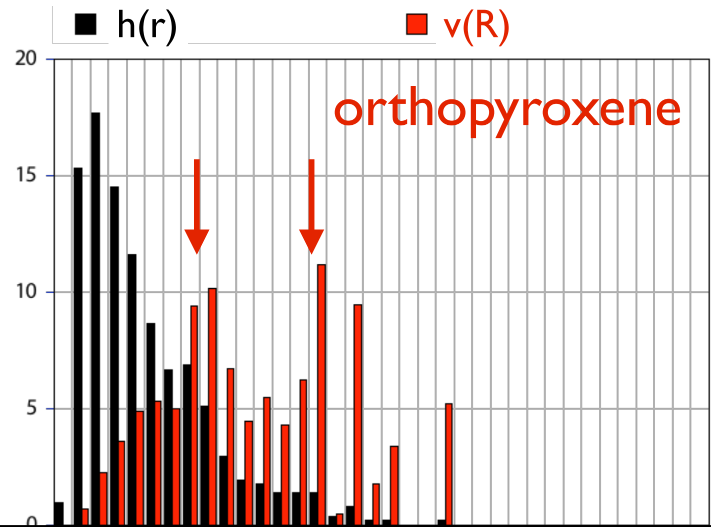
segmentation
grain boundary map
grain map (segments)

