

my piezometer is stronger than yours ... !

EGU2019-10088

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What's the problem ?

Grain sizes determined from general shear experiments on BHQ do not coincide with the piezometer for published by Stipp and Tullis (2003).

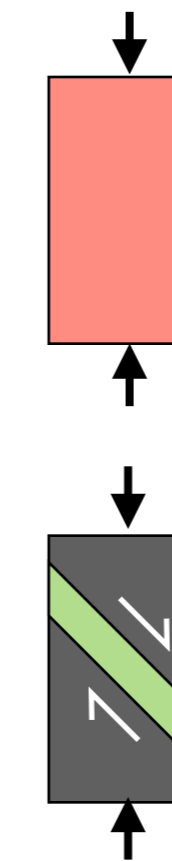
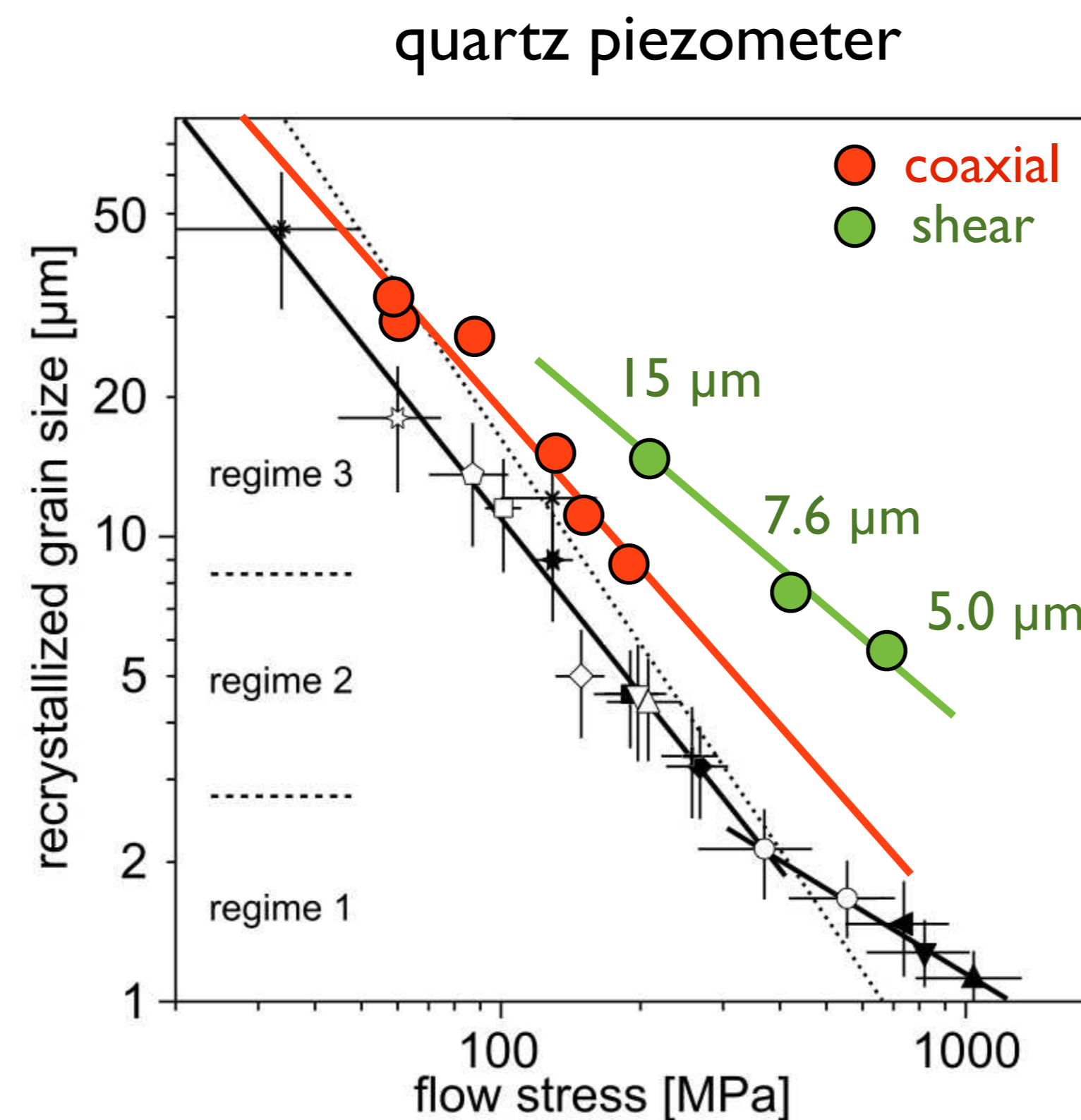
Possible explanation ?

The piezometer depends on the mode of deformation. In simple shear, higher stress are required to achieve a given recrystallized grain size than in pure shear. Deformation in simple shear requires more work than in pure shear.

Great !

... but before we get excited, let's check:

1. Did we measure the grain sizes correctly, especially the very small ones ?
2. What about the difference in confining medium: solid salt in standard experiments versus molten salt for the piezometer ?
3. How do we convert the mechanical data to stress-strain curves ? Are our conversions consistent ?
4. How do we best convert τ of the shearing experiments to $\Delta\sigma$ of coaxial experiments ?
5. ... and, in case Brian Evans is listening in,
... are we sure piezometers work at all ??



published piezometer
 $d(\mu\text{m}) = 3631 \Delta\sigma^{-1.26}$
 recalculated as mode of 3D grains
 $D(\mu\text{m}) = 3325 \Delta\sigma^{-1.13}$
 mode of 3D grains
 $D(\mu\text{m}) = 1473 \Delta\sigma^{-0.86}$

What have we done to solve the problem ?

1. The grain sizes of shear and coaxial experiments and those of the piezometer experiments have been repeated, using EBSD data...
2. The grain sizes of a set of standard coax experiments (solid salt assembly) was measured.
3. The existing software for the conversion of the mechanical data (rigP, rigC, and rigS) has been re-written with the aim of making every step fully transparent. Choices concerning hitpoint definition, area correction, etc. now have to be made explicitly.
4. The run records of coaxial and shear experiments were re-analyzed demonstrating a large effect of the options on the calculated stress levels.
5. ... and keep thinking about piezometers ...

And now ?

Was the discrepancy between the shearing and the coaxial piezometer only an artefact of the experimental set-up ?
 In other words, does the published piezometer still hold for coaxial and shearing situations, i.e., for pure shear and simple shear ?
 Or does the discrepancy between shearing and coaxial remain ?

Find the answers on my PICO !

overview

go to

Title page

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Introduction to the problem

Solving the problem

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1. Repeating the grains sizes measurements using EBSD data....

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2. Introducing new software for the conversion of the mechanical data.

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b. rigC converting coaxial experiments

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c. rigS converting general shear experiments

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Getting new results

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My piezometer is stronger than yours ...

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Recent studies (e.g., Heilbronner & Kilian, 2017; Richter, 2016) indicate that, for general shearing experiments, the quartz piezometer (Stipp & Tullis, 2003) does not correctly predict the recrystallized grain size (from the measured flow stress) or the flow stress (from the measured grain size). One may speculate whether there is an inherent strength difference between simple shear and pure shear deformation, which would then require the calibration of a second piezometer. However, before considering this possibility, it is necessary to ensure that the differential stresses and strains of the coaxial and general shearing experiments are correctly determined.

In this presentation, the focus is on Grigg's type solid medium deformation apparatuses, the general conclusions, however, may apply to other machines and other experimental set-ups too. The major concerns are: (1) How does the force applied externally to the loading piston, in combination with the axially compressed, solid confining medium, translate to the state of stress that exists inside the sample? (2) How much of the sample is homogeneously deforming and how is the strain and the strain rate best quantified?

Coaxial and general shearing experiments carried out in the dislocation creep regimes 1, 2, and 3 (as defined by Hirth & Tullis, 1992) are used to show how the stresses and strains derived from the force-displacement record depend on the choice of mechanical and geometrical corrections. Together with the less than 100% reproducibility of the Grigg's apparatus, the different corrections may lead to a rather large range of results for one and the same experiment, as will be demonstrated. Such discrepancies need to be considered when comparing coaxial and shearing experiments, or when comparing different results from different labs.

With constantly improved machine design, more and more highly resolved data can be retrieved during the experiments. To make full use of these improvements, experimentalists are urged to carefully check the choices made by the software they use (or better still, to write their own software) and to be explicit about the corrections they apply when publishing the resulting stress-strain data. - As the list of calibrations and conversions presented in this PICO is probably not complete, participants of the conference are invited to contribute.

- Heilbronner, R. & Kilian, R. (2017). The grain size(s) of Black Hills Quartzite deformed in the dislocation creep regime. *Solid Earth*.
- Hirth, G. & Tullis, J. (1992). Dislocation creep regimes in quartz aggregates. *Journal of Structural Geology* 14, 145±159.
- Richter, B. (2016). The brittle-to-viscous transition in experimentally deformed quartz gouge, Basel University PhD thesis.
- Stipp, M., and J. Tullis (2003). The recrystallized grain size piezometer for quartz, *Geophys. Res. Lett.*, 30(21), 2088, doi:10.1029/2003GL018444.

previously on '... the piezometer ...'

2002 coaxial & shearing experiments confining medium = solid salt

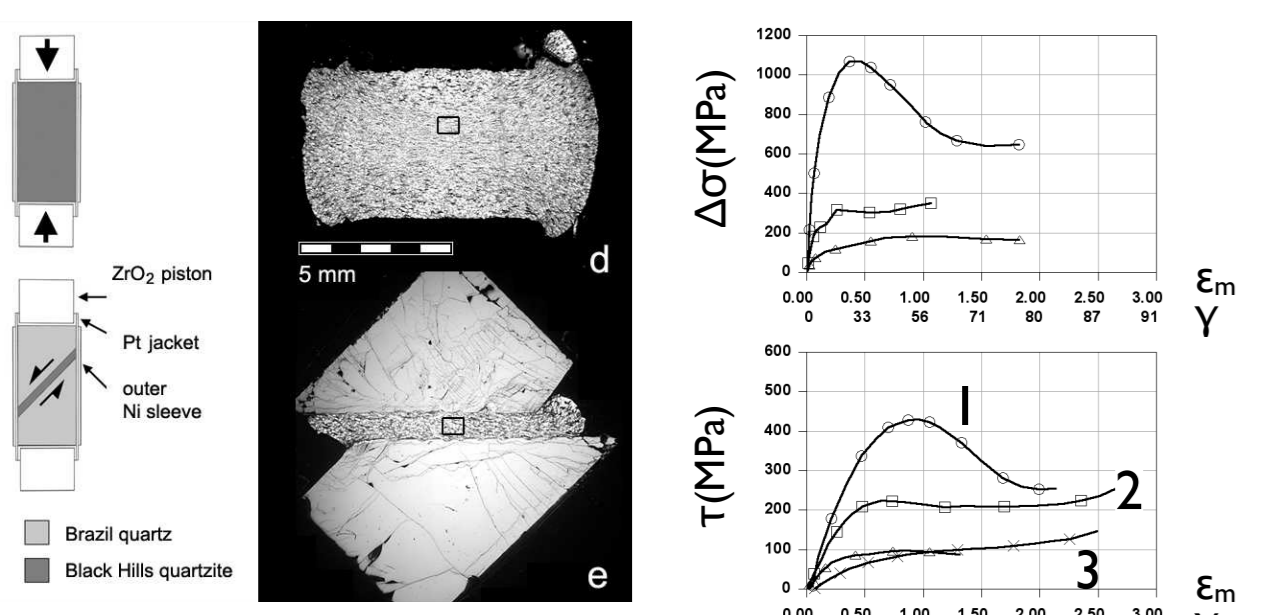


Table 1. Experimental conditions

Regime	Deformed sample #	Confining pressure (GPa)	T (°C)	H ₂ O (wt%)	Axial strain rate (s ⁻¹)	Max. short strain (%)	Max. strain mag.	Flow stress Δσ (MPa) ¹	Annealing T (°C)	Annealing t (h)
1	w871	1.5	850	0	1.5 × 10 ⁻³	77	1.82	650	850	w871
2	w872	1.5	900	0.17	1.5 × 10 ⁻³	58	1.87	710	900	w872
3	w873	1.5	900	0.17	1.5 × 10 ⁻³	78	1.84	180	900	w873

Table 2. Results of analyses

Sample # (regime)	Vol % recryst.	Vol % annealed	Mode grain diameter (μm)	CPO max. density (bulk texture)	CPO max. density of recryst. fraction ²	Mode of orient. gradient distrib. (°)	Measured perimeter/perimeter of equivalent circle	PARIS factor (%)	Grain boundary surface per volume (μm ⁻¹)
w871 (1)	50	5	3.76	3.44	1.80	33.3	1.08	33.3	0.99
w872 (2)	40	7	4.12	3.96	1.79	17.9	1.87	14.4	0.53
w873 (3)	85	30	4.48	2.57	1.87	14.4	0.53	1.53	0.2

Heilbronner, R. and Tullis, J. (2002). The effect of static annealing on microstructure and crystallographic preferred orientations of quartzites experimentally deformed in axial compression and shear. In: S. de Meer, M.R. Drury, J.H.P. de Bresser and G.M. Pennock (Editors), *Deformation Mechanisms, Rheology and Tectonics: Current Status and Future Perspectives*. Geological Society, London, Special Publication, pp. 191-218.

2003 (coaxial) piezometer experiments confining medium = molten salt

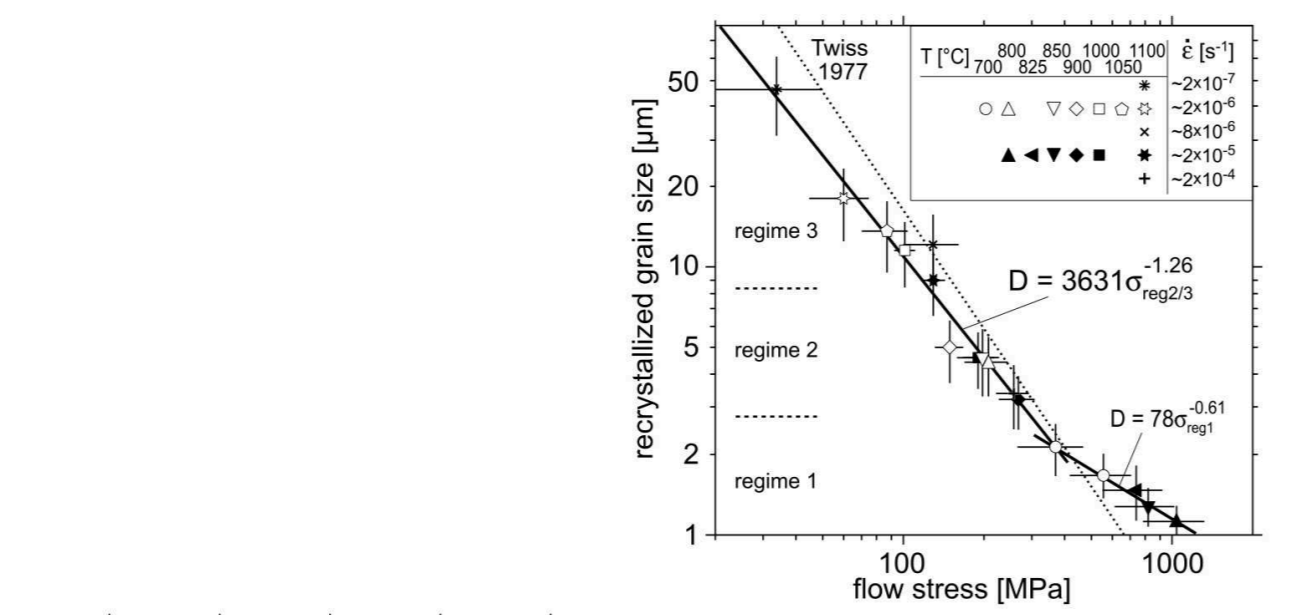


Figure 2. Stress/strain data and sample numbers for the MSC experiments on BHQ; friction corrections are described in the text. Flow stress values in Table 1 are averaged from 10% strain until the end of each run.

