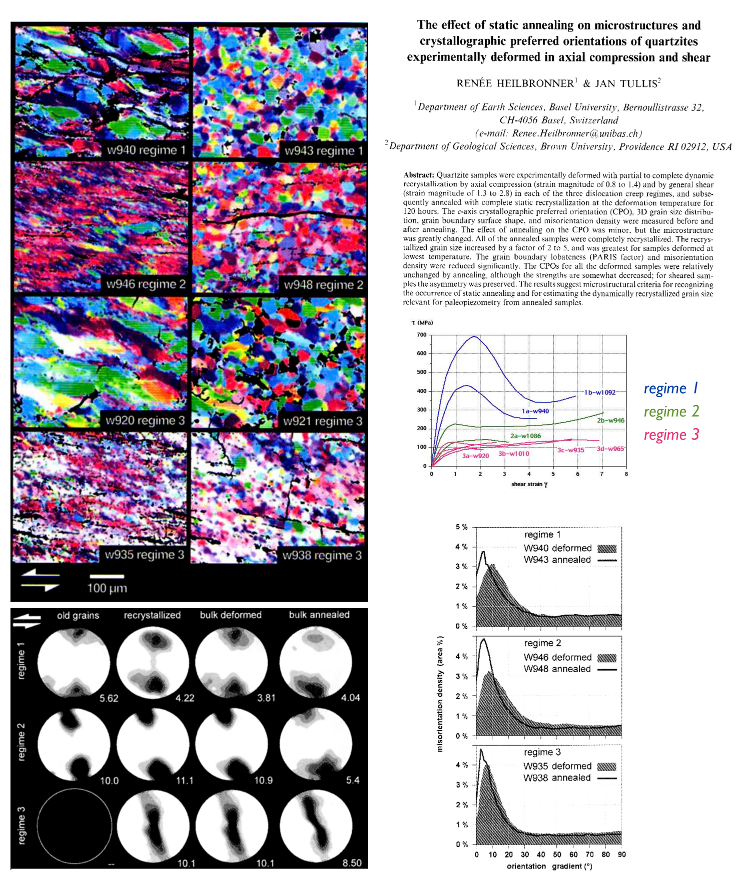
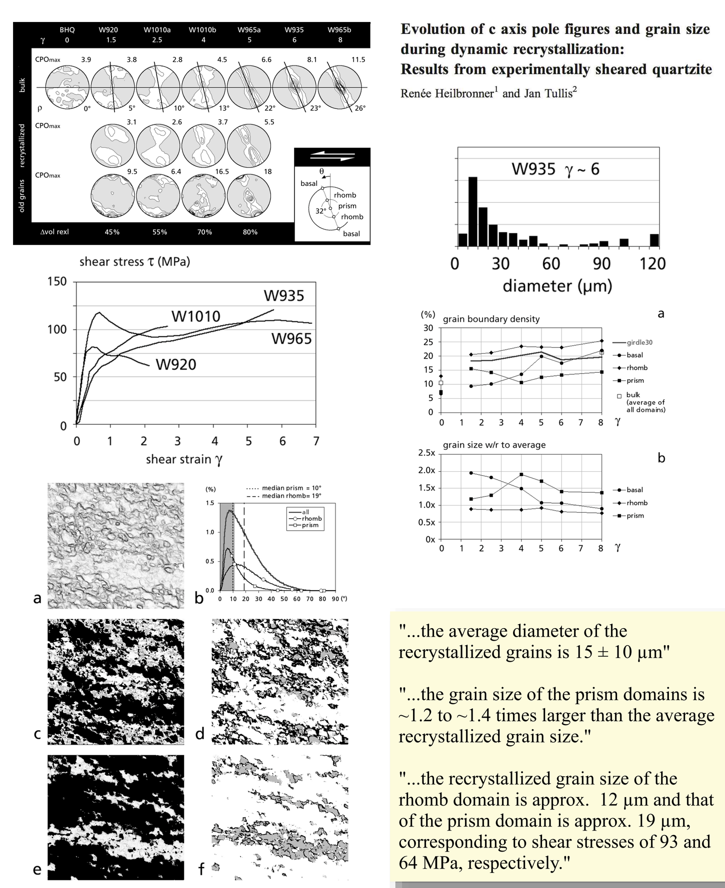
EGU2016-8731 BHQ revisited (1) - Looking at grain size