2D and 3D grain size distributions



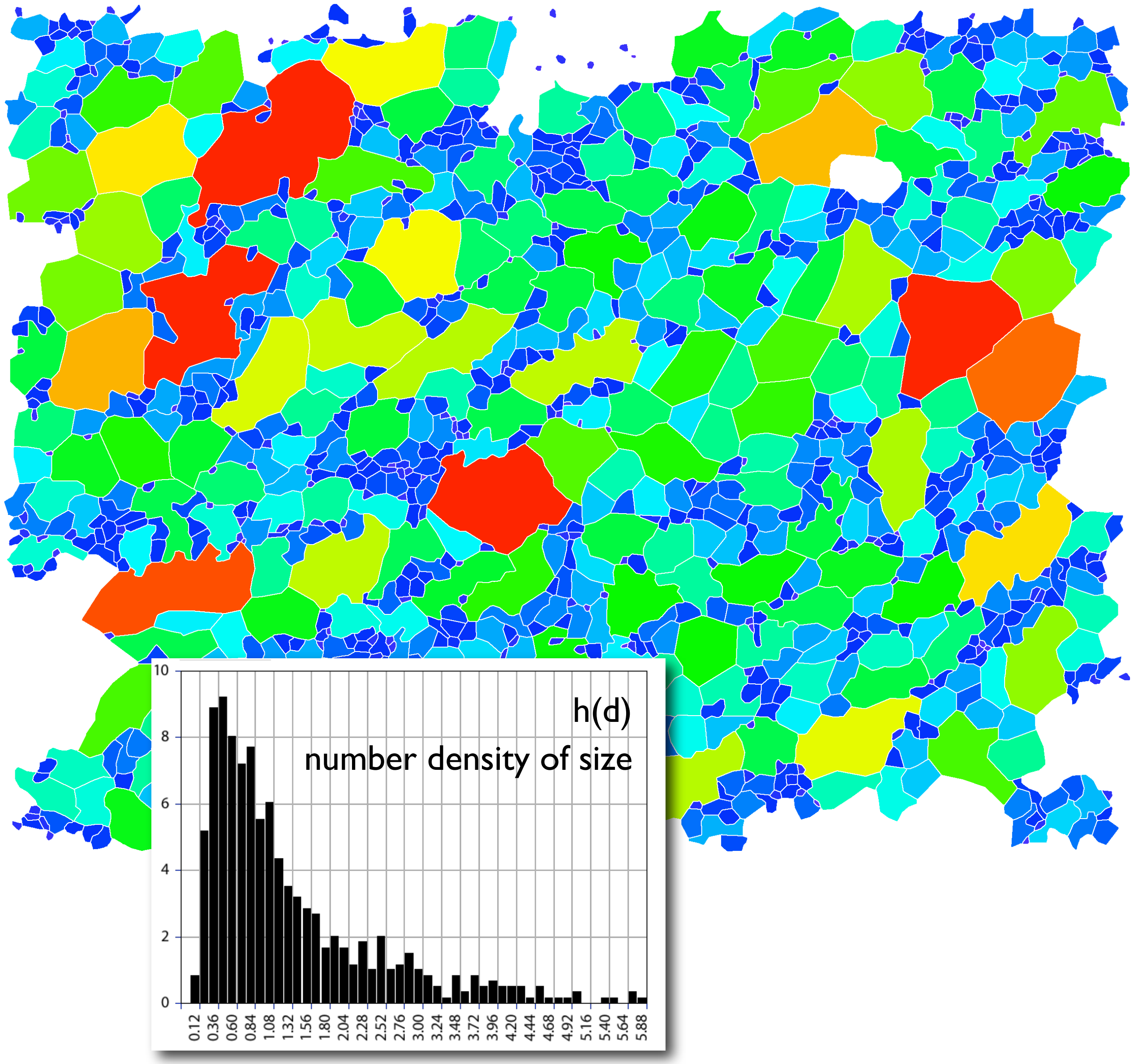


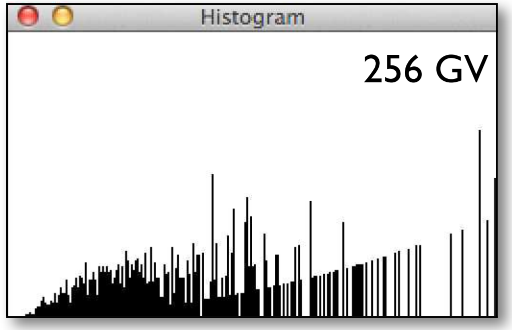


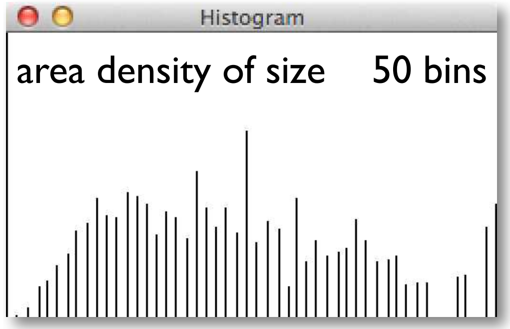


⇒ *detect multiple modes in 3D*