Table 1. Experimental Deformation Conditions and Recrystallized Grain Size Data

sample	temperature [°C]	strain rate [s ⁻¹]	axial strain [%]	flow stress [MPa]	recrystallized grain size [μm]	number of grains
W-1126 - β	1100	2.1 - 2.3 × 10 ⁻³	17	34 ± 16	46 ± 15	734
W-1116 - β	1100	2.1 - 2.3 × 10 ⁻³	21	34 ± 16	18.0 ± 5.5	676
W-1096 - β	1100	2.0 - 2.5 × 10 ⁻³	31	60 ± 15	12.1 ± 3.6	804
W-1022 - β	1100	0.8 - 1.0 × 10 ⁻³	30	130 ± 30	9.0 ± 2.4	876
W-1029 - β	1100	2.1 - 2.8 × 10 ⁻³	46	257 ± 35	3.4 ± 0.9	931

1 and 3 refer to quartz stability field. Repeated runs for microstructure and mechanical data are listed within the same row. The instantaneous strain rate increase over the interval from 10% to final strain is indicated. The number of recrystallized grains measured in each sample is given. For sample W-739 from Bishop (1996) the recrystallized grain size was re-measured. See text for further explanations.

Stipp, M., and J. Tullis (2003). The recrystallized grain size piezometer for quartz. *Geophys. Res. Lett.*, 30(21), 2088, doi: 10.1029/2003GL018444.

2006 shearing experiments confining medium = solid salt

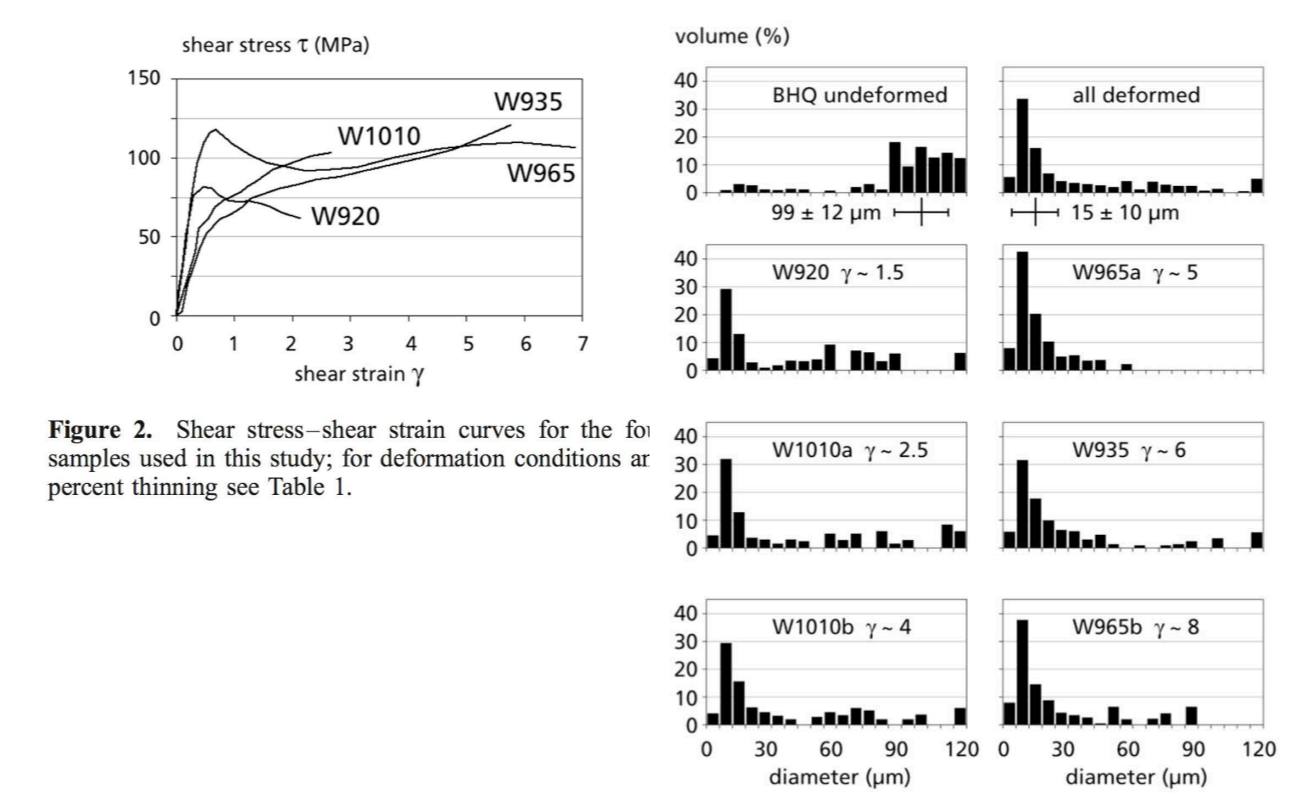


Figure 2. Shear stress-shear strain curves for the samples used in this study; for deformation conditions at percent thinning see Table 1.

Figure 5. Grain size distribution of undeformed Black Hills quartzite (BHQ) and three experimentally sheared samples. Volume percent of 3-D grains as a function of the diameter of a sphere of the same size. Undeformed BHQ, summary of all deformed samples; W920 (γ ≈ 1.5); W1010a (γ ≈ 2.5); W1010b (γ ≈ 4); W965a (γ ≈ 5); W935 (γ ≈ 6); and W965b (γ ≈ 8). Average grain diameters of undeformed BHQ and recrystallized grains have been determined for grain sizes >60 and <48 μm, respectively.

Table 1. Experimental Conditions^a

Experiment	T, °C	P _c , GPa	Thickness, mm	γ̇, s ⁻¹	ε _{app} , %	γ _{tot}	t _{exp} , min	t, s
W920	900	1.50	2.03	1.25 × 10 ⁻³	20	2.1	1.3	1.68 × 10 ⁻³
W1010	915	1.55	1.27	1.98 × 10 ⁻³	19	2.7	1.6	1.28 × 10 ⁻³
W935	915	1.50	1.27	1.98 × 10 ⁻³	46	5.8	2.5	2.02 × 10 ⁻³
W965	915	1.55	1.25	2.01 × 10 ⁻³	40	6.9	2.8	2.41 × 10 ⁻³

^aValues are for T, temperature; P_c, confining pressure; thickness, original thickness of sample; γ̇, shear strain rate; ε_{app}, thinning strain (percent of original thickness); γ_{tot}, total bulk shear strain; t_{exp}, duration of experiment. All samples had 0.17 wt % H₂O added.

Heilbronner, R. and Tullis, J. (2006). Evolution of c-axis pole figures and grain size during dynamic recrystallization: Results from experimentally sheared quartzite. *J. Geophys. Res.*, 111: B10202, doi:10.1029/2005JB004194.

2017 shearing experiments, revisited, EBSD data

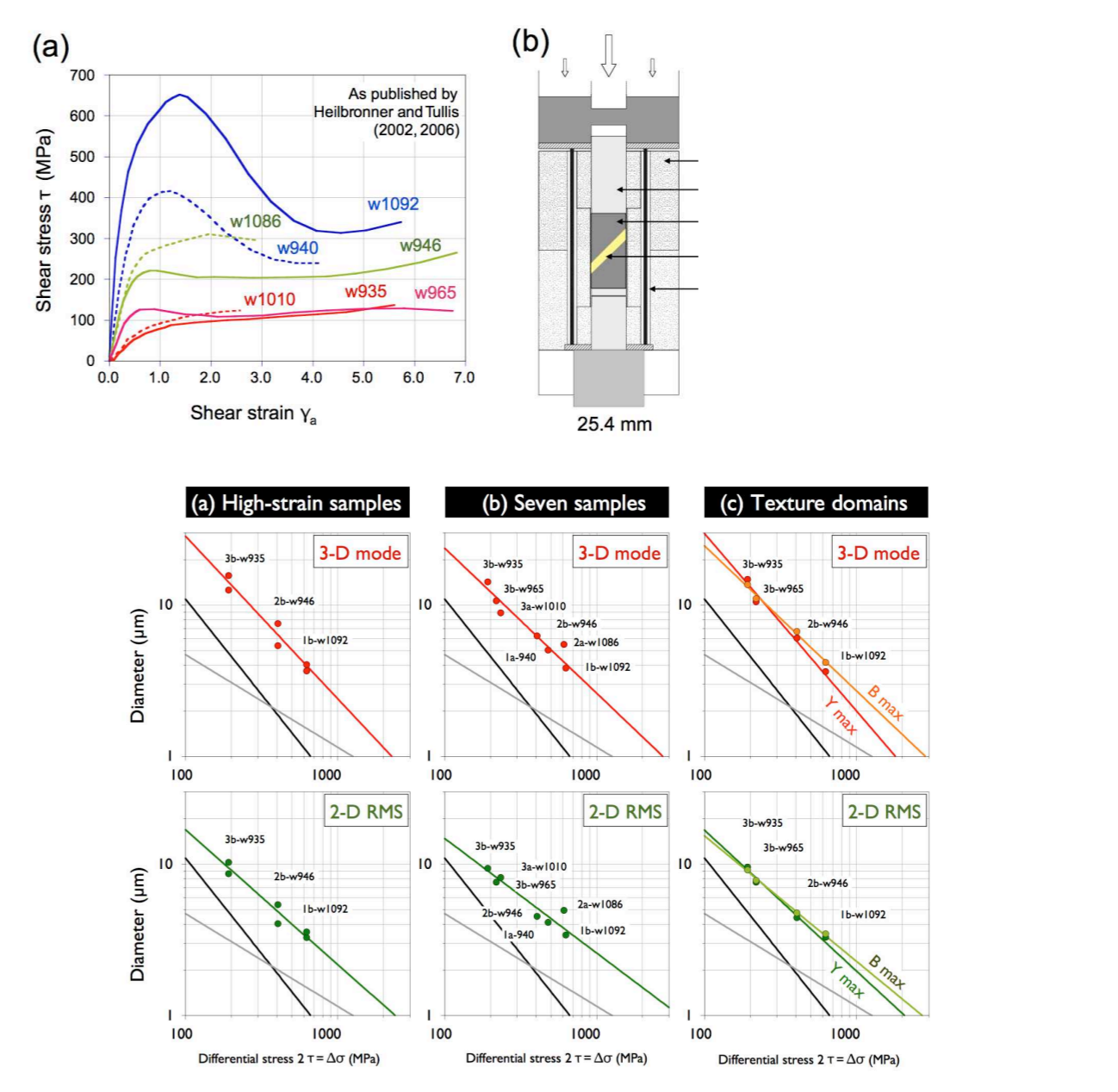


Table 1. Mechanical data for general shear experiments of Black Hills Quartzite.

Regime	Sample	T [°C]	γ̇ [10 ⁻³ s ⁻¹]	P [GPa]	H ₂ O [wt%]	Flow stress [MPa]	Δσ [MPa]	45° d. [mm]	th ₀ [mm]	th _f [mm]	γ _{tot} [%]	γ _{app} [%]	γ _{tot} [°]
1a	w940	850	1.46	2.5	1.5	413	238	476	3.6	1.27	0.87	4.1	2.8
1b	w1002	850	0.5	2.43	1.55	647	314	338	5.05	1.45	0.88	5.7	3.5
2a	w1086	875	1.33	2.6	1.58	269	300	294	6.00	2.49	1.14	0.87	2.2

Table 1. List of Samples Together With Differential Stress and Grain Size (From Stipp and Tullis, 2003; Stipp et al., 2006) Together With EBSD Step Size, Area, Indexing Rate, and Statistics Extracted From EBSD Data^a

Sample	Stress (MPa)	Step Size (μm)	Area (mm ²)	Raw Indexing Rate (%)	Total No. of Grains	No. of Relict Grains	No. of Recr. Grains	Stipp and Tullis d (μm)	EBSD RMS d (μm)	EBSD Arithmetic d (μm)	EBSD Geometric d (μm)	EBSD Median d (μm)	EBSD Mode d (μm)	EBSD Error (μm)
W1126	34 ± 16	1 μm	3.61	97.1	1064	236	828	46 ± 15	61.0	53.5	45.5	49.0	5.16	29.5
W1143	58 ± 18	1 μm	1.40	97.9	1615	255	1360	19.9 ± 4.9	27.5	22.2	18.0	18.1	4.48	16.2
W1066	60 ± 15	1 μm	0.096	98.3	99	25	74	26.1	21.0	17.0	17.5	2.82	15.7	
W1025	87 ± 17	1 μm	2.70	97.3	5973	1201	4772	18 ± 5.5	18.2	15.9	13.6	14.2	3.39	8.82
W1024	102 ± 9	1 μm	1.28	96.4	3832	769	3063	11.6 ± 3.2	12.6	11.0	9.55	9.84	3.04	6.10
W1029	130 ± 13	1 μm	1.12	96.8	396	115	281	10.8	9.64	8.54	8.77	1.94	4.94	
W1081	139 ± 24	1 μm	1.20	96.0	7491	1328	6163	9.0 ± 2.4	9.83	8.70	7.67	7.74	2.88	4.58
W1050	149 ± 18	1 μm	0.894	93.7	6104	762	5342	5.0 ± 1.3	6.09	5.32	4.81	4.58	2.52	2.97
W1051	189 ± 30	1 μm	0.086	91.9	943	234	709	4.6 ± 1.1	4.63	4.14	3.71	3.65	0.492	2.07
W1051	189 ± 30	1 μm	0.905	85.0	8641	1114	7527	4.6 ± 1.1	5.03	4.61	4.32	4.13	2.28	2.01

^aMeasurement errors in differential stress and grain size (1 standard deviation) are indicated. ^bEBSD data used to define the sliding resolution piezometer.

Cross, A.J., Prior, D.J., Stipp, M., Kidder, S. (2017). The recrystallized grain size piezometer for quartz: An EBSD-based calibration. *Geophys. Res. Lett.*, 44, doi:10.1002/2017GL073836.