Texture and grain size of Black Hills Quartzite - as published

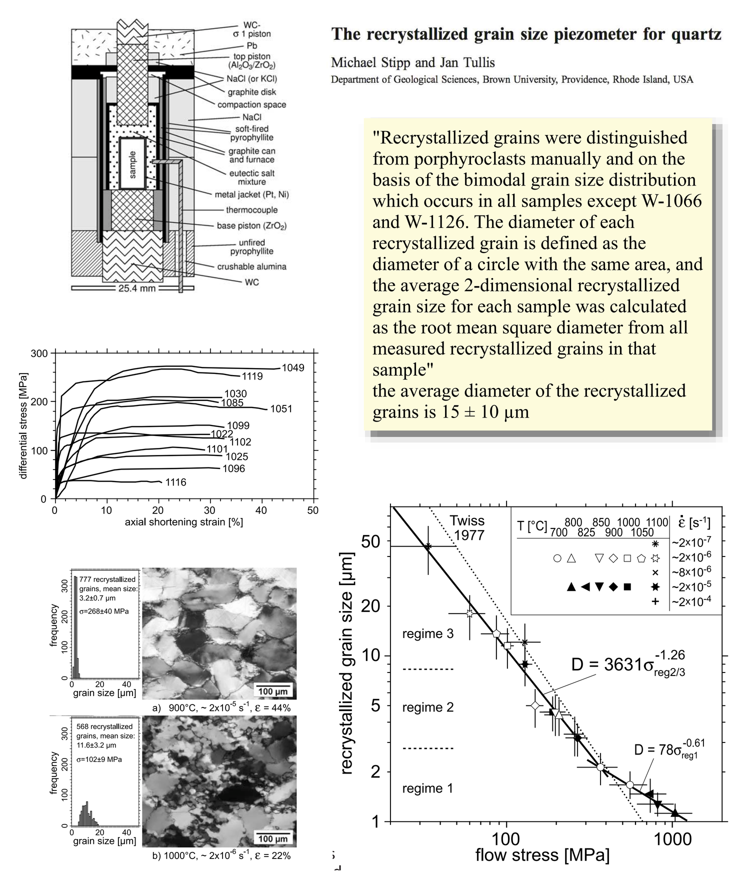
regime 1 2 3 — coaxial and shear



texture domains — shear

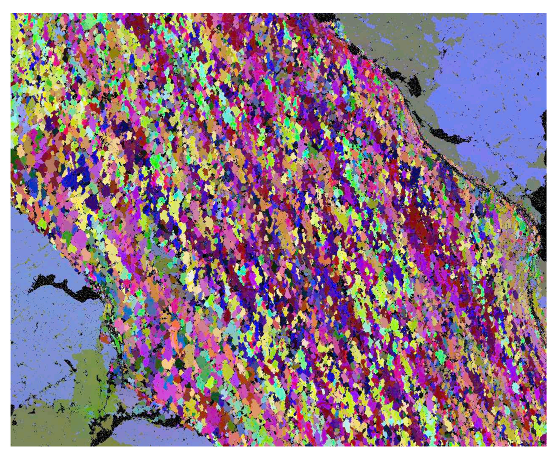


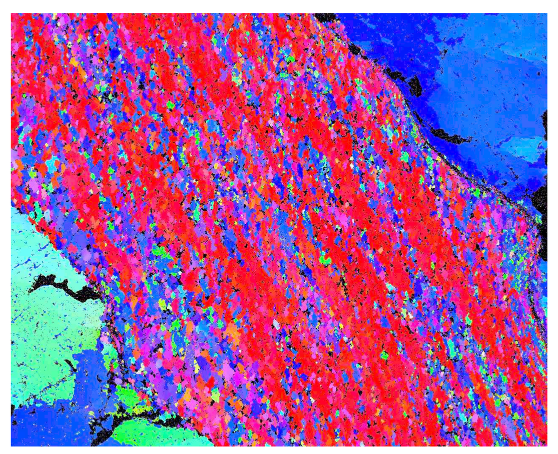
piezometer — coaxial



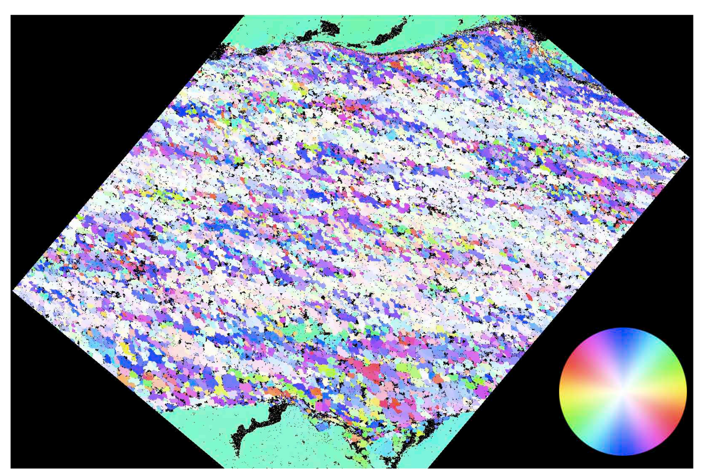
From EBSD to c-axis orientation gradient image to grain map