grain size mapping





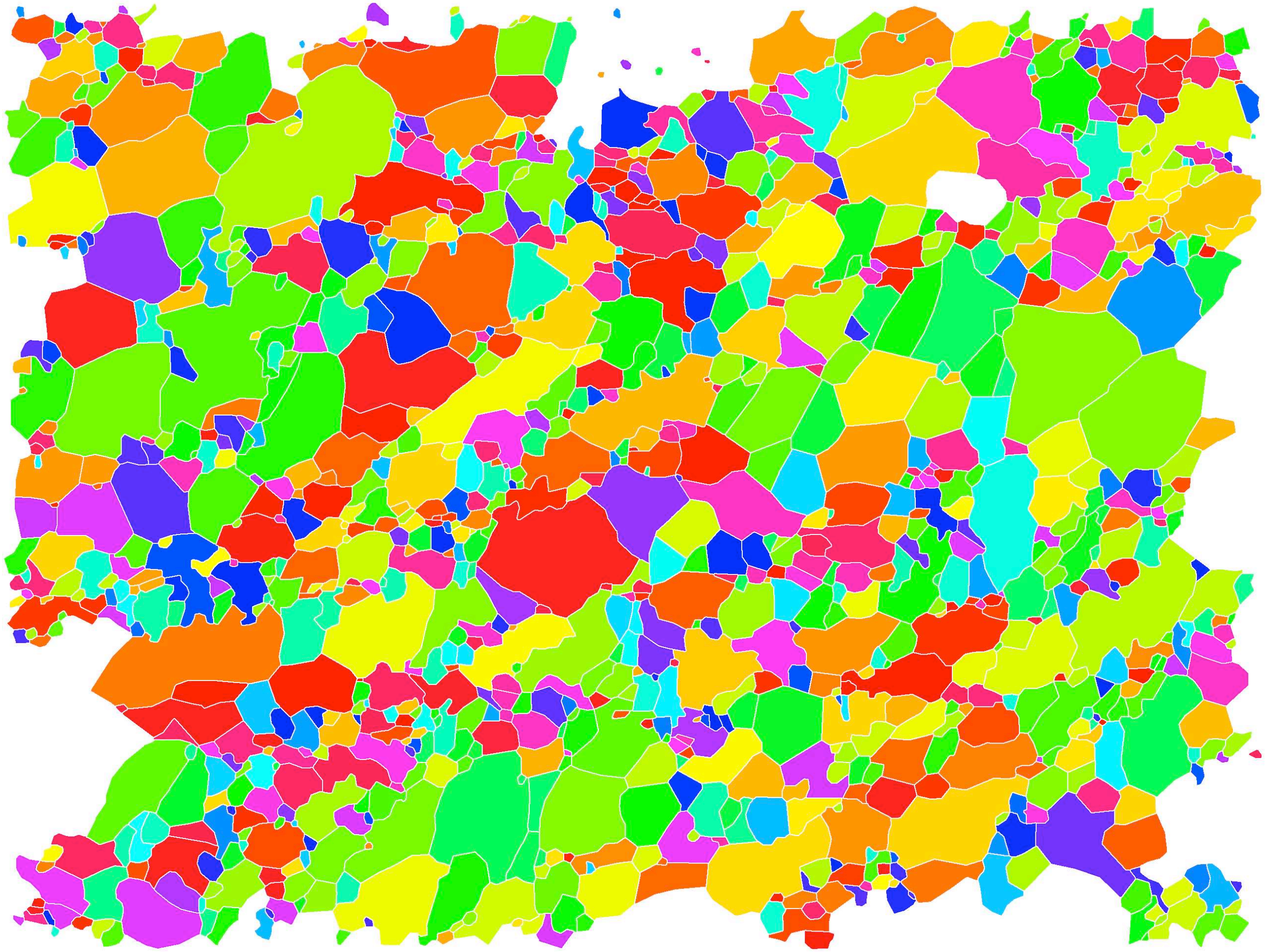


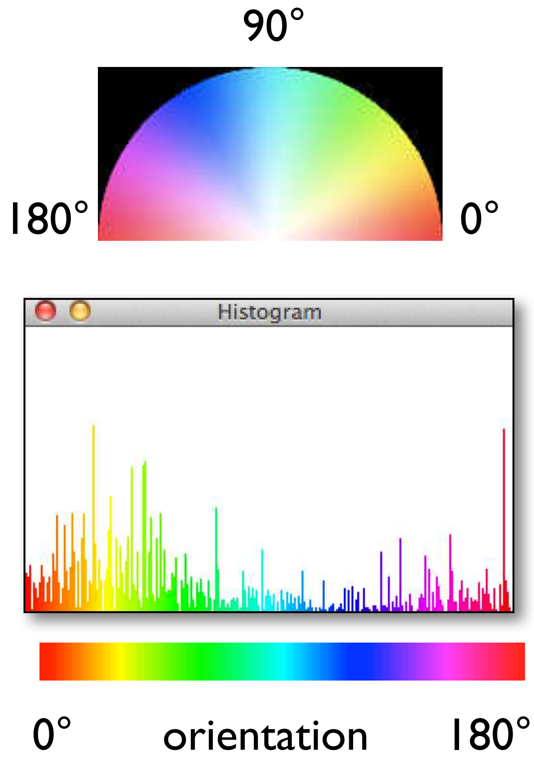
0.0 r_{equ} (μm) 6.0

0.0 d_{equ} (μm) 12.0

⇒ *size domains*