2017 piezometer experiments, revisited, EBSD data

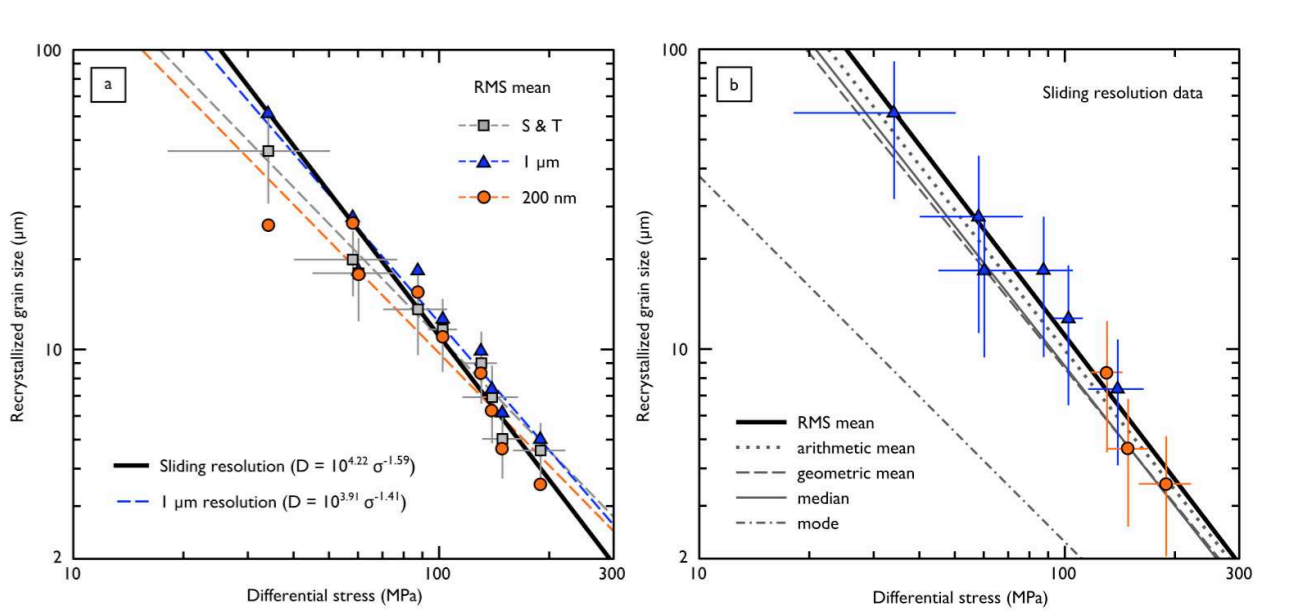
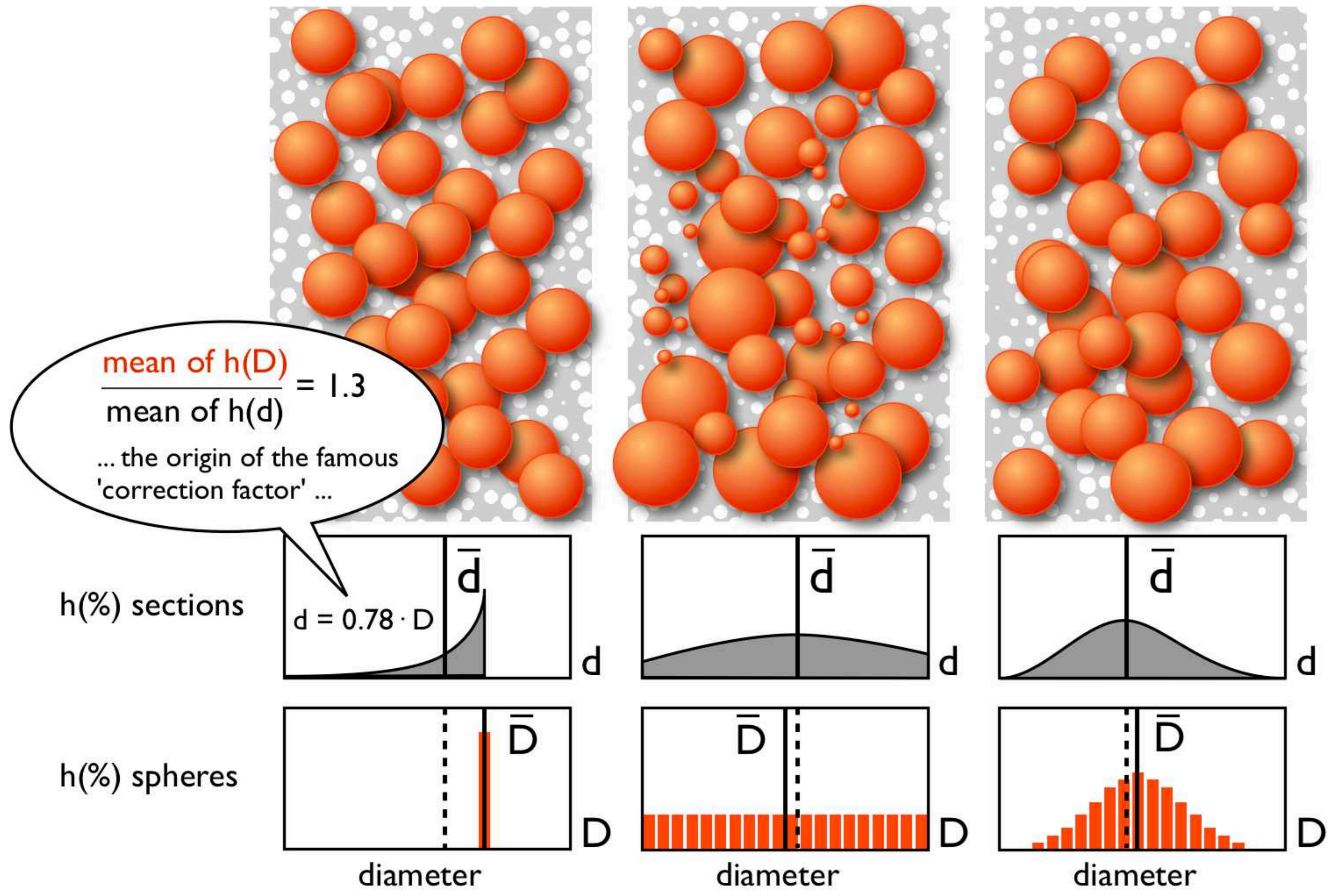


Figure 4. Log-log plots of recrystallized grain size versus differential stress. (a) The published RMS recrystallized grain sizes measured by CIP (Stipp and Tullis, 2003) (dashed grey line), and the RMS mean grain sizes for EBSD defined recrystallized grains extracted from 1 μm (dashed blue) and 200 nm (dashed orange) step size EBSD data. The sliding resolution RMS piezometer, which incorporates data from the 200 nm maps at the highest stresses, is shown in black. (b) The sliding resolution piezometer calculated from RMS, arithmetic and geometric means, and the median and mode data (equations for all are given in the supporting information Table S1). Error bars are shown for the Stipp and Tullis CIP data (Figure 4a) and the sliding resolution EBSD data (Figure 4b).

Cross, A.J., Prior, D.J., Stipp, M., Kidder, S. (2017). The recrystallized grain size piezometer for quartz: An EBSD-based calibration. *Geophys. Res. Lett.*, 44, doi:10.1002/2017GL073836.

which mean grain size ?



Two reasons for using 3D modes of volume fractions:

'2D mean'

The mean (or RMS) of the size distribution of 2D sections depends strongly on the shape of the distribution $h(d)$; depending on the skewness, the mode may be smaller, larger or equal to the mean.

This is the measure used by Stipp & Tullis (2003) and by Cross et al. (2017) to defined the recrystallized grain size of the piezometer.

'3D mode'

Physically, the most important grain size is the mode of the volume-weighted size distribution of 3D spheres $vol\%(D)$; this is the grain size that occupies the largest volume fraction. This is the measure I propose to use instead.

Not convinced ?

More on tomorrows short course SCI.37

SC1.37

[Grain size analysis - 2D, 3D and fractal](#)

Co-organized as CR3.14/EMRP1.92/GMPV7.19/TS13.1

Convener: Renée Heilbronner | Co-convener: Rüdiger Kilian

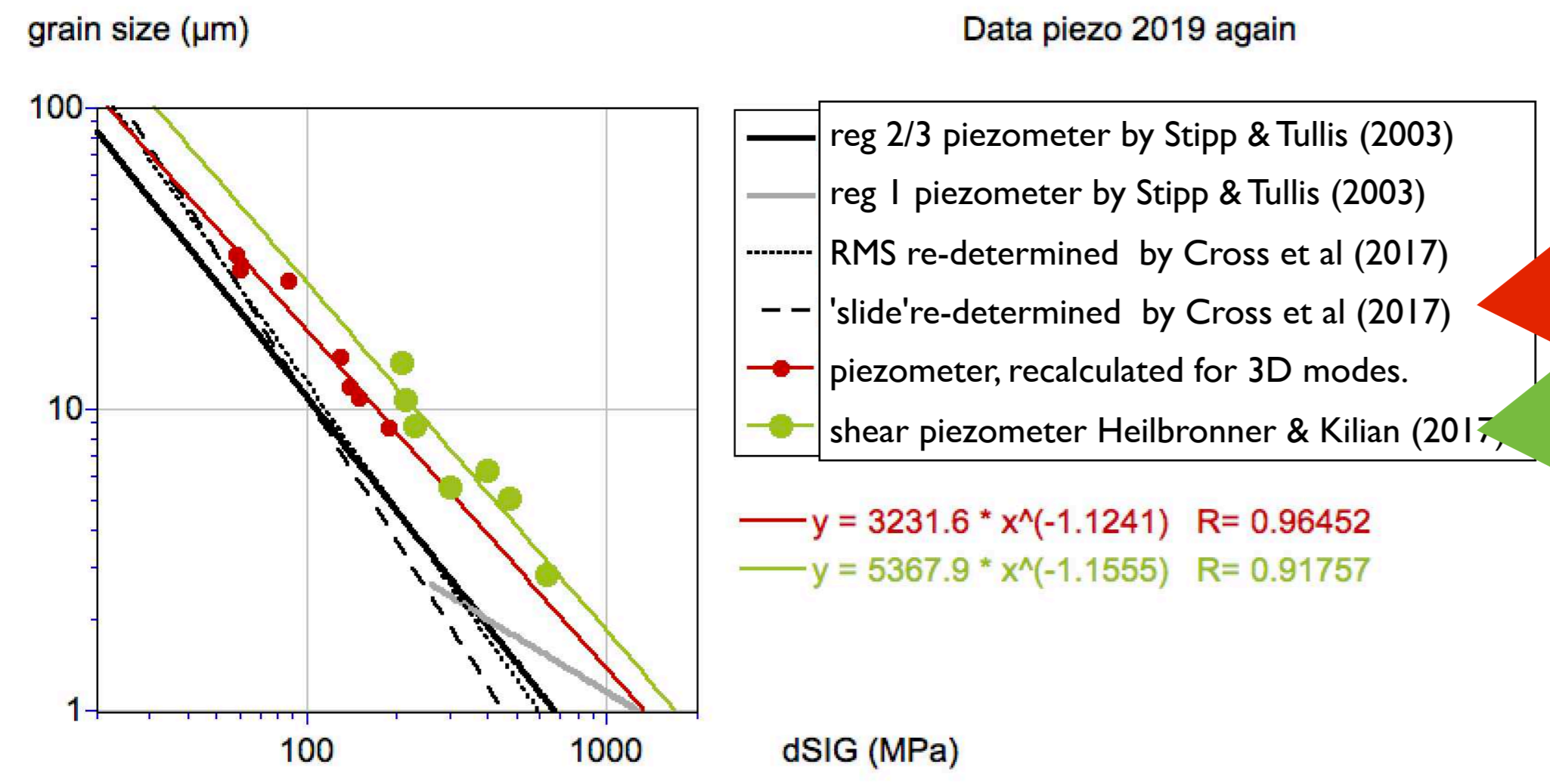
★ Thu, 11 Apr, 10:45-12:30 Room -2.31

check grain size shear SSA vs. piezometer MSA (reg 2/3)

As a first step, the grain sizes of the piezometer experiments (Stipp & Tullis, 2013) and those of the general shear experiments (Heilbronner & Kilian, 2017) are plotted.

In order to compare the 3D modes of the shear experiments (green), the 3D modes of a set of EBSD maps of the piezometer samples (courtesy Stipp & Prior) are calculated (red).

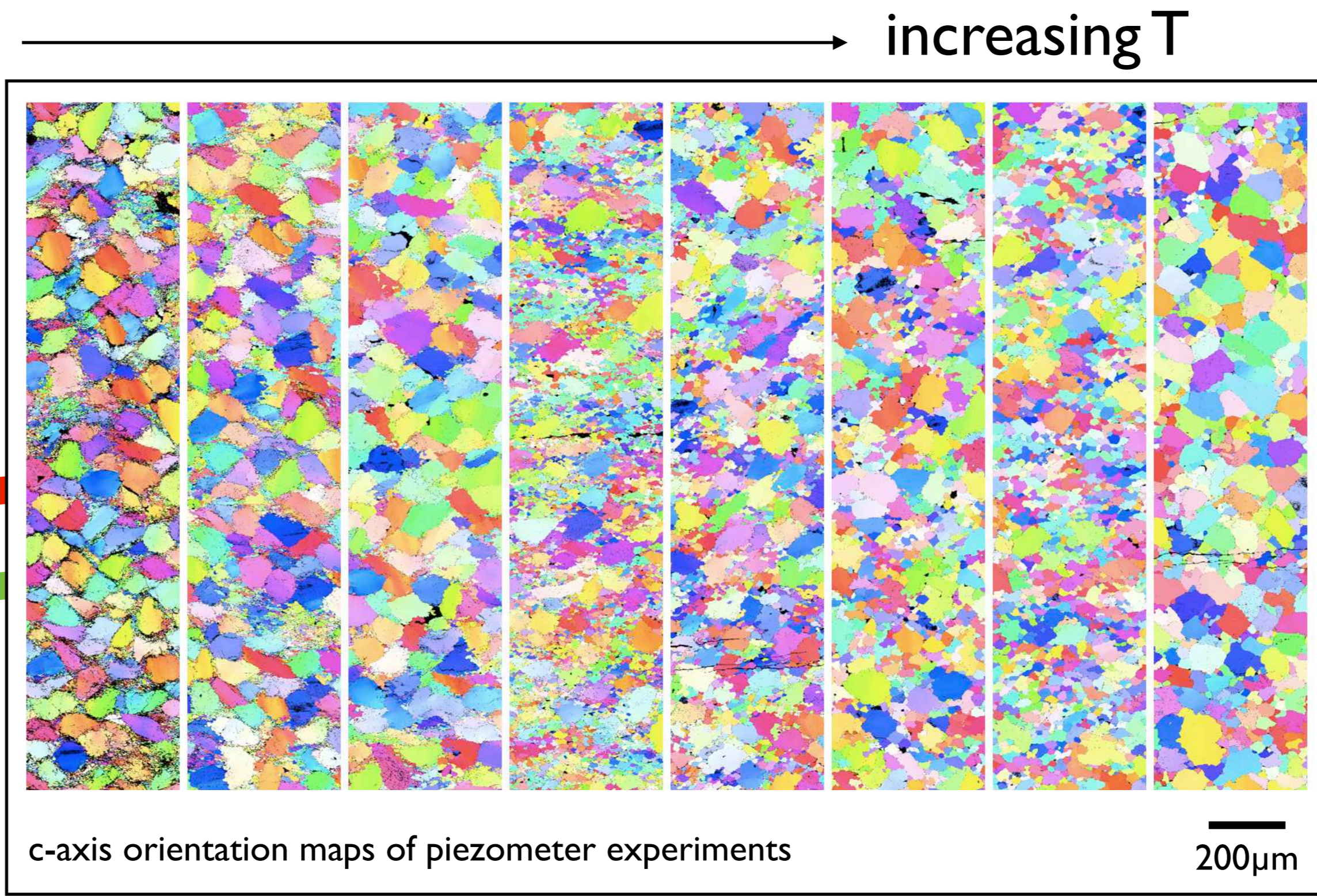
For the definition of 2Dmean and 3Dmode see previous slide