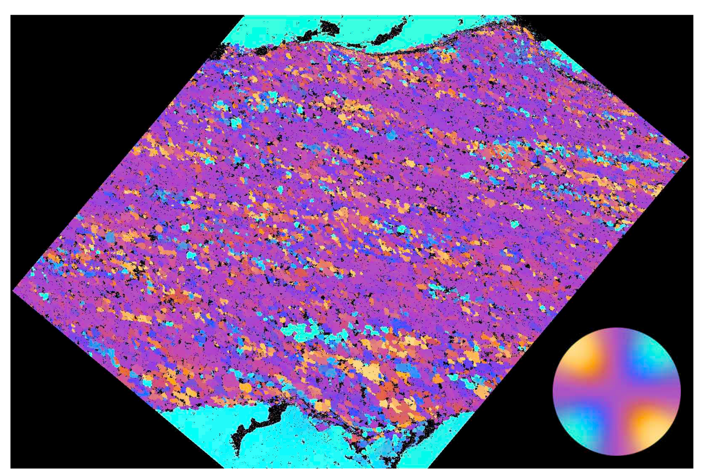
shearing experiments

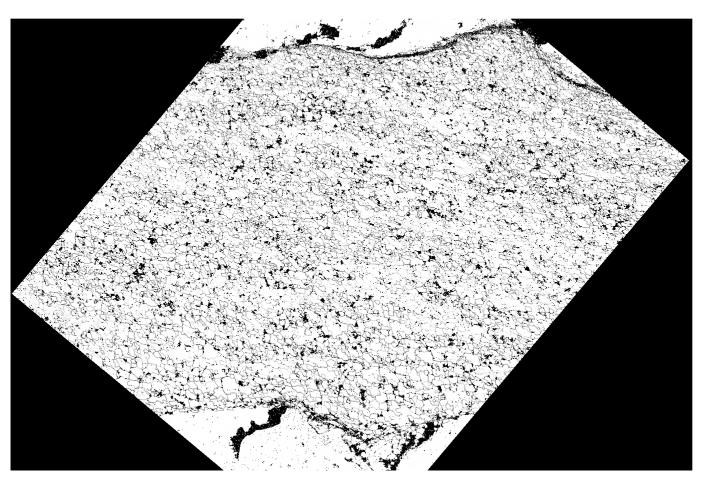

w935.Euler Color

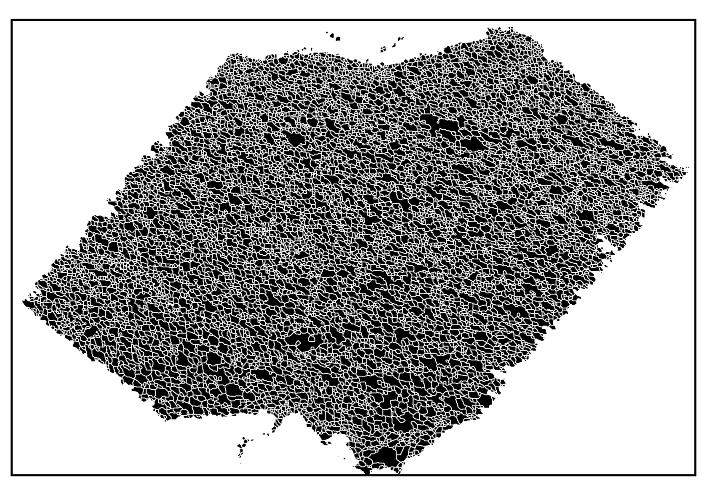

w935.IPFZ Color

500 µm

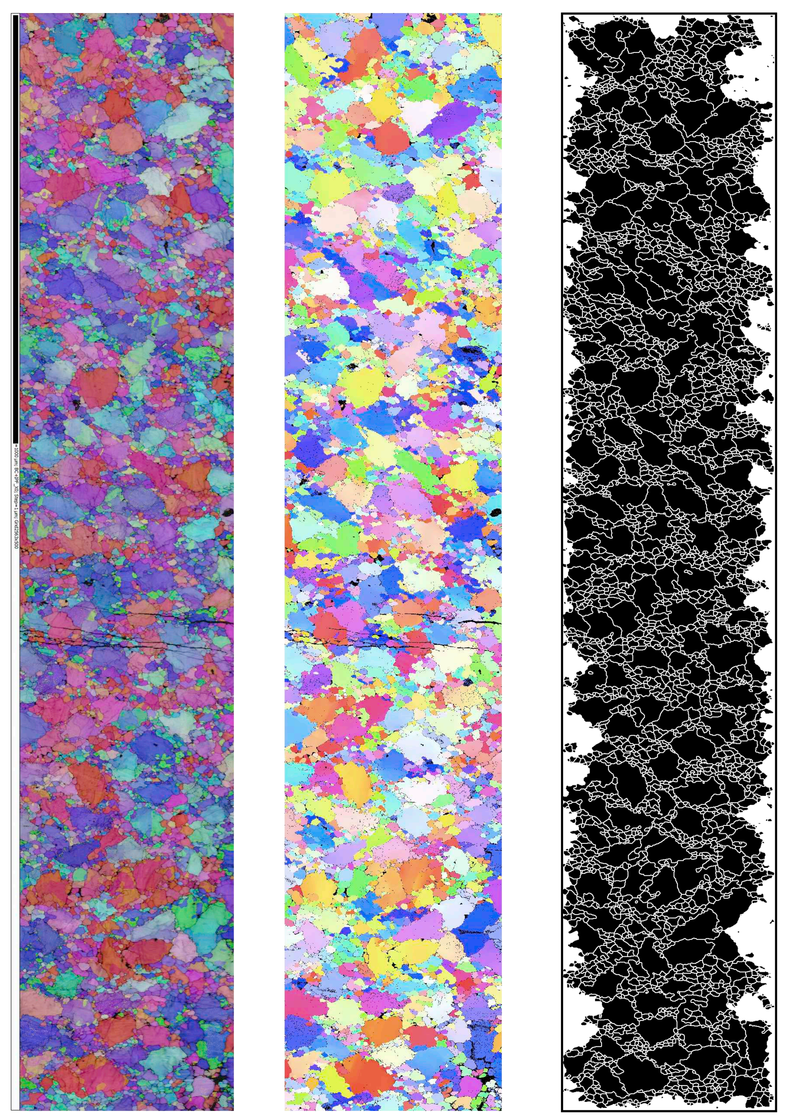

w935.COI2

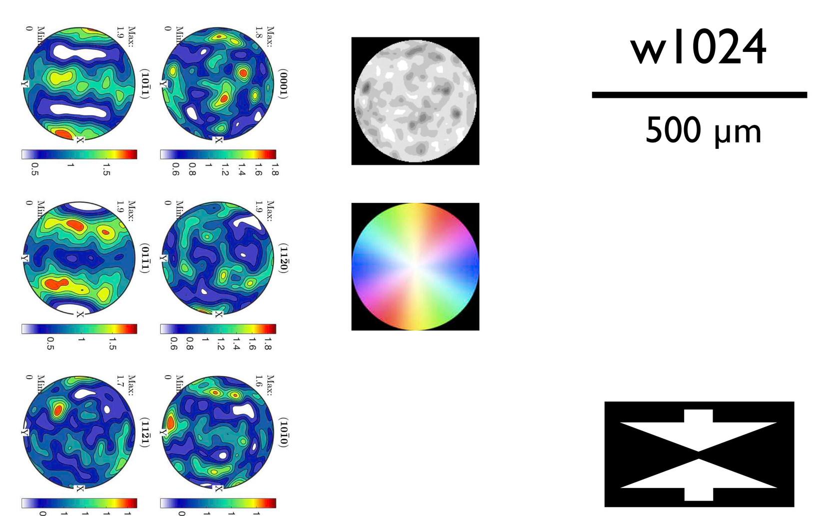

w935.COI4


w935.EDG8a (=OGI)