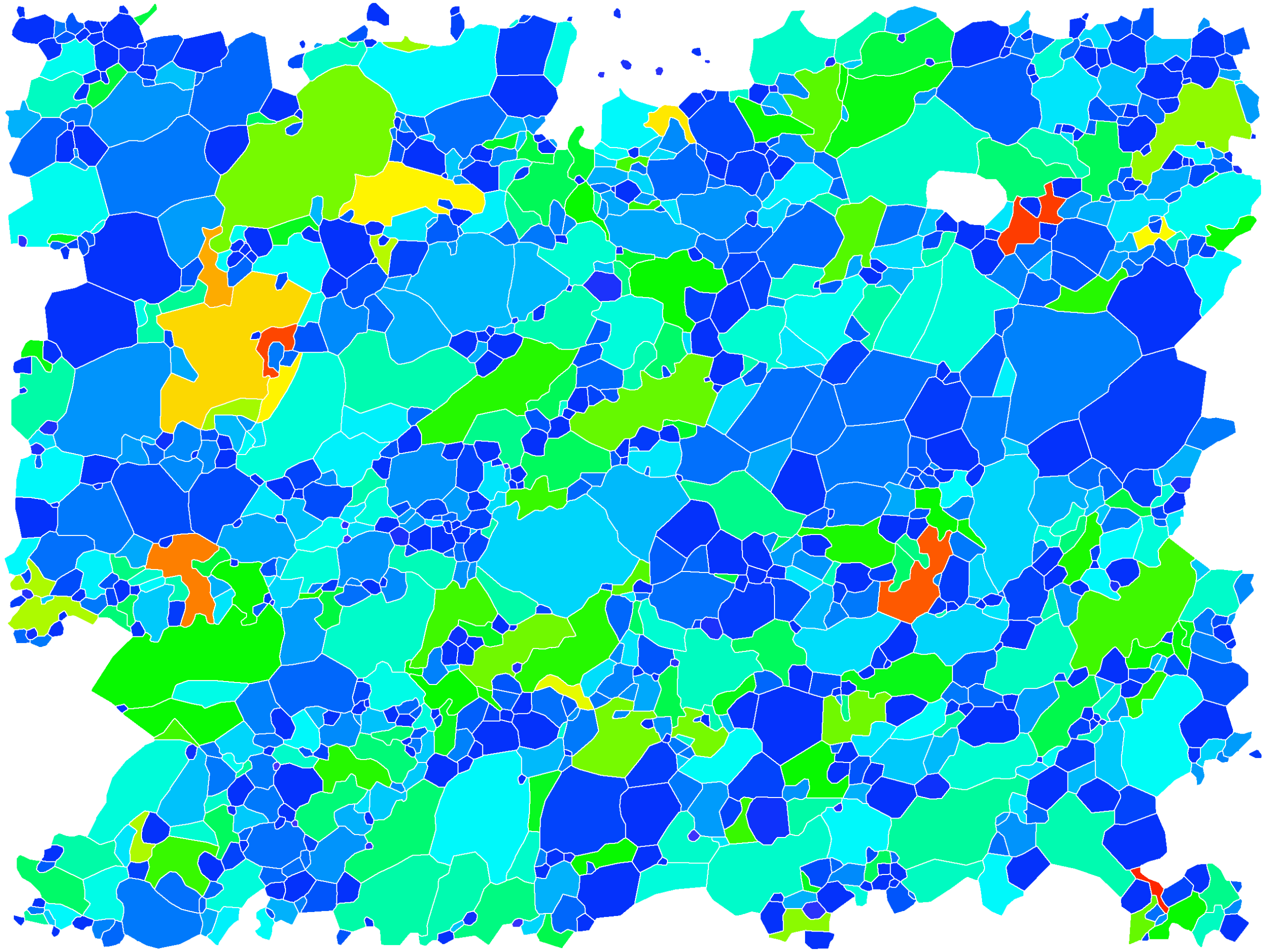
orientation mapping

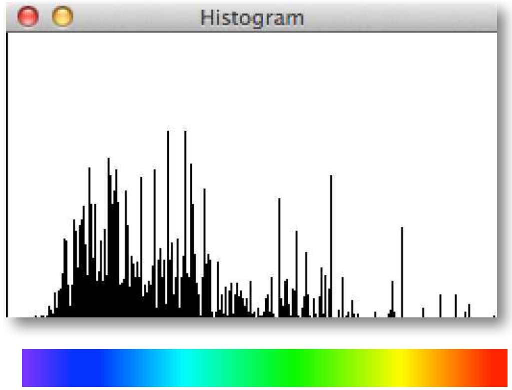




⇒ *random orientation*

shape factor mapping





0.0 P/P_{equ} 2.0