The piezometer experiments were carried out in a molten salt cell (MSC), allegedly for better stress resolution, compared to the general shear experiments which were carried out as standard experiments with a solid salt assembly (SSA). In other words, the comparison was: (SSA shear) versus (MSC coax)

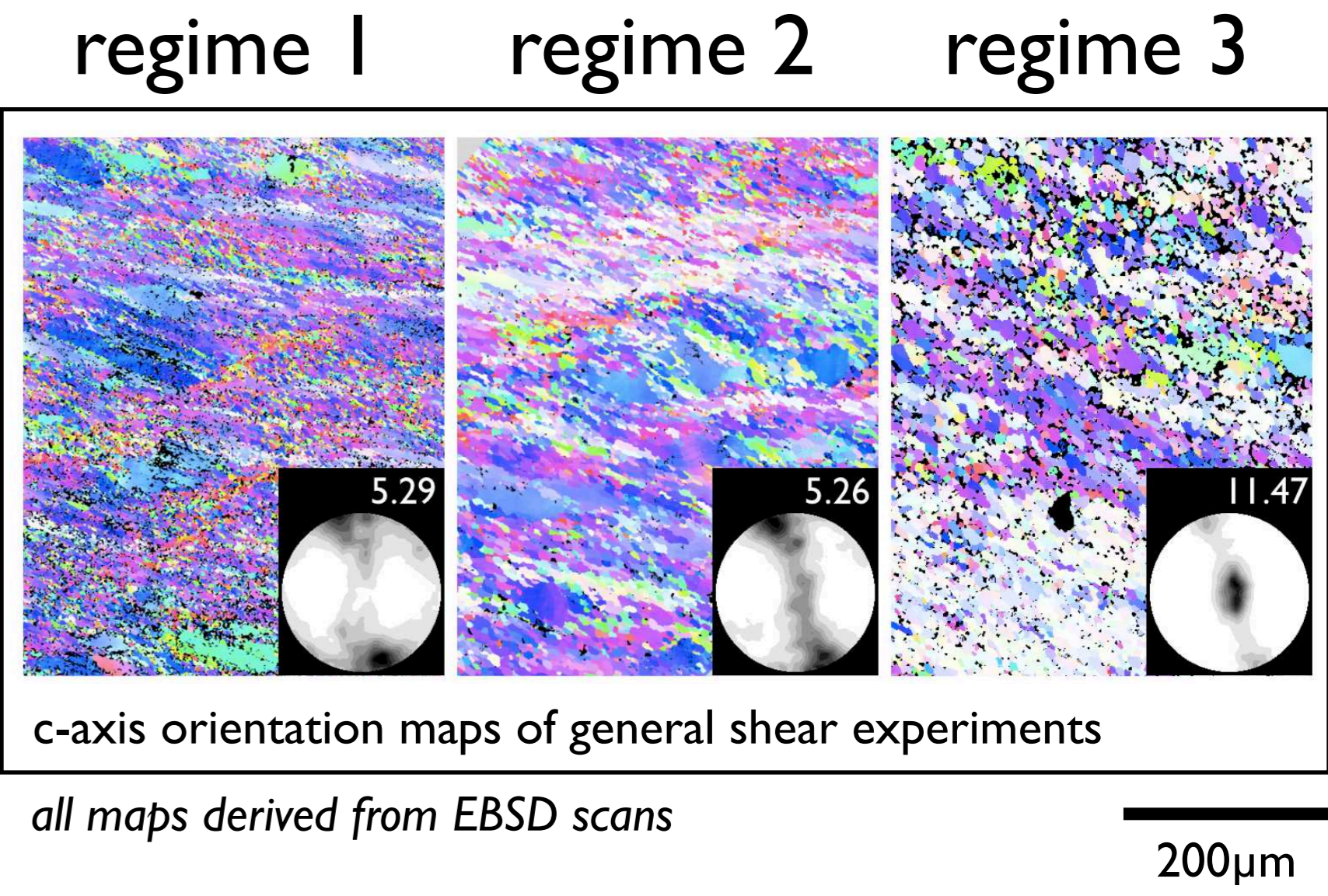
Black Hills quartzite deformed at 900-1100°C, 1.5 GPa and $10^{-5} - 10^{-6} \text{ s}^{-1}$

molten salt cell (MSC)



Black Hills quartzite deformed at 850 - 915°C, 1.5 GPa and 10^{-5} s^{-1}

solid salt assembly (SSA)



Observation:

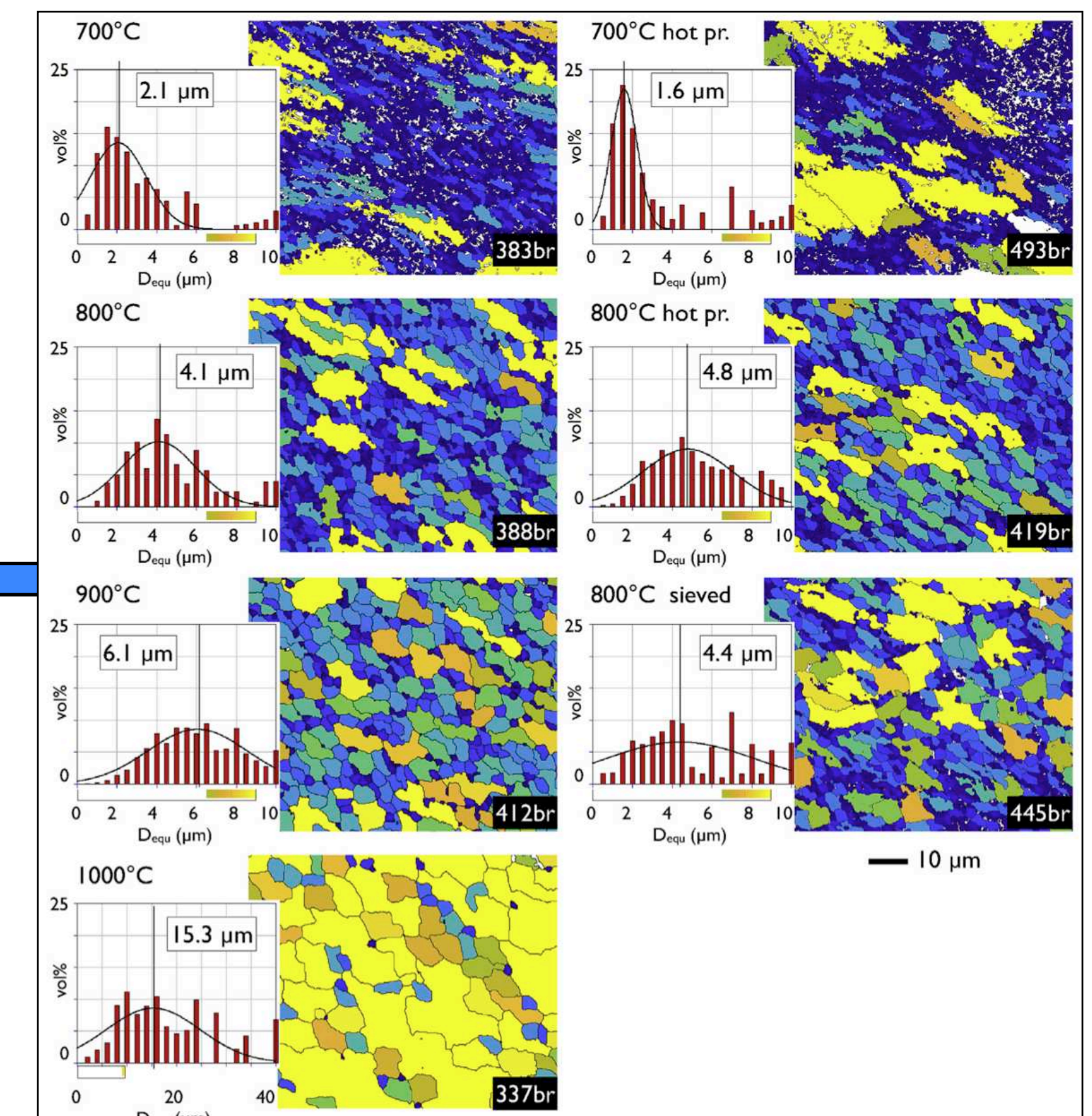
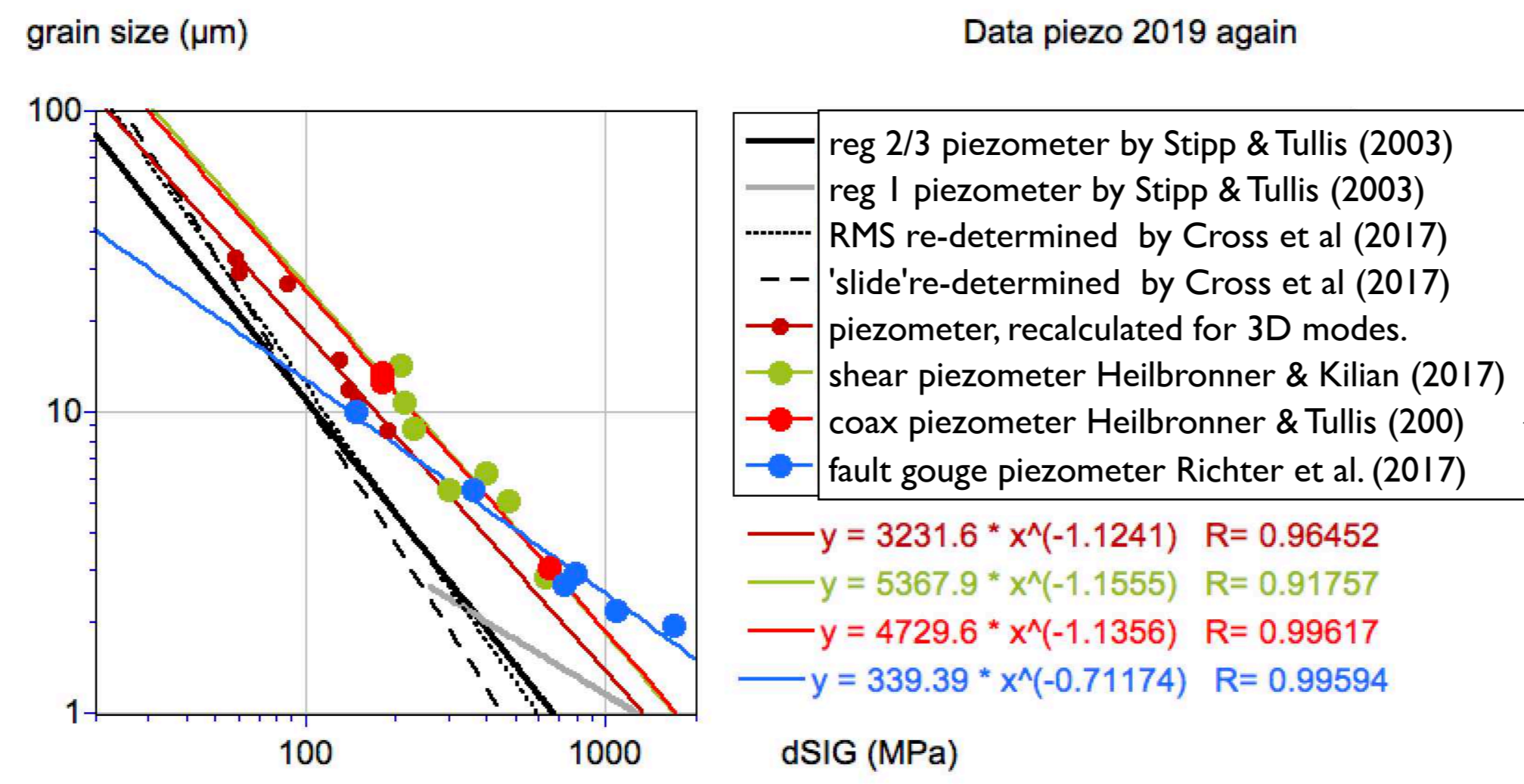
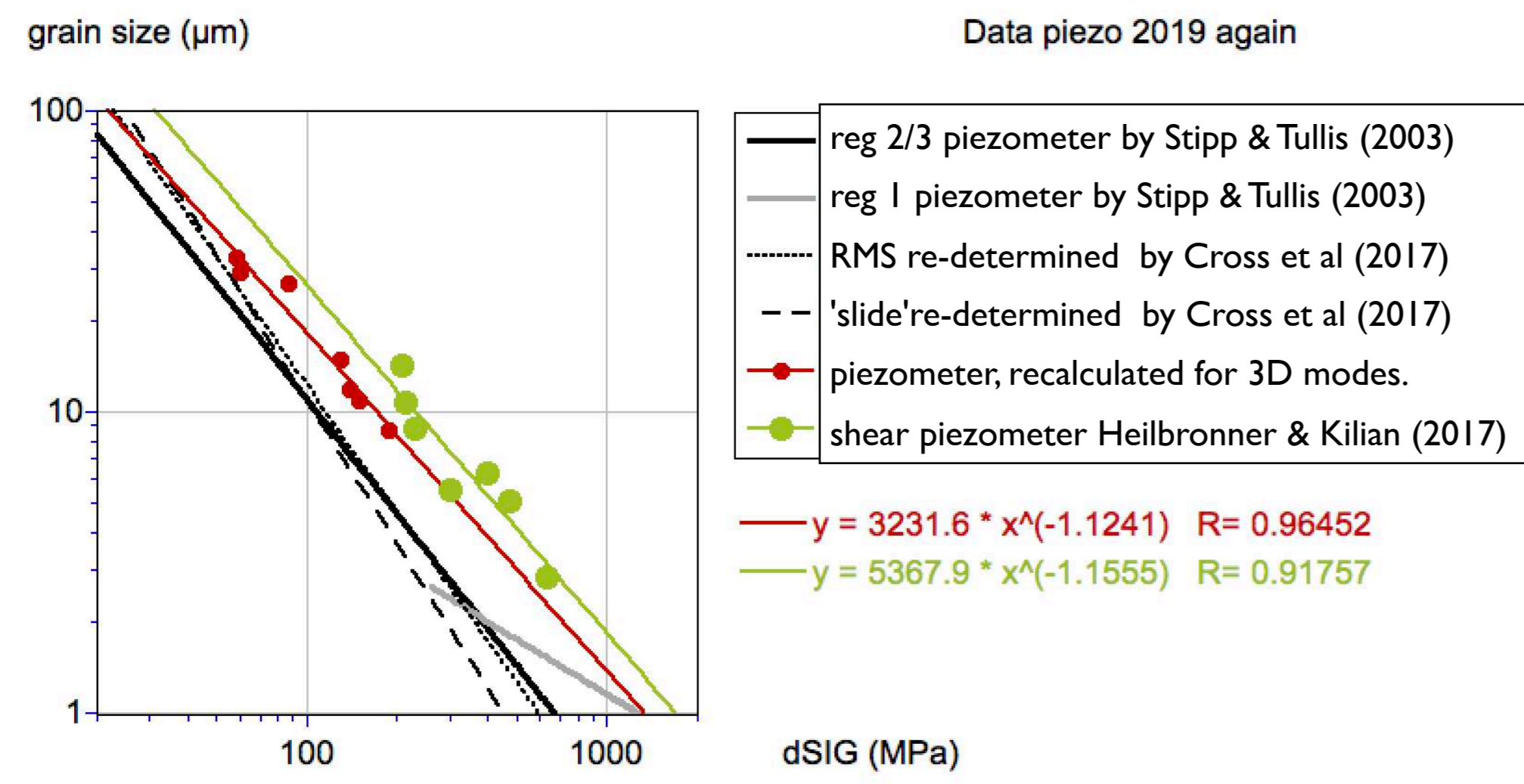
The piezometer (3D modes) of SSA shear experiments plots above the reg2/reg3 branch (dark red line) of the 3Dmodes recalculated for the piezometer published by Stipp & Tullis (2003) !

check grain size fault gouge SSA vs. piezometer MSA (reg I)

Finally, in order to get information for the very small grain size, we also checked the grain size of general shear general experiments (SSA) on fault gouge (crushed single crystal) carried out by Richter et al. (2016, 2018).