w935.TH200.areas

coaxial experiments





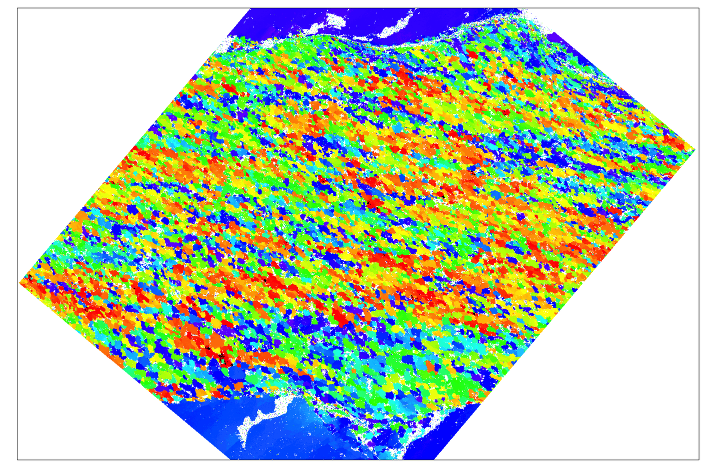
w1024

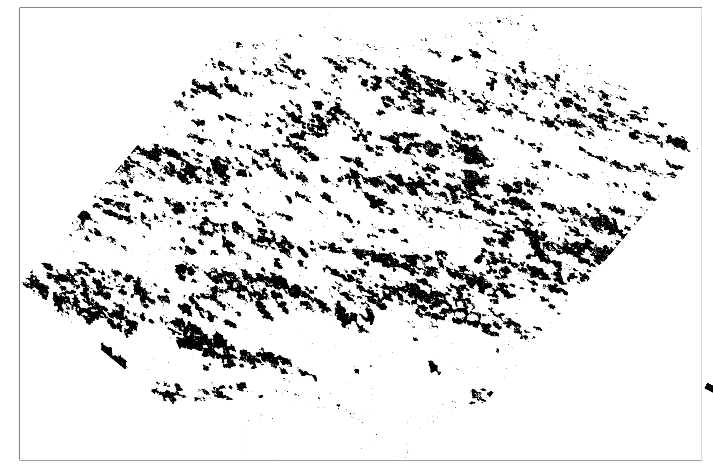
500 µm

Procedure

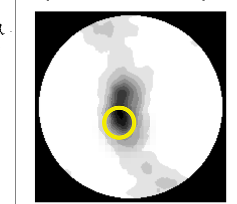
1. convert EBSD map to c-axis azimuth and inclination; non-indexed pixels yield mask
2. calculate CIP orientation images (COI), misorientation images (MOI), and orientation gradient images (OGI)
3. use OGI (average of 8 neighbors) ('orientation Sobel')
4. use Image SXM / Lazy Grain boundaries to prepare grain map
5. use Image SXM to measure cross sectional areas of grains
6. use Kaleidagraph to calculate diameter of area equivalent circles
7. use stripstar to obtain volume-weighted histogram of volume equivalent spheres - mode of this histogram = piezometer grain size

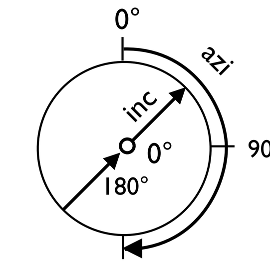
Grain size determinations in Y domain(s)