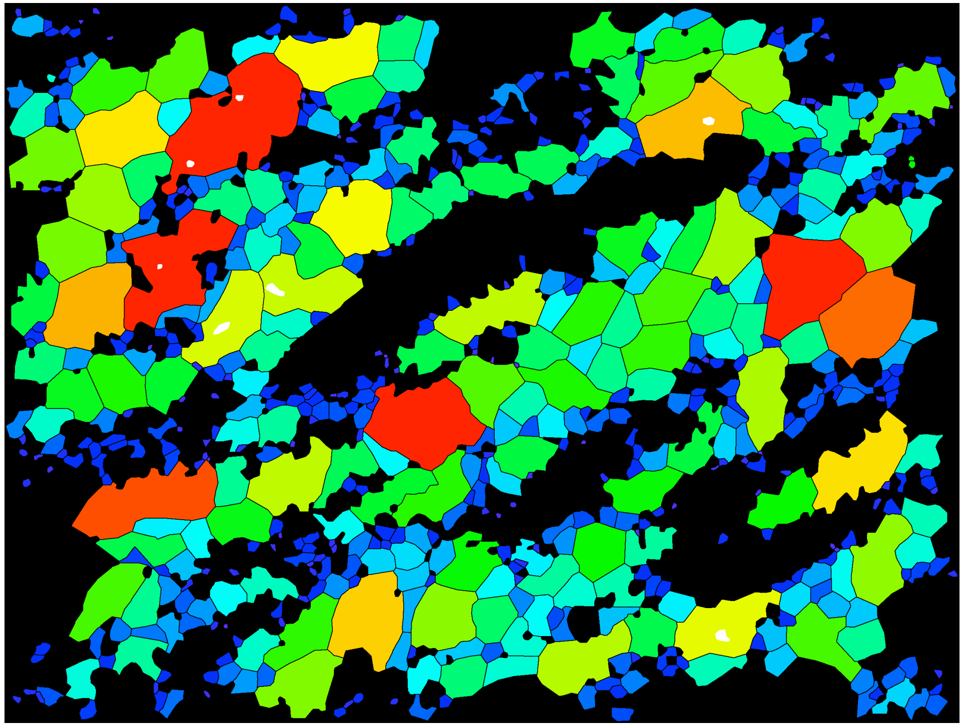
round convex ⟷ elongate convex-concave

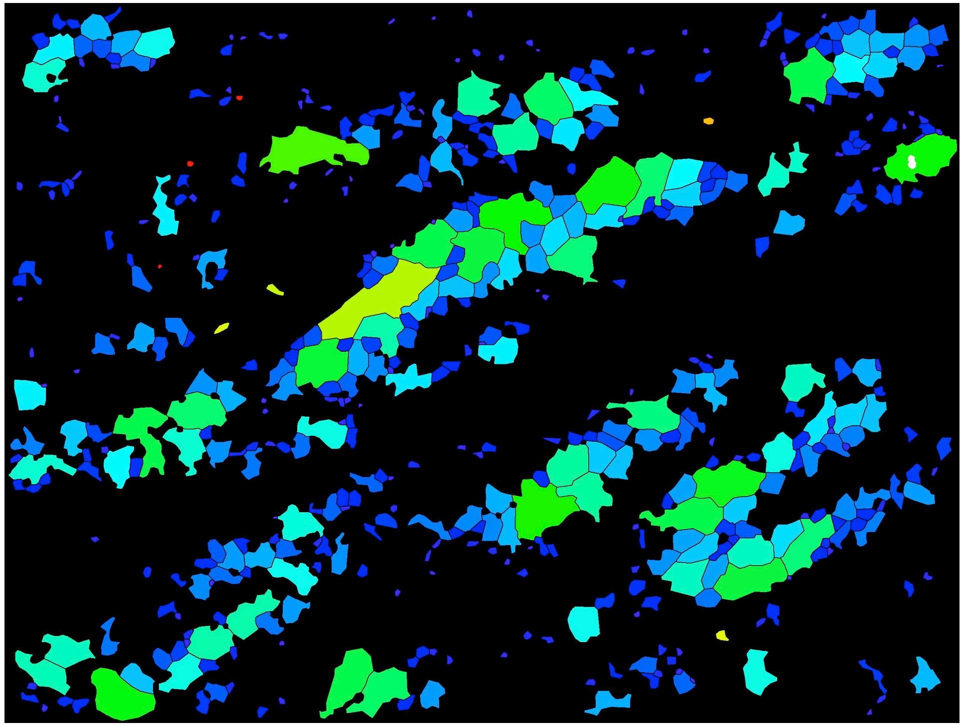
$SF1 = P_{measured} / P_{equivalent}$ = large if grain boundary lobate