Quartz fault gouge deformed at 700 - 900°C, 1.5 GPa and 10⁻⁵ s⁻¹

solid salt assembly (SSA)



Observation:

The piezometer (3D modes) derived from SSA shear experiments on quartz fault gouge also plots above the reg I branch (grey) of the piezometer (2D RMS) published by Stipp & Tullis (2003) !

Note: No EBSD maps were available to re-determine the grain sizes of the reg I branch of the piezometer, nor could their 3D modes be re-calculated.

new software for the conversion of mechanical data

Three new 'rig' programs have been written for the explicit conversion of mechanical data (force, displacement, confining pressure) to stress strain curves and other mechanical output (strain rate, equivalent viscosity etc.)

rigP: convert run record to input file XXX.in.txt

rigC: calculates stress strain from XXX.in.txt for coaxial experiments

rigS: calculates stress strain from XXX.in.txt for shearing experiments

The idea is to make every choice explicit (no default values)

- type of hitpoint
- friction correction
- value of σ_1 and σ_3 at start of experiment
- area correction
- strain determination
- etc.

It is hoped that by writing rig programs in this manner, in future, stress strain curves from different labs should return the same results (... if the same experiments are analyzed with the same options, of course).

Two 'standard' conversions routines are examined in the following

- Brown for axial experiments
- Tromsø for shearing experiments

The effect of changing the options (type of hitpoint, area correction, etc.) will be demonstrated.

rigP (= Prepare)

This program prepares the input file for rigC and rigS.
(the format of this input file is the same for both)

rigP is only concerned with converting the recorded data to:

- time (s),
- axial load (kN) as $f(t)$,
- confining pressure (MPa) as $f(t)$,
- displacement of loading piston (mm),

and to provide information concerning:

- sample geometry (lengths (mm), widths (mm), angles ($^\circ$)),
- temperature setting ($^\circ\text{C}$).

rigC (= Coaxial) rigS (= Shear)

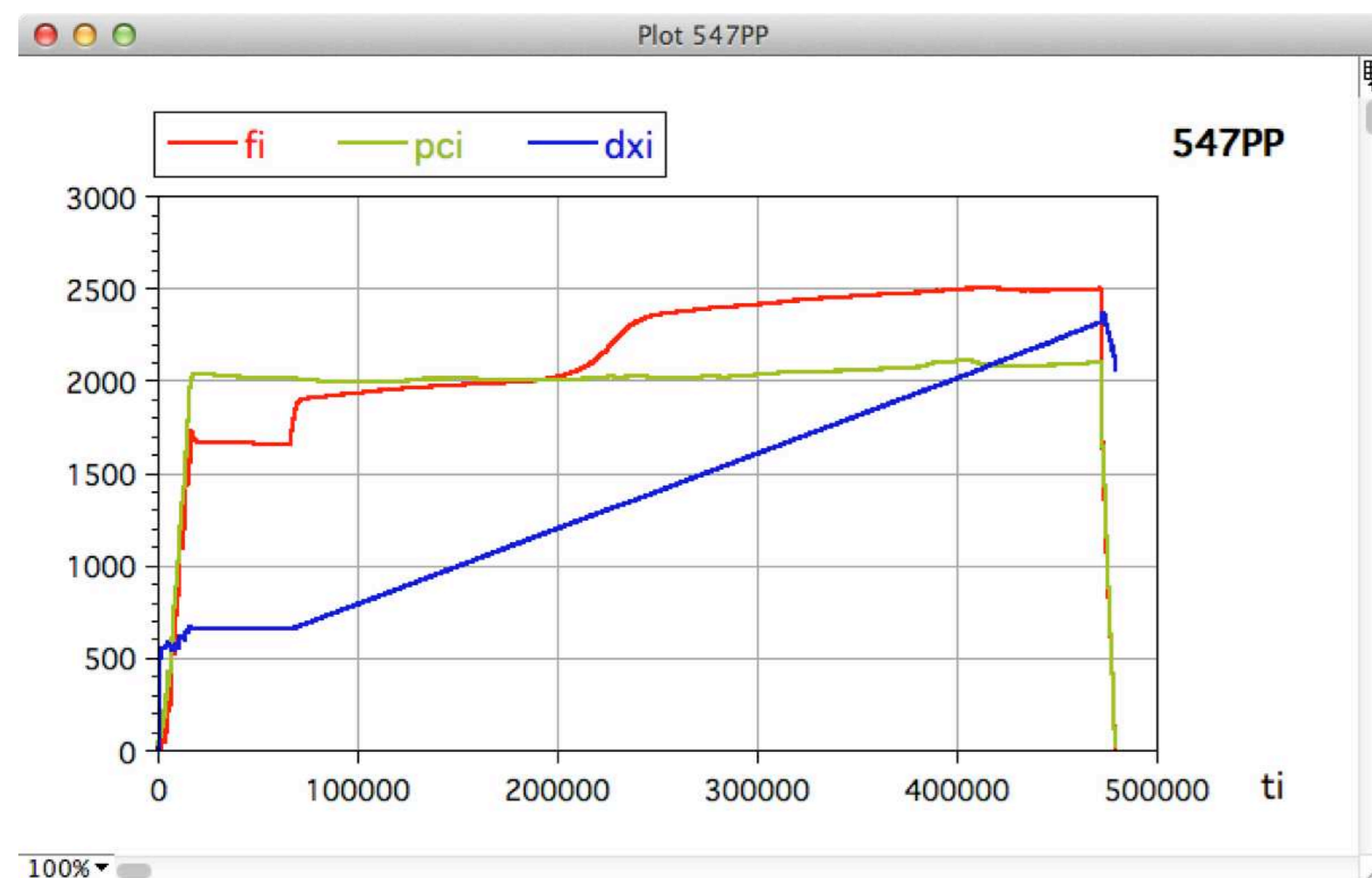
The following options are selected via dialogue:

- choice of hitpoint,
- friction correction (yes/no),
- salt correction (confining pressure increase) (yes/no),
- value of principal stresses, σ_1 and σ_3 , at the start of the experiment,
- definition of differential stress, $\Delta\sigma$,
- choice of area corrections,
- geometrical choices concerning sample thinning,
- choice of strain calculations.

rigP (= Prepare input files)

plot of Labview file

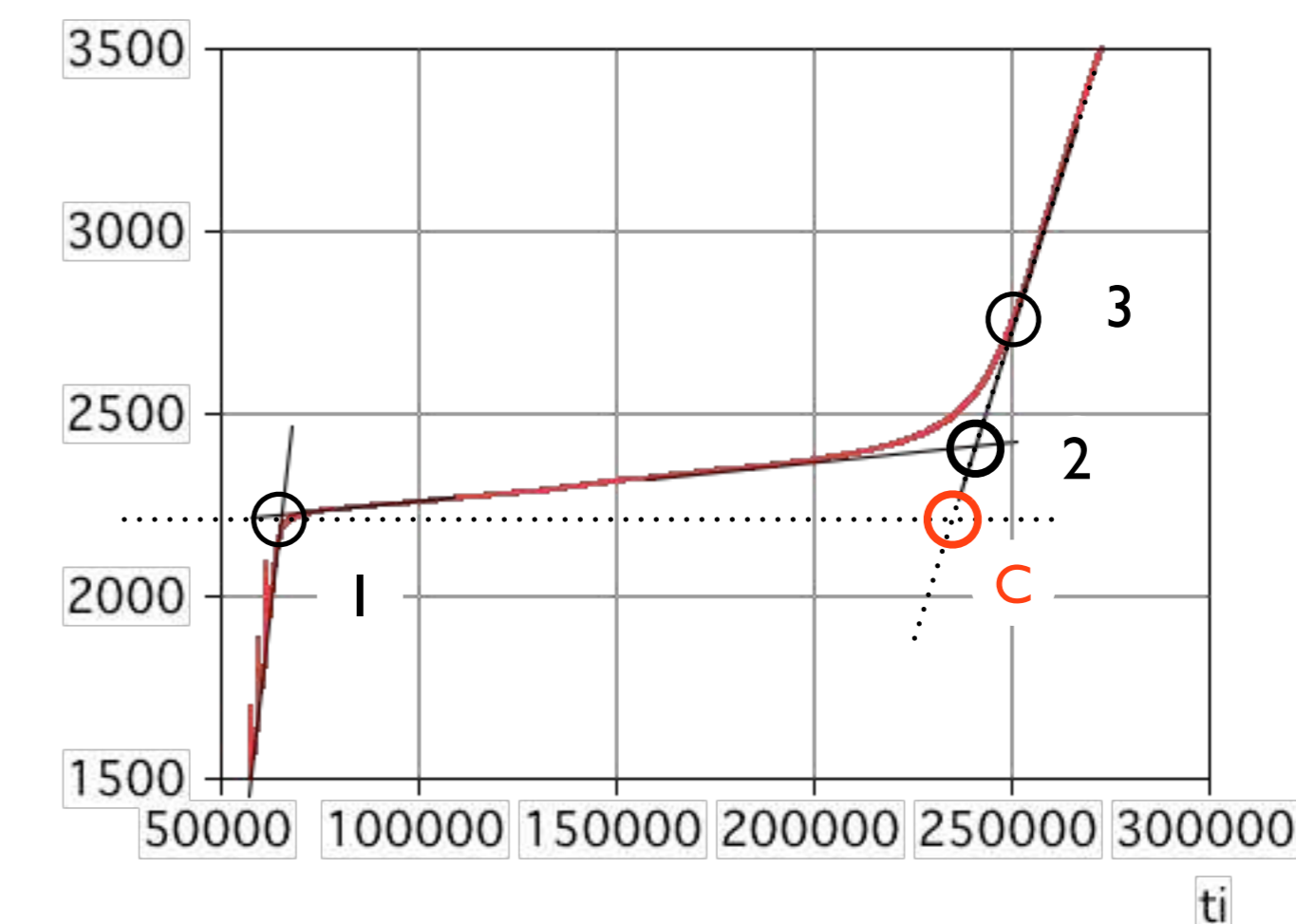
Plot 547PP.qpc



ti time (s)
 fi force
 pci confining pressure
 dxi displacement
 DLTi displacement

source:
 Labview file547PP.txt

determination of hitpoint



Numbers from run record

① = start of run-in
 ti = ilead = 65432
 fi = loadlead = 2222

② = hitpoint (2) = 'classical' hitpoint
 intersection of line fits
 ti = ihit = 240586
 fi = loadhit = 2410

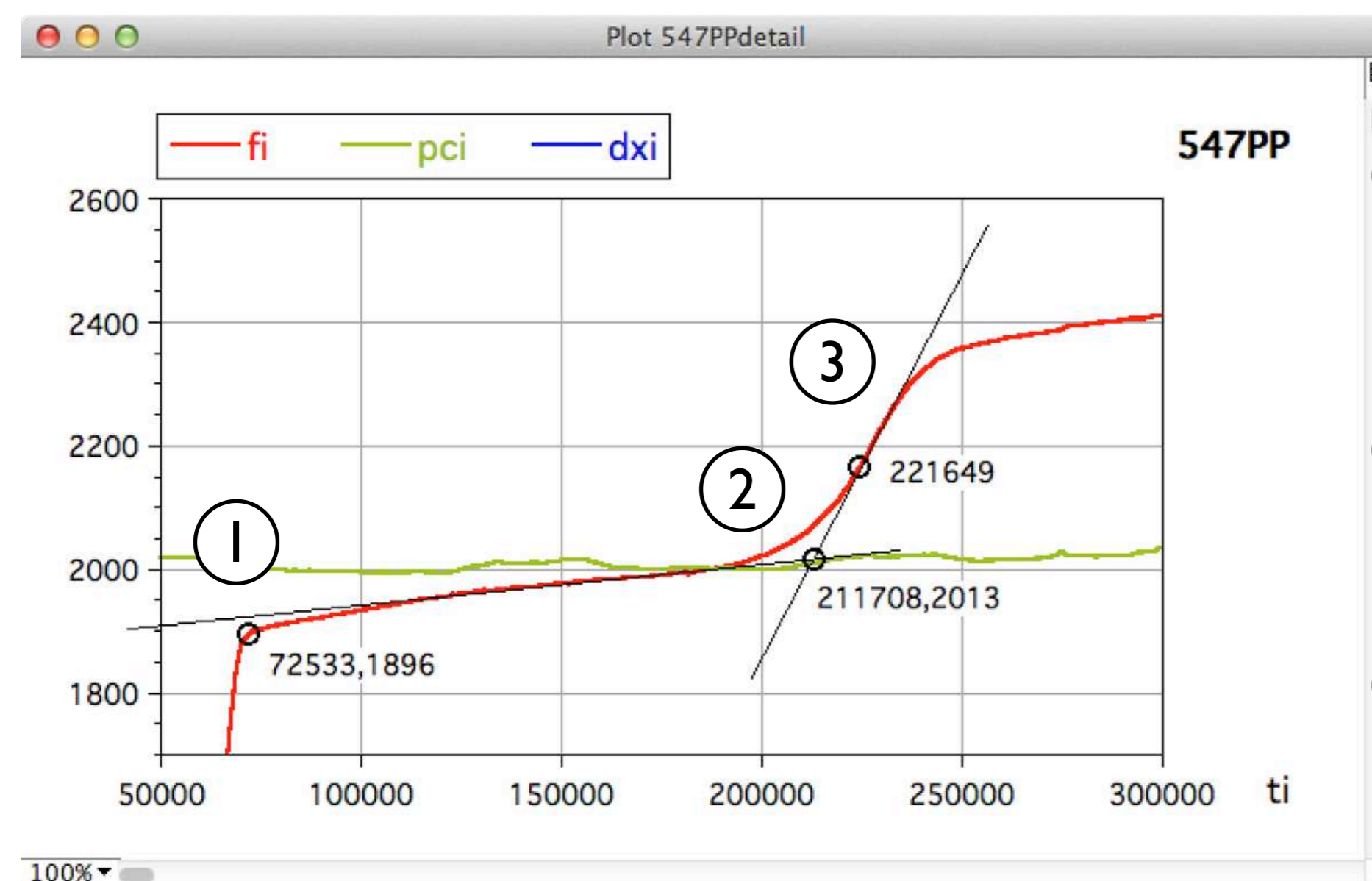
③ = from here on recorded data is used
 ti = istart = 251775
 fi = from load curve