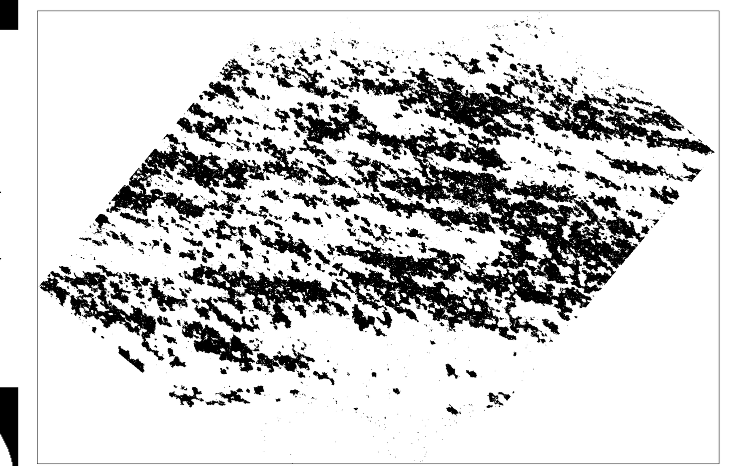

w935.MISr2_141_167

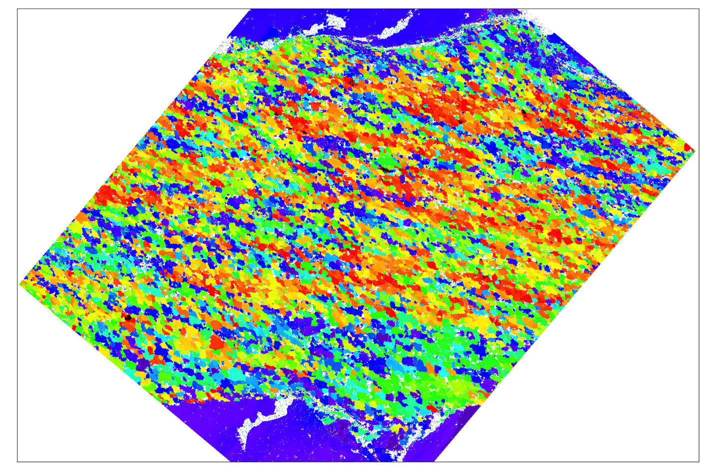

w935.MISr2_141_167-th15

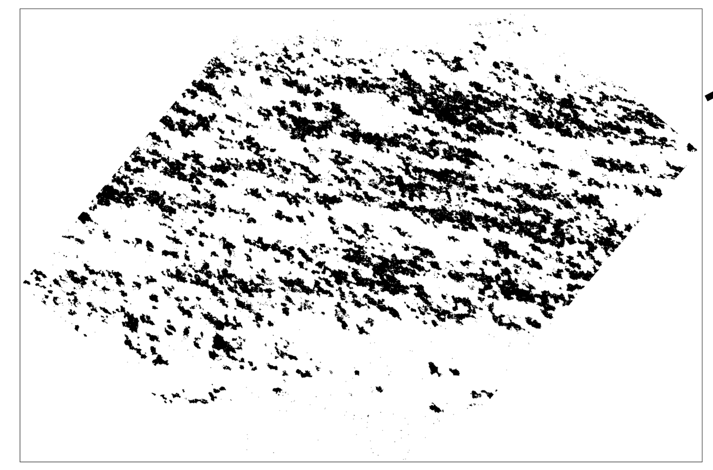
max.1 (141°,167°)

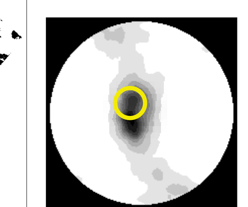




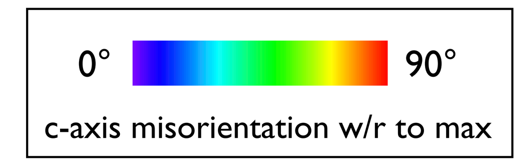

w935.MISr1_MISr2_ combined


w935.MISr1_052_169


w935.MISr1_052_169-th15



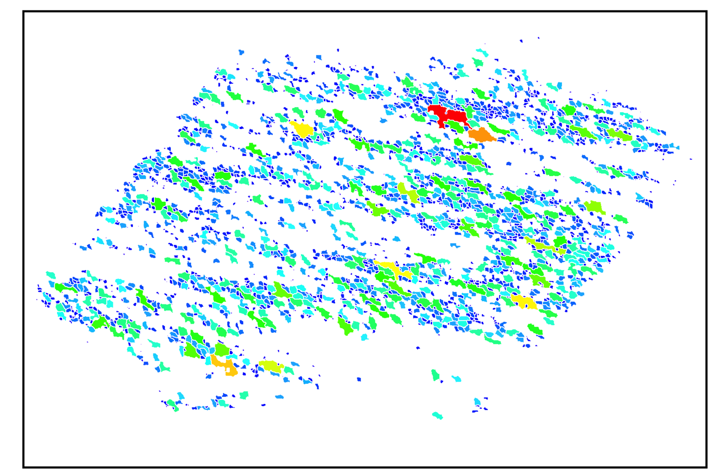
max.2 (52°,169°)