$(0.00 < SF1 < \infty)$

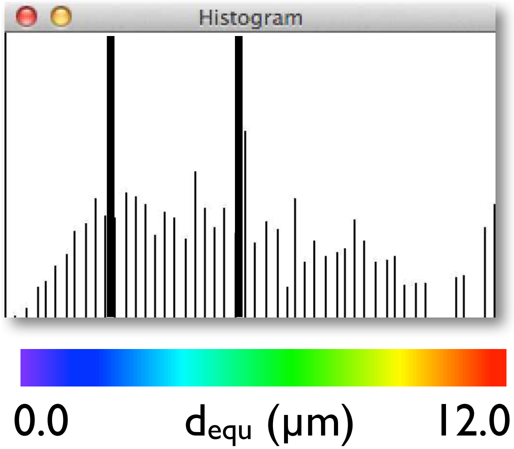
$(0.00 < SF1 < \infty)$

2 phases - 4 grain sizes !

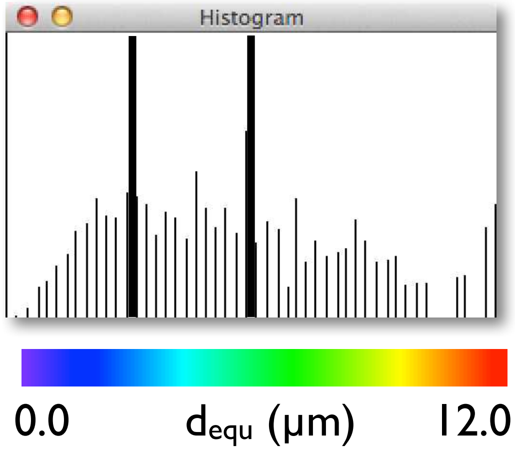




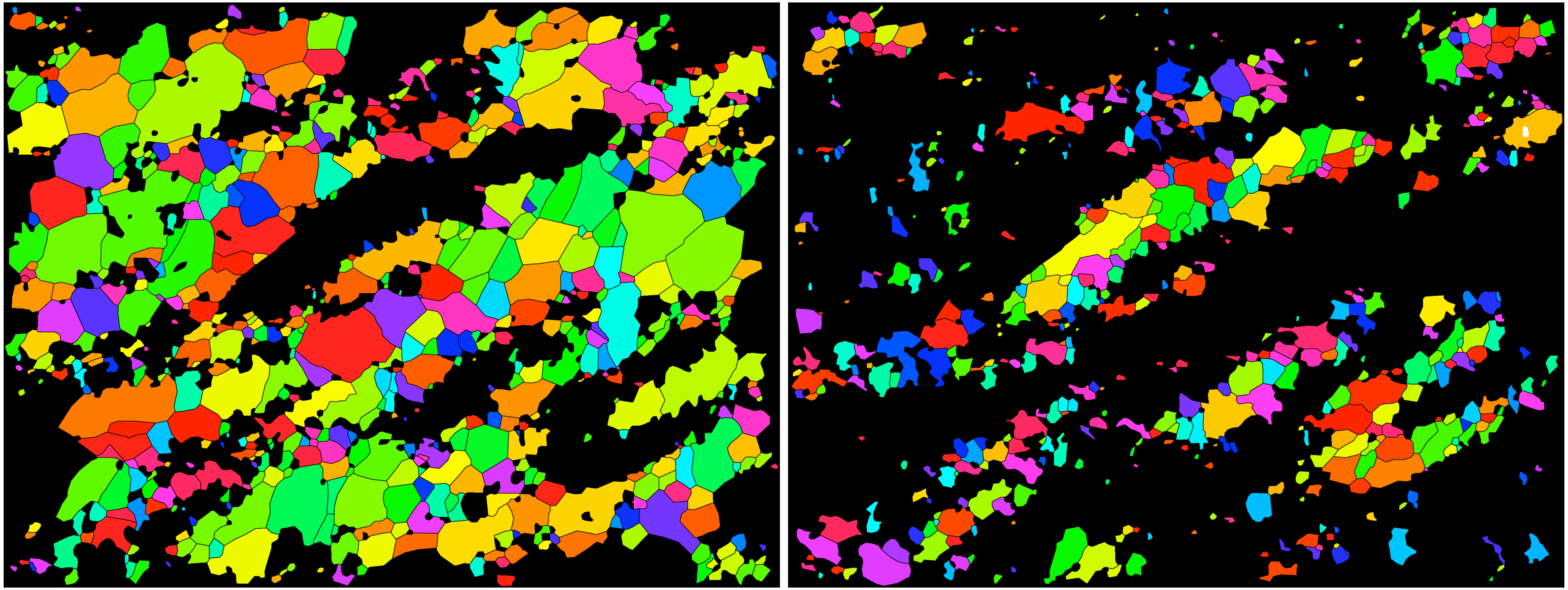
ol

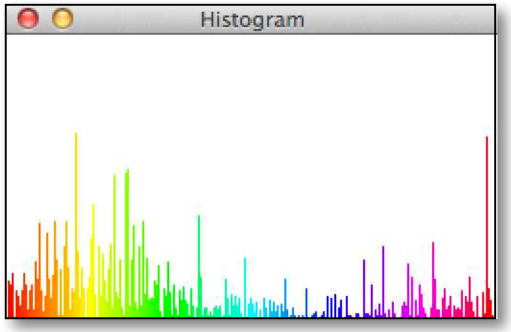


opx



preferred orientation ?