Calculated in rigP from A, B and C

Ⓒ = hitpoint (1) = 'lead hitpoint'
 ti = extrapolated
 line fit (A→B)
 fi = loadlead = 2222
 same as C

data points selected in run record

Plot 547PPdetail.qpc



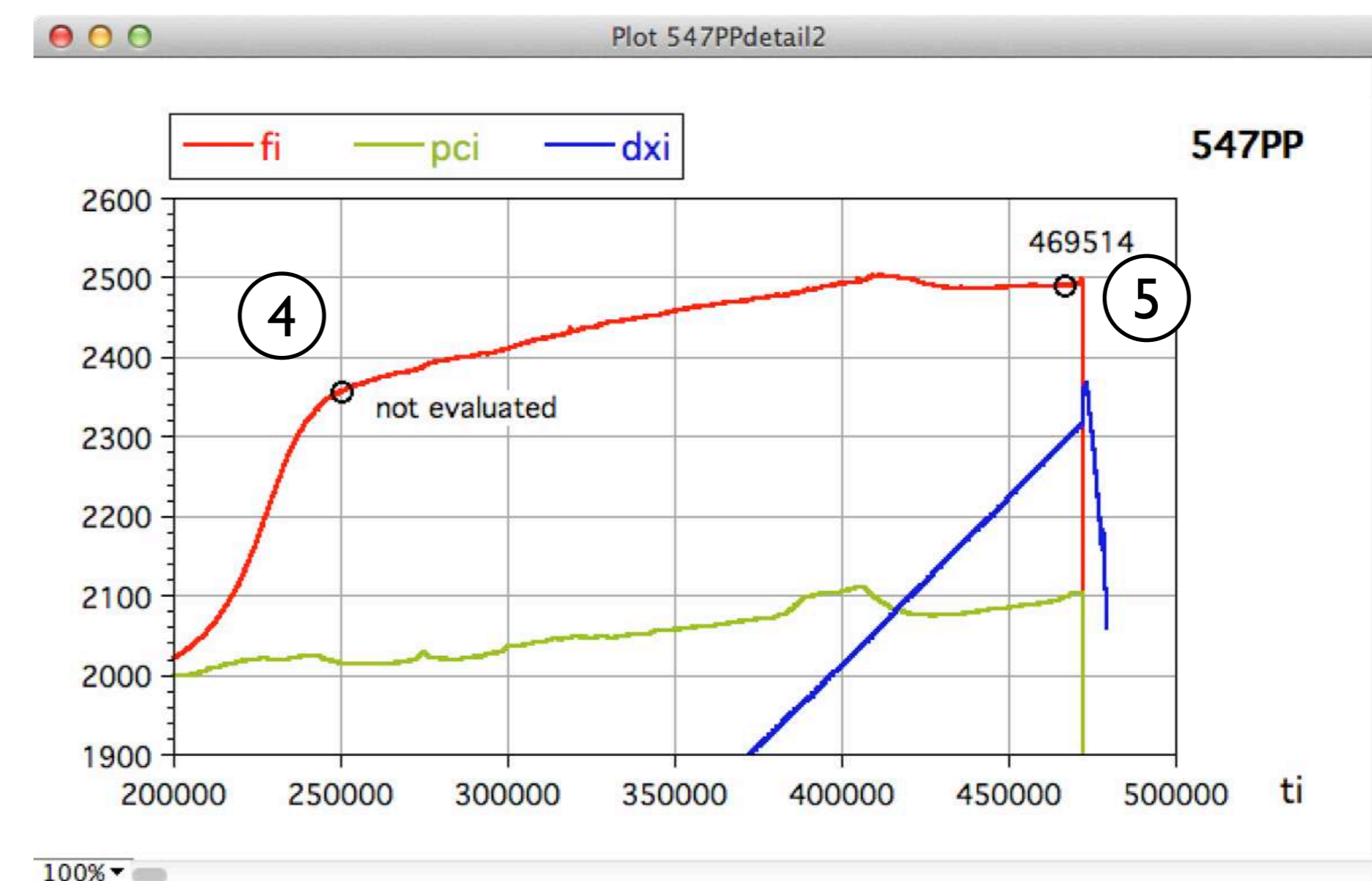
① point where σ_1 piston touches lead
 ti = ilead = 72533
 fi = loadlead = 1896

② hitpoint 2 (classical)
 ti = ihit1 = 211708
 fi = loadhit1 = 2013

③ first point to be used from curve
 ti = istart = 221649

data points selected in run record

Plot 547PPdetail2.qpc



④ peak load / yield point
 start of 'steady state'
 ti = ishear = 0

⑤ end experiment
 ti = iend = 469514

rigC (for coaxial experiments)

how to run rigC

using file **XXX.in.txt**

```

rigC06
-----
--
Program rigC              Basel,
2018-12-06
Uses input file with header and 5 columns
t(s), F(kN), Pc(MPa), d(mm), x(free)
1st line = hitpoint 1  2nd line = hitpoint 2
Explicit options
1-Select hitpoint (1=lead, 2=classical)
2-Friction correction for F
3-Salt correction for pc
4-Defining sig1(0) and sig3(0) at time=0
5-Options for area correction for sig1
6-Definition of sig3(t)
7-Options for differential stress Dsig(t)
8-Optional re-definition of sig1(t)
-----
Name of input file:
858rr.in.txt
-----
input file: 858rr.in.txt
header: Run record = w858 manual re-done
RH 201-11-27
rig number:          1
nominal Pc(MPa):    1500
temperature(°C):    900
log displacement rate of sig1
piston(ms-1):      -8

Correction for rig stiffness is not optional

```

red = input

```

Select hitpoint (1=lead, 2=classical)
2
Friction correction for F ? (1=yes 0=no)
0
Salt correction for pc ? (1=yes 0=no)
0
Defining sig1(0) and sig3(0) at time=0
1: set sig1(0) and sig3(0) to value of pc(0)
2: sig1(0)=sig3(0)=1/16*F/A(0)+15/16*pc(0)
3: use sig1(0) and sig3(0)=pc(0) as measured
1
Options for area correction
1: Homogeneous shortening of sample
2: Barreling of sample
3: No area correction
1
Definition of sig3(t)
1: sig3(t) = sig3(0) + salt corr. (can be = 0)
2: sig3(t) = sig3(0) + salt corr. (can be = 0)
1
Options for differential stress Dsig(t)
1: Dsig(t) = sig1(t)-sig1(0)
2: Dsig(t) = sig1(t)-sig3(t)
1
Optional re-definition of sig1(t)
1: sig1(t) = sig1(0) + Dsig(t)
2: sig1(t) = sig3(t) + Dsig(t)
3: leave sig1(t) as calculated
1
Name of output file ? [547PP.10122223.out.txt]
(return=default) >
<return>
-----
result file = 858rr.20011111.out.txt
-----

```

output file of rigC

```

Input file = 858rr.in.txt
Run record = w858 manual re-done RH 201-

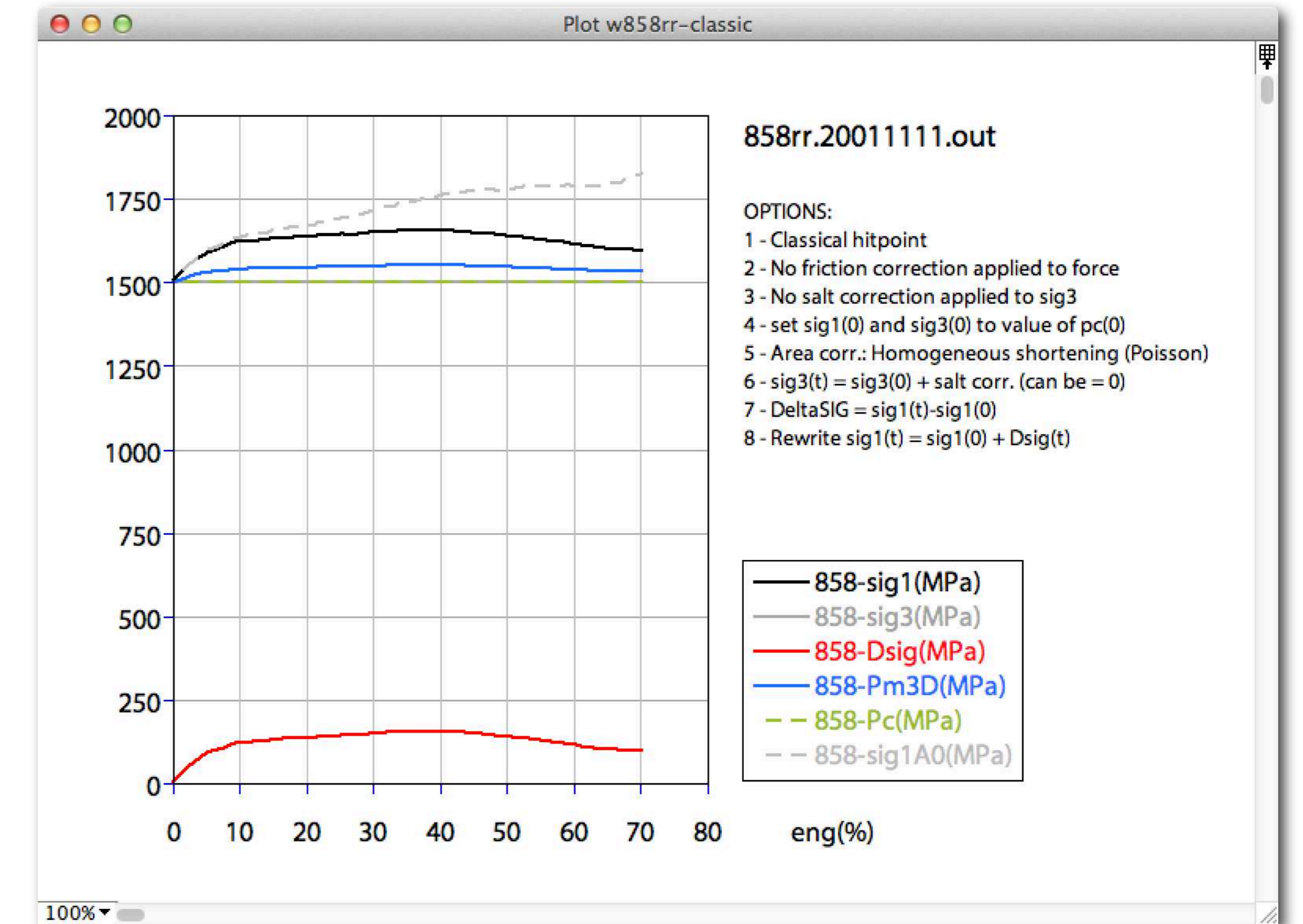
rig      Pc(MPa)   T(°C)   d-rate(ms-1)
Tromsø 1      1500    900      -8
diameter(mm) length(mm) time of max load
          6.220   13.820   0.0

OPTIONS:
1 - Classical hitpoint
2 - No friction correction applied to force
3 - No salt correction applied to sig3
4 - set sig1(0) and sig3(0) to value of pc(0)
5 - Area corr.: Homogeneous shortening (Poisson)
6 - sig3(t) = sig3(0) + salt corr. (can be = 0)
7 - DeltaSIG = sig1(t)-sig1(0)
8 - Rewrite sig1(t) = sig1(0) + Dsig(t)

```

t(s)	858-Fc(kN)	858-Pc(MPa)	d(mm)	dcorr(mm)	858-sig1A0(MPa)	858-sig1(MPa)	858-sig3(MPa)	858-Dsig(MPa)	858-Pm2D(MPa)	...	
0.0	5.1463	1500.0000	0.0000	0.0000	0.0000	1500.0000	1500.0000	1500.0000	0.0000	1500.0000	... etc.
6236.0	5.7401	1500.0000	0.1168	0.1163	1519.5420	1519.3774	1500.0000	19.3775	1509.6887		
12471.0	6.3339	1500.0000	0.2464	0.2454	1539.0841	1538.3901	1500.0000	38.3902	1519.1951		
18707.0	6.9277	1500.0000	0.3683	0.3668	1558.6261	1557.0701	1500.0000	57.0701	1528.5350		
24942.0	7.4028	1500.0000	0.4877	0.4858	1574.2617	1571.6512	1500.0000	71.6513	1535.8257		
31178.0	7.7986	1500.0000	0.6096	0.6074	1587.2876	1583.4514	1500.0000	83.4514	1541.7257		
37413.0	8.1153	1500.0000	0.7290	0.7265	1597.7102	1592.5737	1500.0000	92.5737	1546.2869		
43649.0	8.3133	1500.0000	0.8458	0.8431	1604.2263	1597.8677	1500.0000	97.8677	1548.9338		
49884.0	8.5508	1500.0000	0.9652	0.9623	1612.0426	1604.2407	1500.0000	104.2407	1552.1204		
56120.0	8.7091	1500.0000	1.0871	1.0841	1617.2522	1608.0544	1500.0000	108.0544	1554.0272		
62355.0	9.1050	1500.0000	1.2065	1.2032	1630.2814	1618.9391	1500.0000	118.9391	1559.4695		
68591.0	9.2634	1500.0000	1.3335	1.3300	1635.4944	1622.4545	1500.0000	122.4544	1561.2273		
74826.0	9.3029	1500.0000	1.4580	1.4545	1636.7943	1622.3972	1500.0000	122.3973	1561.1986		
81062.0	9.4217	1500.0000	1.5799	1.5763	1640.7040	1624.6554	1500.0000	124.6554	1562.3276		
87297.0	9.5009	1500.0000	1.6916	1.6879	1643.3105	1625.8070	1500.0000	125.8070	1562.9036		

... etc.