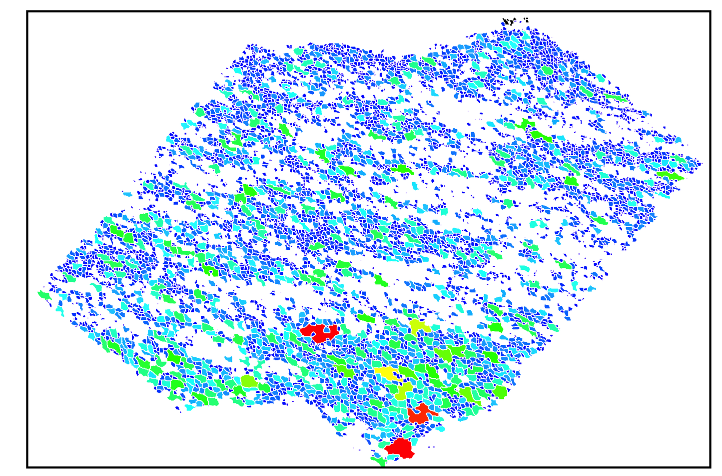


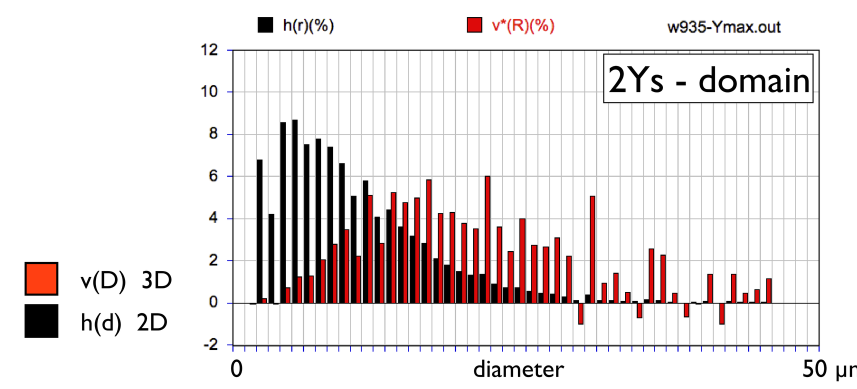
Procedure

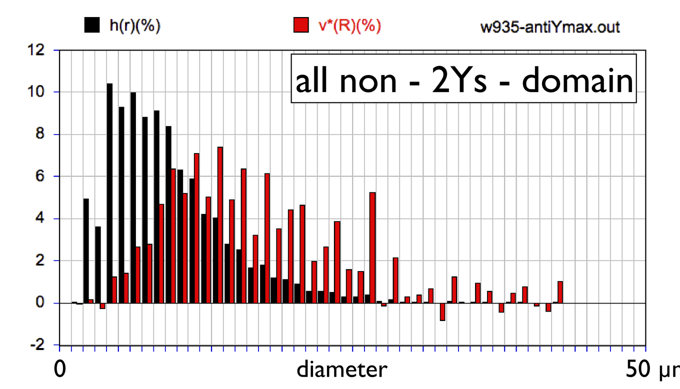
1. use misorientation images w/r to Y maxima (pole figure)
   w935.MISr2_141_167
   w935.MISr1_052_169
2. threshold at GV = 15° = 15 ° = 30° opening angle of cone
   w935.MISr2_141_167-th15
   w935.MISr1_052_169-th15
3. merge both subdomains
   w935.MISr1_MISr2_ combined
4. grain map is separated into '2Ys' domain and 'anti2Ys' domain
5. use Image SXM to measure cross sectional areas of grains in '2Ys' domain and 'anti2Ys' domain
6. use Kaleidagraph to calculate diameter of area equivalent circles
7. use stripstar to obtain volume-weighted histogram of volume equivalent spheres - mode of this histogram = piezometer grain size

NOTE
EBSD analysis reveals that the so-called 'Y-max' (Heilbronner & Tullis 2006) is composed of 2 distinct maxima - therefore, the 'Y domain' has to be merged from these two sub domains.