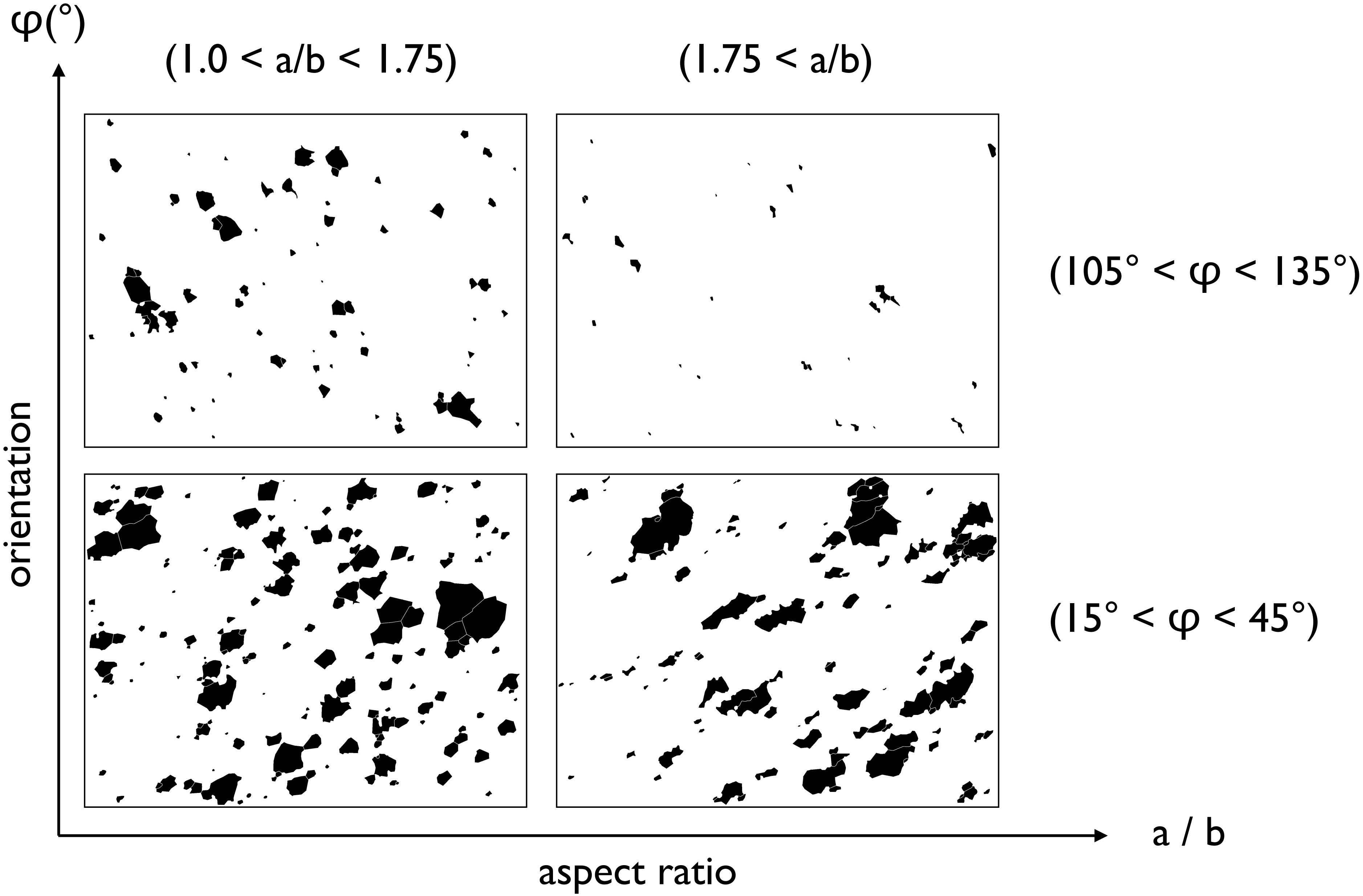
0° orientation 180°

90°

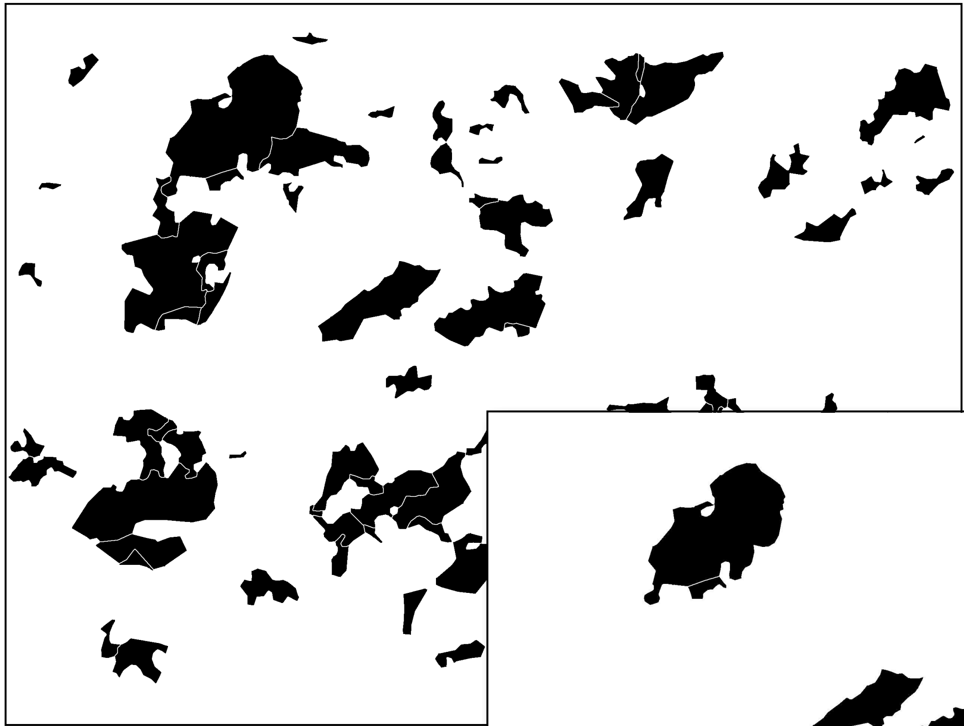
180° 0°

⇒ *Ol and Opx = random orientation*

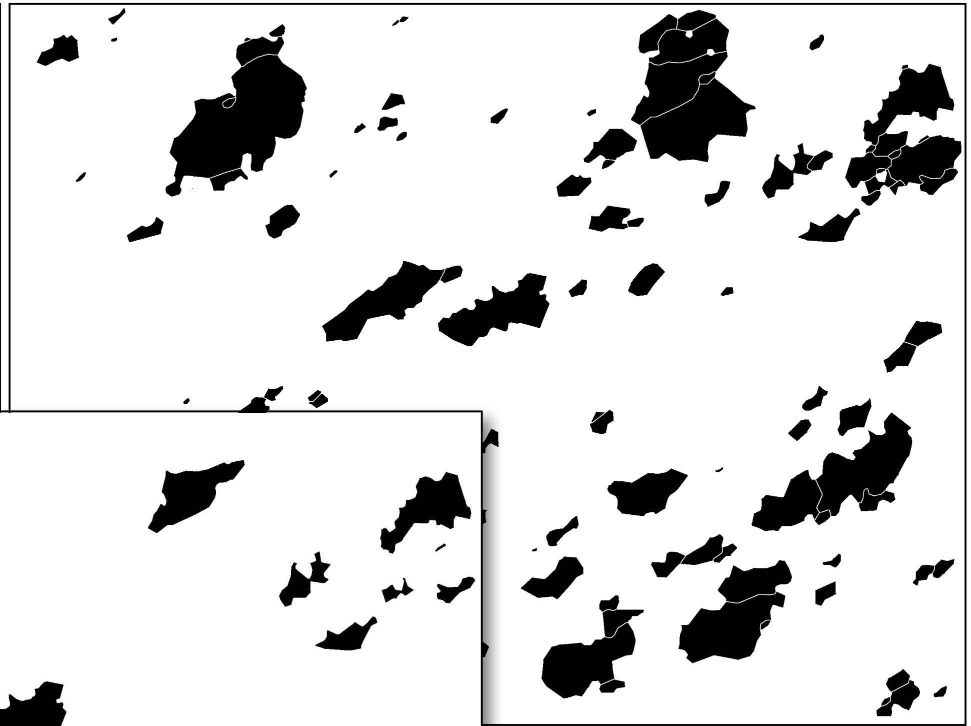
intersecting 2 feature bitmaps



intersecting 3 feature bitmaps



(1.5 < SF1 < 2.0)