rigS (for general shearing experiments)

how to run rigC

using file **XXX.in.txt**

```
rigS
-----
--
Program rigS              Basel,
2018-12-10
Uses input file with header and 5 columns
t(s), F(kN), Pc(MPa), d(mm), x(free)
1st line = hitpoint 1  2nd line = hitpoint 2
Explicit options
1-Select hitpoint (1=lead, 2=classical)
2-Friction correction for F
3-Salt correction for pc
4-Defining sig1(0) and sig3(0) at time=0
5-Definition of sig3(t)
6-Options for differential stress Dsig(t)
7-Optional re-definition of sig1(t)
8-Options for area (overlap) correction
-----
Name of input file:
1092rr.in.txt
-----
input file: 1092rr.in.txt
header: Run record = w1092 manual re-done
RH 201
rig number:          1
nominal Pc(MPa):     1550
temperature(°C):     850
log displacement rate of sig1
piston(ms-1):        -8
Correction for rig stiffness is not optional
```

red = input

```
Select hitpoint (1=lead, 2=classical)
2
Friction correction for F ? (1=yes 0=no)
0
Salt correction for pc ? (1=yes 0=no)
0
Defining sig1(0) and sig3(0) at time=0
1: set sig1(0) and sig3(0) to value of pc(0)
2: sig1(0)=sig3(0)=1/16*F/A(0)+15/16*pc(0)
3: use sig1(0) and sig3(0)=pc(0) as measured
1
Area correction for piston overlap
1: ACF (auto correlation function)
2: Modified ACF (delayed overlap)
3: Cosine square overlap
4: No area correction
1
Definition of sig3(t)
1: sig3(t) = sig3(0) + salt corr. (can be = 0)
2: sig3(t) = sig3(t) + salt corr. (can be = 0)
1
Options for differential stress Dsig(t)
1: Dsig(t) = sig1(t)-sig1(0)
2: Dsig(t) = sig1(t)-sig3(t)
1
Optional re-definition of sig1(t)
1: sig1(t) = sig1(0) + Dsig(t)
2: sig1(t) = sig3(t) + Dsig(t)
3: leave sig1(t) as calculated
1
Name of output file ? [1092rr.20011111.out.txt]
(return=default) >
result file = 1092rr.20011111.out.txt
<return>
----- done -----
surfer-172-30-2-210-hotspot:Desktop heilbronner$
```

output file of rigS

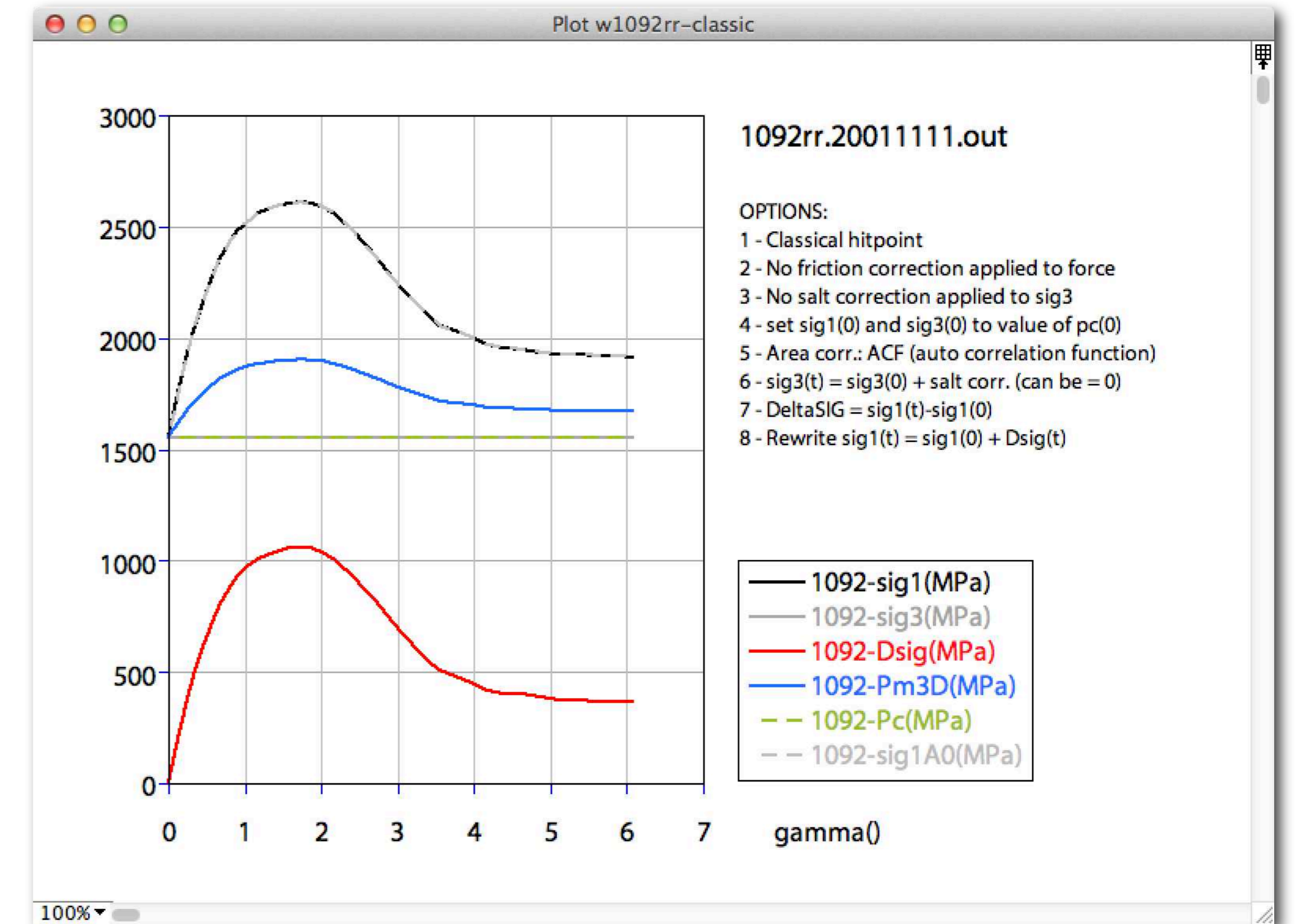
```
Input file = 1092rr.in.txt
Run record = w1092 manual re-done RH 201

rig      Pc(MPa)   T(°C)   d-rate(ms-1)
Tromsoe 1      1550    850      -8
diameter(mm) length(mm) time of max load
          6.300    0.000    0.0

OPTIONS:
1 - Classical hitpoint
2 - No friction correction applied to force
3 - No salt correction applied to sig3
4 - set sig1(0) and sig3(0) to value of pc(0)
5 - Area corr.: ACF (auto correlation function)
6 - sig3(t) = sig3(0) + salt corr. (can be = 0)
7 - DeltaSIG = sig1(t)-sig1(0)
8 - Rewrite sig1(t) = sig1(0) + Dsig(t)
```

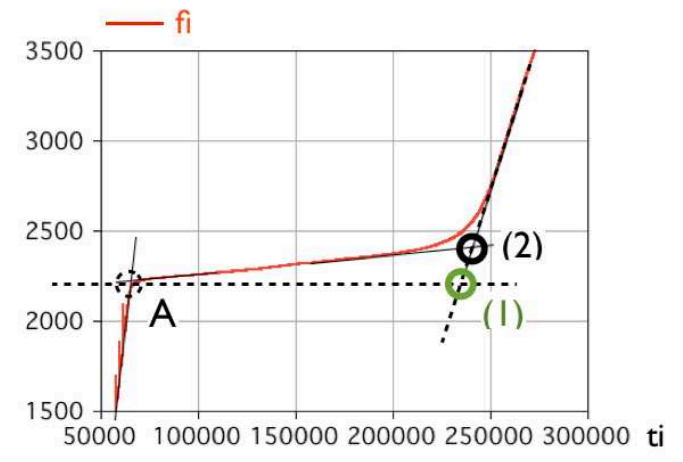
t(s)	1092-Fc(kN)	1092-Pc(MPa)	d(mm)	dcorr(mm)	1092-sig1A0(MPa)	1092-sig1(MPa)	1092-sig3(MPa)	1092-Dsig(MPa)	...
0.0	55.4210	1550.0000	0.0000	0.0000	0.0000	1550.0000	1550.0000	0.0000	1550.0000
9353.0	62.5470	1550.0000	0.1905	0.1845	1778.5995	1778.5995	1550.0000	228.5995	1664.2998
15589.0	67.1390	1550.0000	0.3175	0.3076	1925.9091	1925.9091	1550.0000	375.9091	1737.9546
21824.0	71.2560	1550.0000	0.4445	0.4312	2057.9810	2057.9810	1550.0000	507.9810	1803.9905
28060.0	75.0560	1550.0000	0.5715	0.5550	2179.8833	2179.8833	1550.0000	629.8833	1864.9417
34295.0	77.9060	1550.0000	0.6960	0.6770	2271.3105	2271.3105	1550.0000	721.3105	1910.6553
40531.0	80.5980	1550.0000	0.8204	0.7992	2357.6689	2357.6689	1550.0000	807.6689	1953.8345
46766.0	82.8150	1550.0000	0.9449	0.9219	2428.7896	2428.7896	1550.0000	878.7896	1989.3948
53002.0	84.7150	1550.0000	1.0719	1.0473	2489.7407	2489.7407	1550.0000	939.7407	2019.8704
59237.0	85.9820	1550.0000	1.1963	1.1706	2530.3857	2530.3857	1550.0000	980.3857	2040.1929
65473.0	86.9320	1550.0000	1.3259	1.2994	2560.8613	2560.8613	1550.0000	1010.8613	2055.4307
71708.0	87.5650	1550.0000	1.4605	1.4335	2581.1675	2581.1675	1550.0000	1031.1675	2065.5837
77944.0	88.0400	1550.0000	1.5875	1.5601	2596.4053	2596.4053	1550.0000	1046.4053	2073.2026
84179.0	88.3570	1550.0000	1.7145	1.6868	2606.5747	2606.5747	1550.0000	1056.5747	2078.2874
90415.0	88.5150	1550.0000	1.8415	1.8137	2611.6431	2611.6431	1550.0000	1061.6431	2080.8215

... etc.



options for rigC and rigS

DEFINITION OF THE HIT POINT

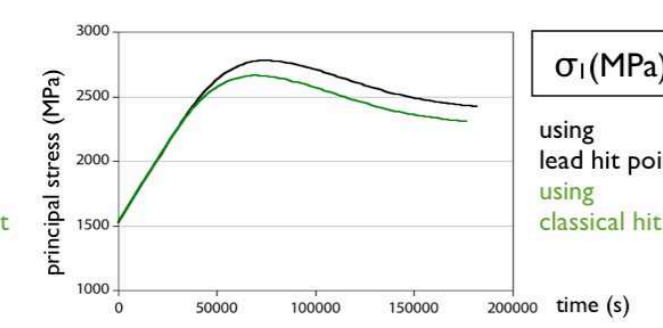
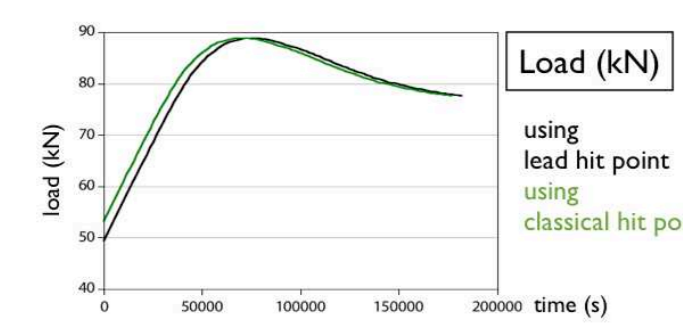


We use hit point (1) defined by the 'leadhit'. Justification: Fluid inclusions indicate that the load accumulated during run-in is 'felt' by sample., See Tarantola et al. (2010)

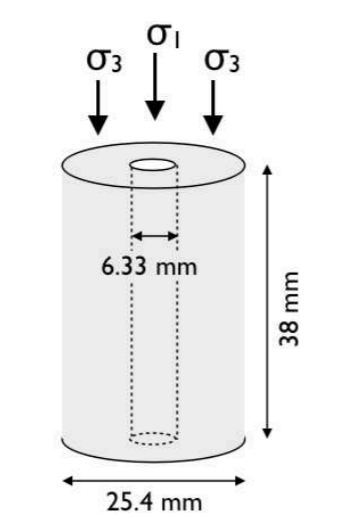
A = lead hit
 ti = ilead = 65432
 fi = loadlead = 2222
 hit point (1)
 ti = extrapolated
 fi = loadlead = 2222
 hit point (2) = 'classical' hitpoint
 ti = ihit = 240586
 fi = loadhit = 2410

Raw data of sample 383
 fi = force
 ti = time

Experimental conditions:
 T = 700°C, Pc = 1.5 GPa, $\dot{\gamma} = 10^{-5} \text{ s}^{-1}$



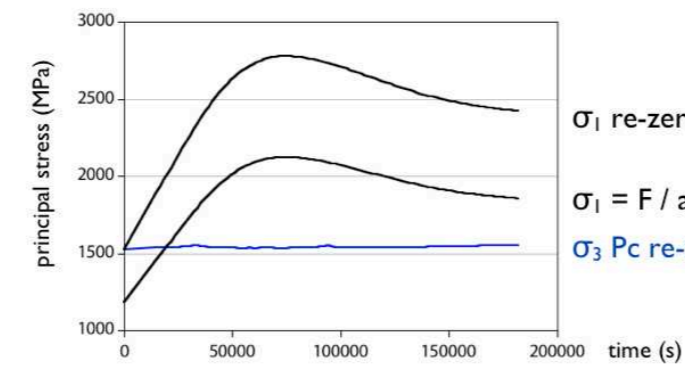
CALCULATING PRINCIPAL STRESSES



What is confining pressure at start of experiment ?

Measured values at $t = 0$:
 $\sigma_1(0) = \text{Load} / (\text{Area of } \sigma_1 \text{ - piston})$
 $\sigma_3(0) = \text{Confining pressure } P_c$
 Note: typically $\sigma_1(0) \neq \sigma_3(0)$

Set $\sigma_3(0) = \sigma_1(0) = \text{confining pressure}$
 $\sigma_0 = 1/16 \cdot (\sigma_1(0) + 15 \cdot \sigma_3(0))$
 $\sigma_0 = 1/16 \cdot (F(0)/A + 15 \cdot P_c(0))$
 Note: diameter of σ_1 - piston = 1/4 of diameter of sample assembly (including confining medium)

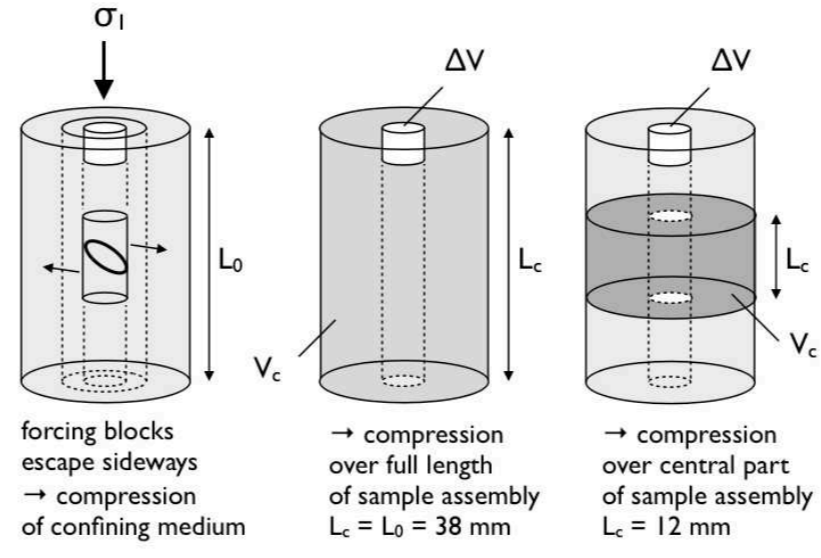


$\sigma_1(t) = \sigma_0 + (F(t) - F(0)) / A$
 $\sigma_3(t) = \sigma_0 + (P_c(t) - P_c(0))$

$\Delta\sigma(t) = \sigma_1(t) - \sigma_3(t)$
 $p_{\text{mean}} = 0.5 \cdot (\sigma_1(t) + \sigma_3(t))$

$\tau(t) = \sin(2\alpha) \cdot 0.5 \cdot \Delta\sigma(t)$
 $\sigma_n(t) = p_{\text{mean}} + \cos(2\alpha) \cdot \Delta\sigma(t)$

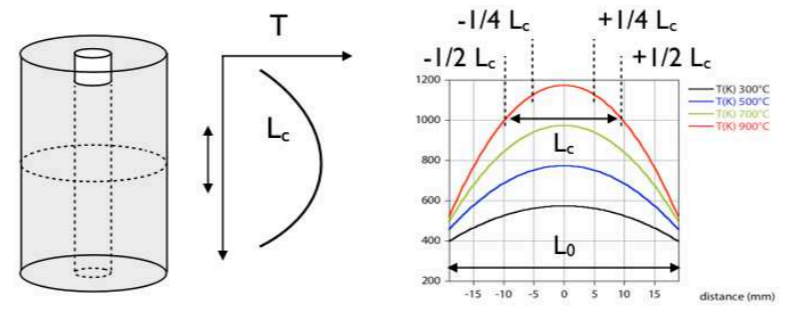
THE SALT CORRECTION



Basic idea:
 As the σ_1 piston advances, the confining medium cannot expand and is therefore subjected to an additional bulk compression. The additional confining pressure ΔP_c is calculated as

$\Delta P_c = \beta \cdot \Delta V / V_c$

where
 β = bulk modulus and
 $\Delta V / V_c$ = relative volume decrease
 V_c = compressed part of confining medium

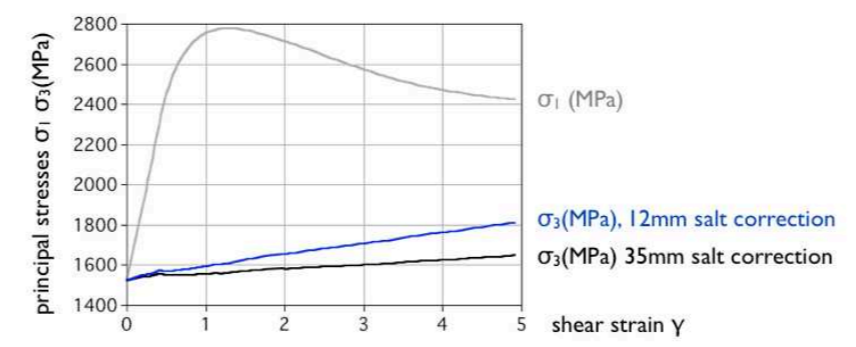


Considering only the central part of the confining medium is based on the T-gradient. The high conductivity induces cooling from the center towards both ends of assembly.