w935.TH200.areas-gsmap-2Ys

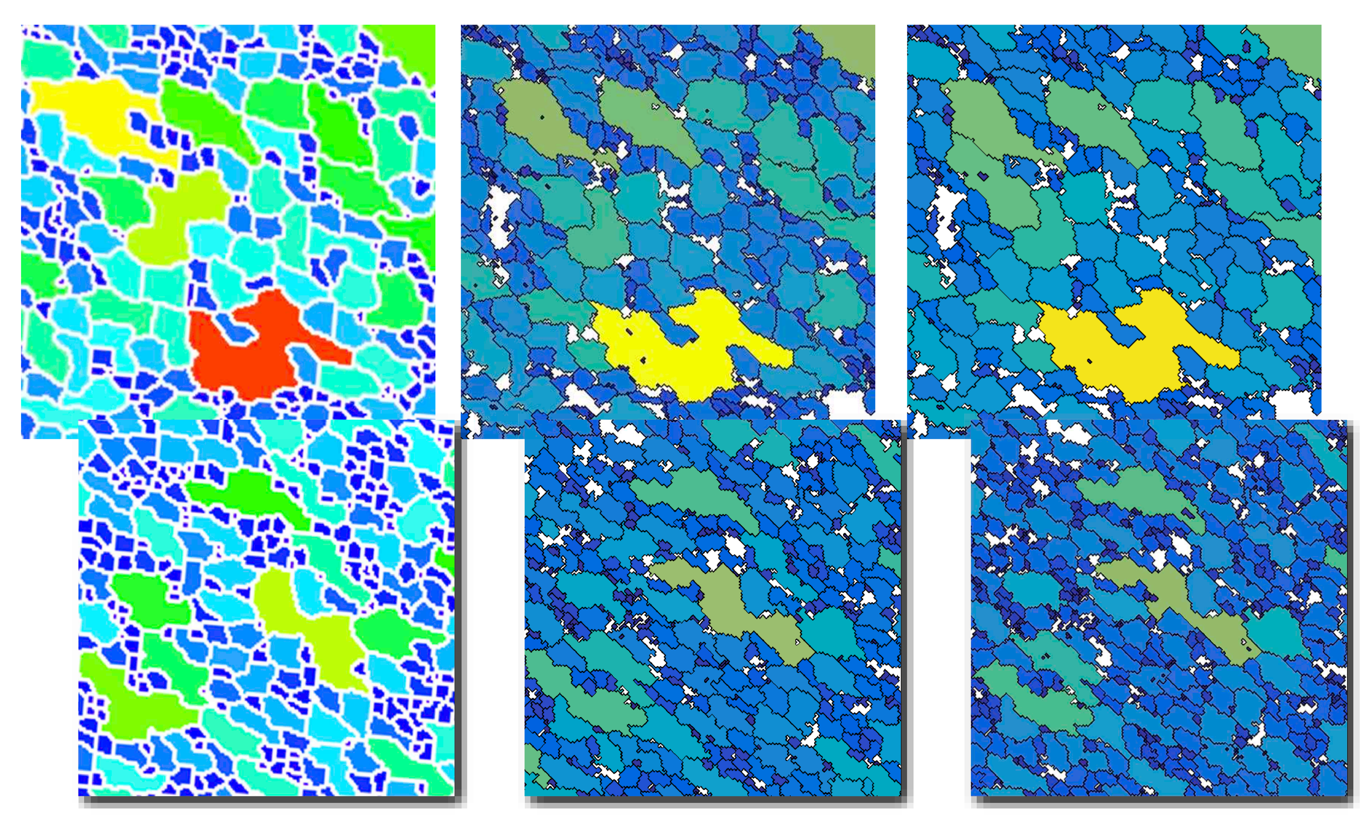

w935.TH200.areas-gsmap-anti2Ys





Segmentation based on c-axis versus full crystal orientation

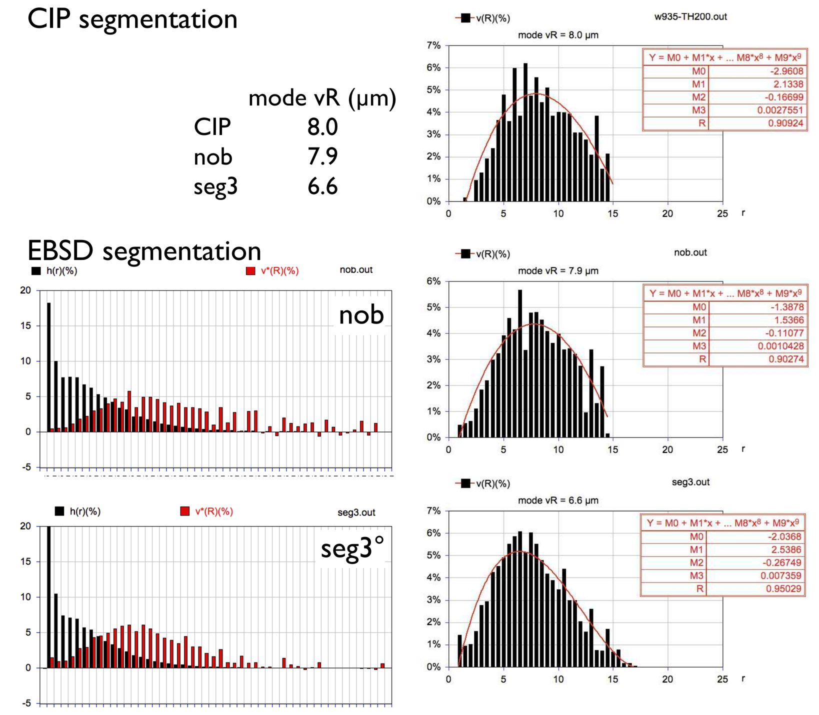
CIPseg > 1° EDG8a & tji — seg >6° misorientation — seg >3° misorientation



CIP segmentation

	mode vR (µm)
CIP	8.0
nob	7.9
seg3	6.6

EBSD segmentation

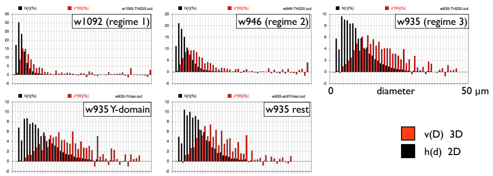


COMMENT:
Setting different thresholds for grain boundary - subgrain boundary angles does not appreciably influence segmentation / grain size
(More on companion poster BHQ revisited (2))

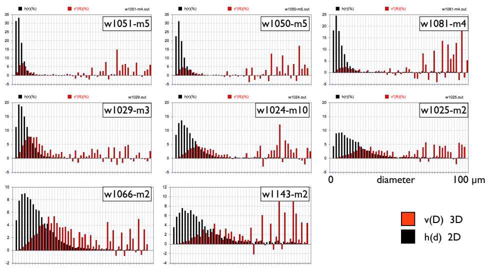
Renée Heilbronner, Tromsø University (renee.heilbronner@unibas.ch)
Rüdiger Kilian, Basel University (ruediger.kilian@unibas.ch)
Jan Tullis, Brown University (jan_tullis@brown.edu)

Comparison of shear grain sizes with piezometer

2D h(d) → stripstar → 3D v%(D)

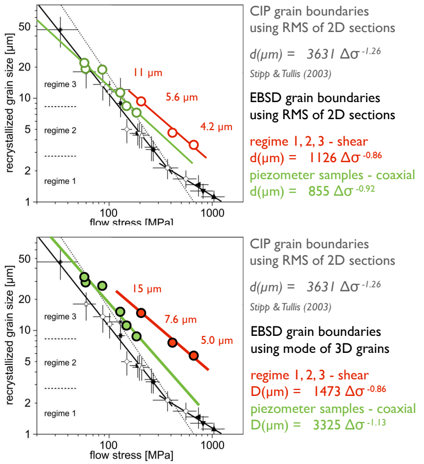


		CIP	EBSD			
	2τ(MPa)	JGR 2006	stripstar 2D-3D - Kaleidagraph statistics & mode poly-fit 3rd			
sample		mode v%(D)	mean h(d)	median h(d)	RMS h(0<d<50µm)	mode v%(D)
w1092	680		3.5	3.1	4.3	5.0
w946	420		4.7	3.9	5.6	7.6
w935	210	15 ± 10	9.7	8.5	11.3	16.0
		from 1/gbd				
w935 Ymax	210	19	10.3	8.9	12.1	17.6
w935 antiYmax	210	12	9.3	8.3	10.6	13.8