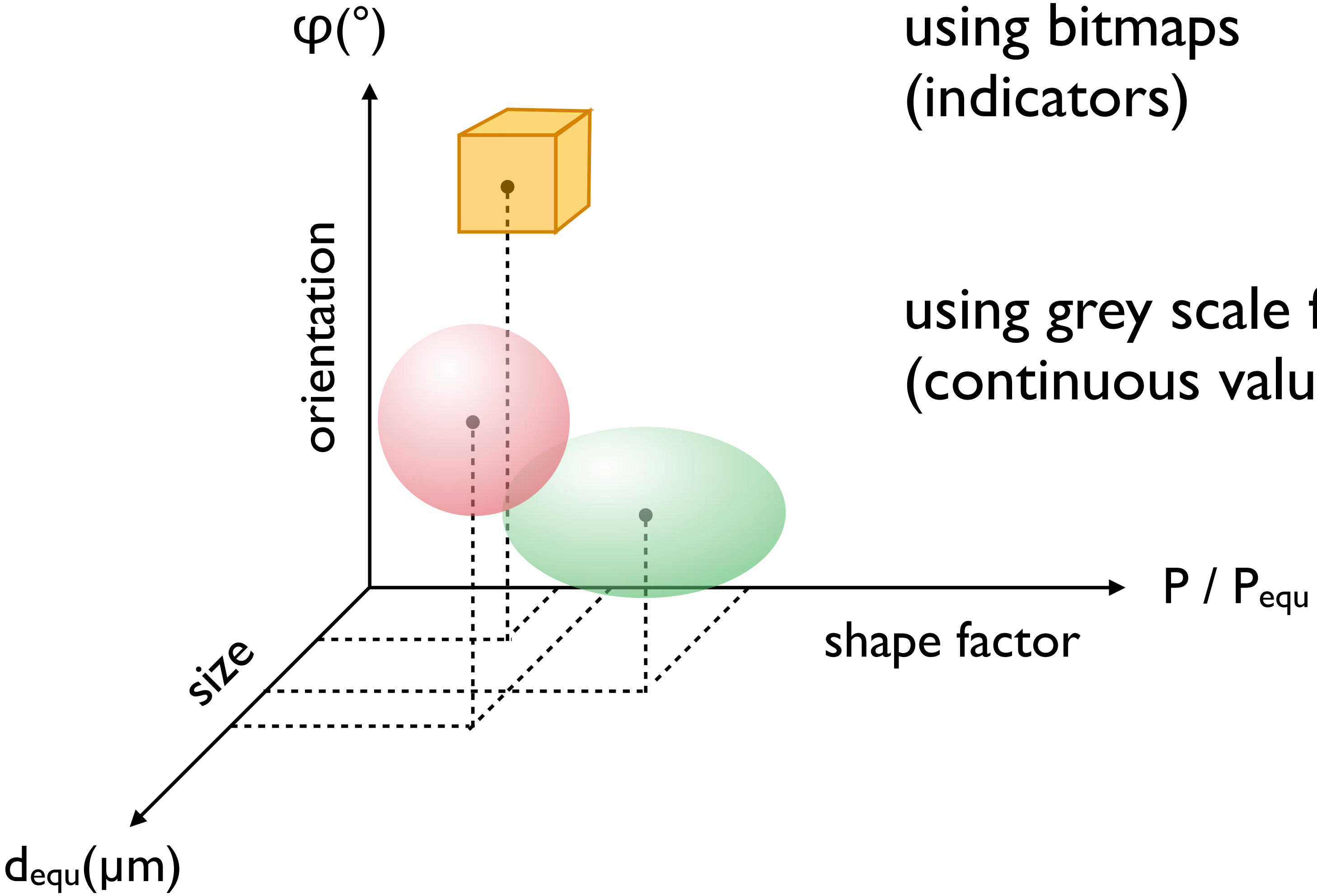


(15° < φ < 45°)

∩ (1.75 < a/b)

(1.5 < SF1 < 2.0) ∩ (1.75 < a/b) ∩ (15° < φ < 45°)

feature space



using bitmaps
(indicators)

using grey scale feature maps
(continuous values)

take-home message(s)

- use image analysis (processing) to measure - not to illustrate
- use state-of-the-art image analysis to match state-of-the-art experimentation

- think twice before declaring "the mean grain size"
- use modes of 3D grains - they are most meaningful

- put the numbers back into the picture → map → visualize
- think of images as maps → be quantitative → scale and calibrate
  (you can observe a lot by watching) → (you can understand a lot by measuring)

- think of microstructures as multidimensional → plot data in feature space (= intersect images)

... and be happy if you do not get a simple answer

announcement

European Geosciences Union
General Assembly 2016
Vienna | Austria | 17–22 April 2016

EGU.eu

TS1 – Brittle Deformation and Fault-related Processes

Programme Committee Login

Suggest a Session here

TS2 – Ductile Deformation, Metamorphism and Magmatism

Programme Committee Login

Suggest a Session here

Suggested Session
Advances in Microstructure and Texture Analysis
[Suggest a new Title]
Conveners: Renee Heilbronner, Rüdiger Kilian
[Suggest a Convener and Description Change]