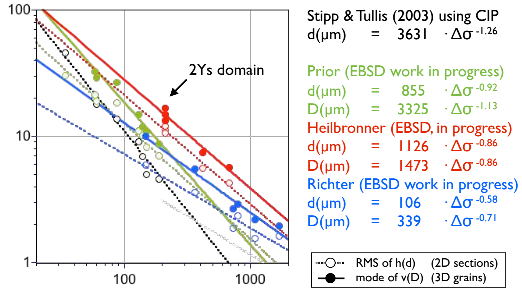


		CIP	EBSD		
	Δσ(MPa)	JGR 2003 piezo	Kaleidagraph statistics	stripstar 2D-3D - MATLAB non-param fit	
sample		RMS h(d)	RMS h(0<d<50µm)	bandwidth (µm)	mode v%(D)
w1051-m5	189	4.6	7.0	3.0	8.6
w1081-m5	139	6.9	5.7	3.0	9.4
w1050-m5	149	5.0	8.2	3.0	10.8
w1081-m4	139	6.9	10.2	2.0	11.8
w1029-m3	130	9.0	11.1	3.0	15.0
w1024-m10	102	11.6	14.1	3.0	19.9
w1025-m2	87	13.6	18.5	3.0	26.6
w1066-m2	60	18.0	19.1	3.0	29.1
w1143-m2	58	19.9	21.3	3.0	32.4

EBSD data on CIP piezometer



BHQ piezometer - revisited



Preliminary conclusions

For better resolution, the CIP derived grain size distributions of experimentally deformed BHQ are being re-measured using EBSD maps of the same samples. Samples are from general shear experiments (Heilbronner & Tullis, 2002, 2006) and coaxial experiments (Stipp & Tullis, 2003), EBSD maps by Dave Prior.

- The c-axis pole figures clearly showed that there are 2 basal and 2 Y maxima symmetrically disposed about the periphery and the center of the pole figure, respectively - in the 2006 paper, such double maxima were suspected to be artefacts of the CIP method and not discussed any further.

- EBSD measurements confirm that the recrystallized grain size (of regime 3) depends on texture: the Y domains yield larger grains, i.e., they deform under lower flow stress than the rest of the sample.

- The recrystallized grain size also depends on the type of deformation: rotational versus irrotational. Samples deformed in general shear yield larger grain size / higher flow stress than predicted by the piezometer. Note, however, that the strain in the coaxial experiments is much lower and hence the fraction of recrystallized grains much smaller than in the shear experiments.

For comparison with the piezometer, all grain sizes are calculated both as RMS of the 2D sections and as the mode of the volume weighted histogram of 3D grains. The latter measure is preferred - it is statistically more stable and physically more meaningful. However it has no influence on the above findings.

Abstract

BHQ revisited (1) - Looking at grain size

Renée Heilbronner (1,2), Rüdiger Kilian (1), and Jan Tullis (3)

(1) Geological Institute, Basel University, Switzerland (renee.heilbronner@unibas.ch), (2) Department of Geology, Tromsø University, Norway, (3) Department of Earth, Environmental and Planetary Sceinces, Brown University, U.S.A.

Black Hills Quartzite (BHQ) has been used extensively in experimental rock deformation for numerous studies. Coaxial and general shear experiments have been carried out, for example, to define the dislocation creep regimes of quartz (Hirth & Tullis, 1992), to determine the effect of annealing (Heilbronner & Tullis, 2002) or to study the development of texture and microstructure with strain (Heilbronner & Tullis, 2006). BHQ was also used to determine the widely used quartz piezometer by Stipp & Tullis (2003).

Among the microstructure analyses that were performed in those original papers, grain size was usually determined using CIP misorientation images. However, the CIP method (= computer-integrated polarization microscopy, details in Heilbronner and Barrett, 2014) is only capable of detecting the c-axis orientation of optically uniaxial materials and hence is only capable of detecting grain boundaries between grains that differ in c-axis orientation.

One of the puzzling results we found (Heilbronner & Tullis, 2006) was that the recrystallized grain size seemed to depend on the crystallographic preferred orientation of the domain. In other words the grain size did not only depend on the flow stress but also on the orientation of the c-axis w/r to the shear direction. At the time, no EBSD analysis (electron back scatter diffraction) was carried out and hence the full crystallographic orientation was not known. In principle it is therefore possible that we missed some grain boundaries (between grains with parallel c-axes) and miscalculated our grain sizes.

In the context of recent shear experiments on quartz gouge at the brittle-viscous transition (see Richter et al., this conference), where EBSD is used to measure the recrystallized grain size, we wanted to re-measure the CIP grain sizes of our 2006 samples (deformed in regime 1, 2 and 3 of dislocation) in exactly the same way. In two companion posters we use EBSD orientation imaging to repeat, refine and expand the microstructure and texture analysis of Heilbronner & Tullis (2006). Here, in poster (1), we focus on the recrystallized grain size with the aim of (a) comparing CIP- and EBSD derived grain size measurements, (b) of comparing the recrystallized grain size of coaxially deformed and sheared BHQ and (c) in order to confirm that the quartz piezometer indeed depends on texture, and (d) to test if it also depends on the type of deformation (irrotational versus rotational deformation).

References cited:
Heilbronner, R., and S.D. Barrett (2014) Image Analysis in Earth Sciences, Springer.
Heilbronner, R., and J. Tullis (2002), The effect of static annealing on micro- structure and crystallographic preferred orientations of quartzites experimentally deformed in axial compression and shear, Geol. Soc. Spec. Publ., 200, 191 – 218.
Heilbronner, R., and J. Tullis (2006), Evolution of c axis pole figures and grain size during dynamic recrystalliza- tion: Results from experimentally sheared quartzite. JGR, 111, B10202, doi: 10.1029/2005JB004194, 2006
Hirth, G., and J. Tullis (1992), Dislocation creep regimes in quartz aggregates, JSG, 14, 145–159.
Stipp, M., and J. Tullis (2003), The recrystallized grain size piezometer for quartz, Geophys. Res. Lett., 30(21), 2088, doi:10.1029/2003GL018444.