The pertinent T for a given pressurized length is at $\pm 1/4$ of L_c .

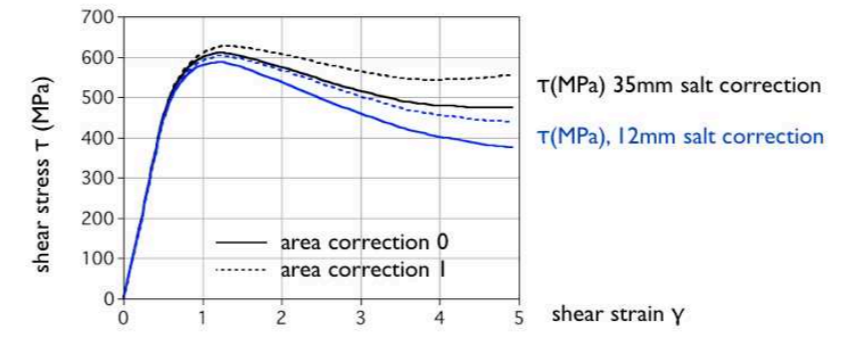
Measured temperature - profiles in sample assemblies (Pec, PhD thesis, 2014)

bulk compressibility β for NaCl							bulk compressibility β for KI						
T (°C)	0 kb	3 kb	5 kb	10 kb	15 kb	20 kb	T (°C)	0 kb	3 kb	5 kb	10 kb	15 kb	20 kb
100							100						
200	39.0	39.9	40.4				200	26.0	26.4	26.6			
300	35.7	36.7	37.4	38.7	39.7	40.4	300	24.5	25.0	25.3	25.9	26.3	26.6
400	32.6	33.7	34.4	35.8	37.0	37.9	400	23.0	23.6	23.9	24.6	25.1	25.5
500	30.0	31.0	31.7	33.2	34.4	35.4	500	21.8	22.3	22.6	23.3	23.9	24.4
600	28.0	28.9	29.5	30.8	32.1	33.1	600	21.0	21.4	21.6	22.2	22.8	23.3
700	26.7	27.4	27.8	29.0	30.0	31.0	700	20.6	20.8	20.9	21.4	21.8	22.3
800	25.9	26.3	26.7	27.5	28.4	29.3	800	20.6	20.6	20.6	20.8	21.2	21.5
900			25.9	26.5	27.2	27.9	900			20.6	20.6	20.7	21.0
1000				25.9	26.4	26.9	1000				20.6	20.6	20.6
1100						26.2	1100						20.5



σ_1 is not affected by the increase in P_c . Only σ_3 is affected by the salt correction.

For increasing salt correction, P_c increases, $\Delta\sigma$ and τ decrease.



$\Delta P_c(d)$ is calculated for given P_c and T as a function of piston advancement.

At higher temperatures, the bulk compressibility β and the effect of the salt correction decrease, i.e., the samples weaken less.

The net effect of choosing the lead hit point versus the classical hit point is to decrease the load at time 0, and thus to increase $\Delta\sigma$ and τ .
 As time 0 is advanced, the total shear displacement and hence the total shear strain is increased

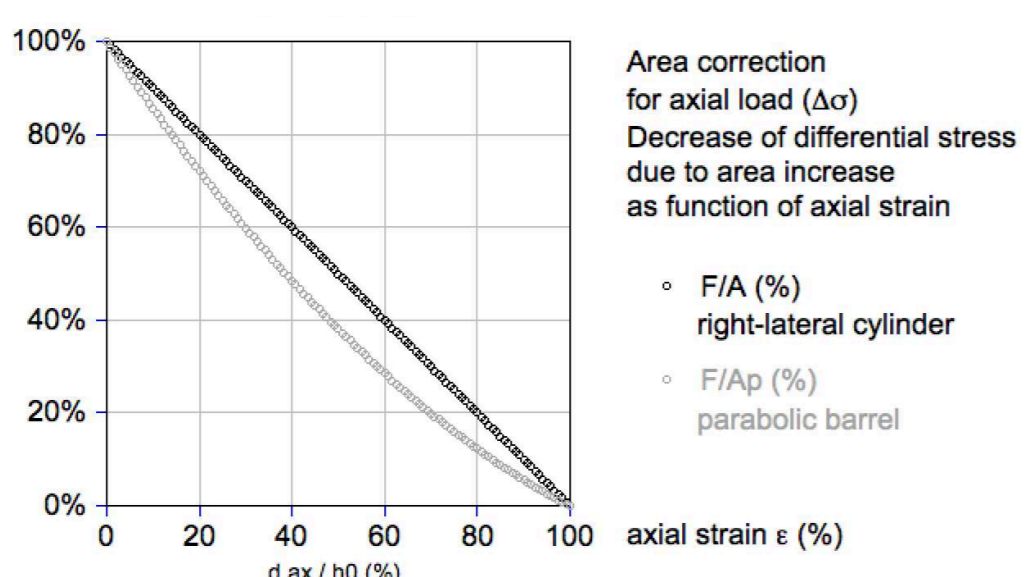
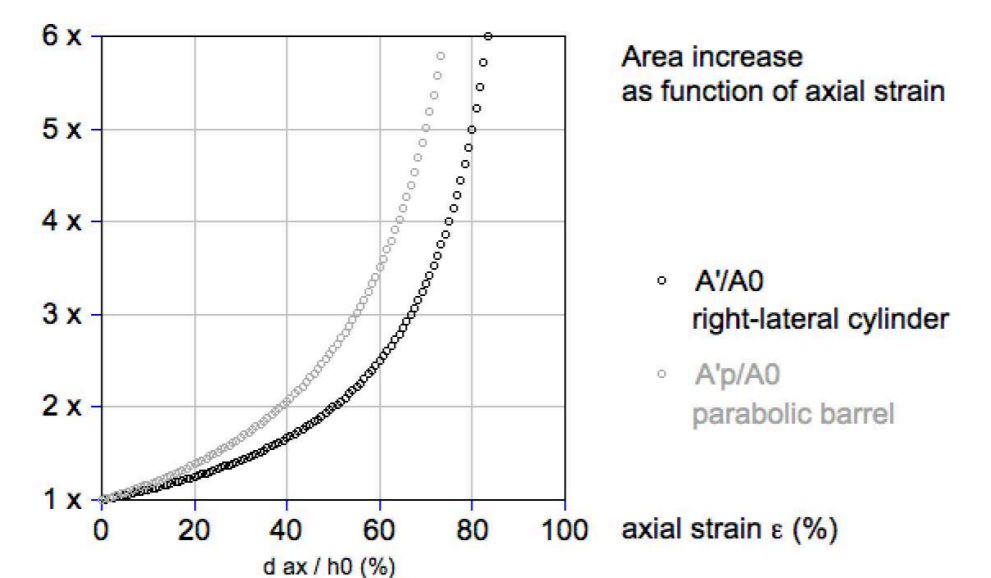
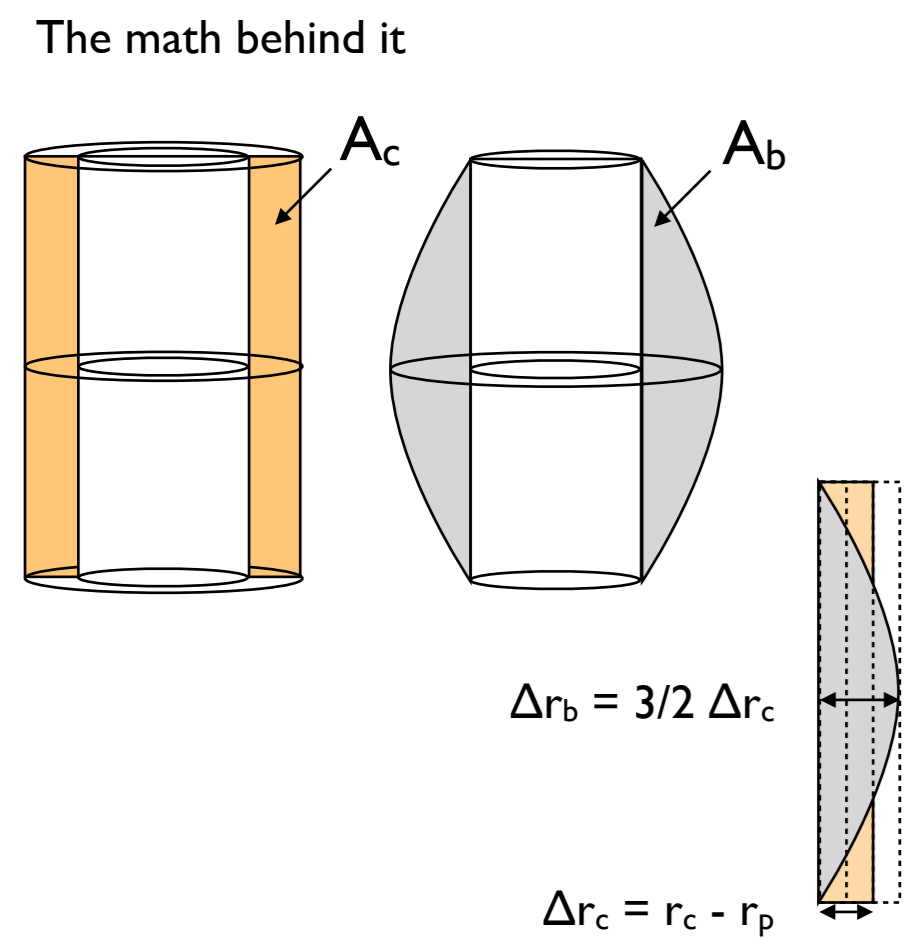
At the start of the experiment (hit point):
 $\Delta\sigma = 0 \Rightarrow \sigma_1(0) = \sigma_3(0) = P_c(0)$
 $\sigma_1(0)$ and $\sigma_3(0)$ can be set to $\sigma_1(0)$ or $\sigma_3(0)$ or a proportion of $\sigma_1(0) : \sigma_3(0) = 1 : 15$ (corresponding to the cross sectional area of the $\sigma_1(0)$ and $\sigma_3(0)$ pistons).

The net effect of the salt correction is to increase the confining pressure.

area correction

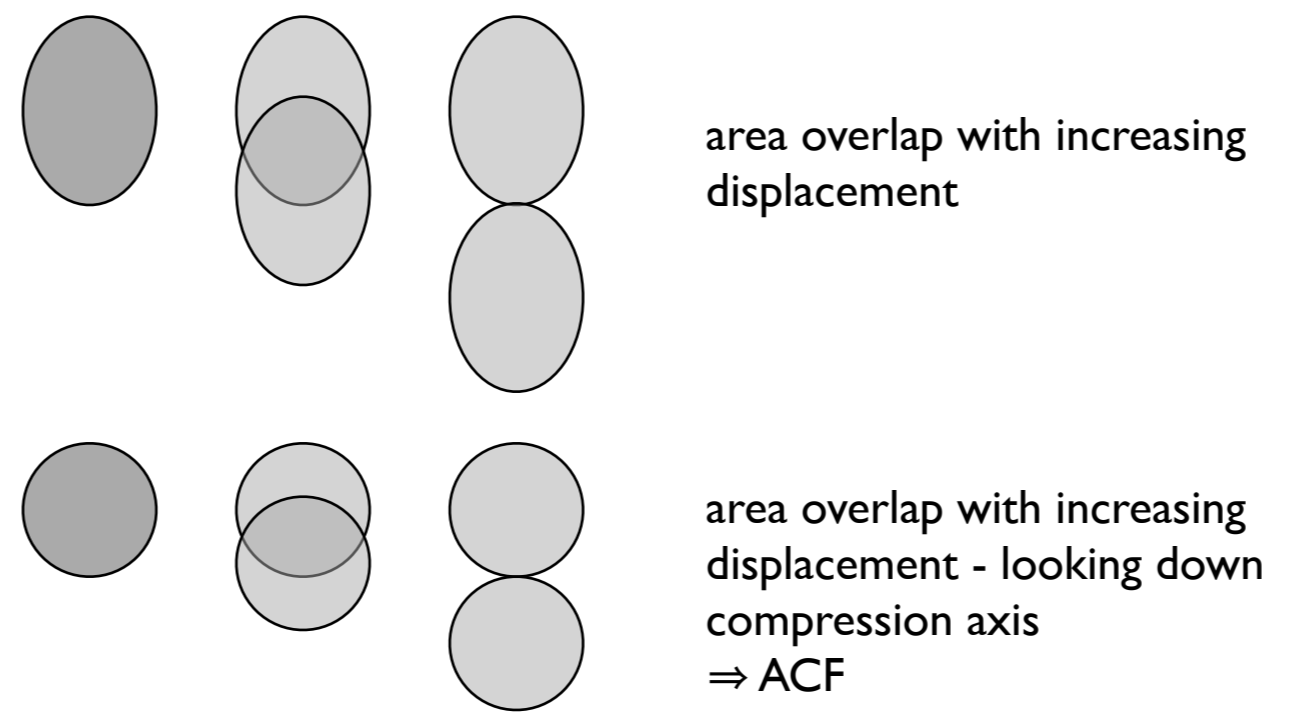
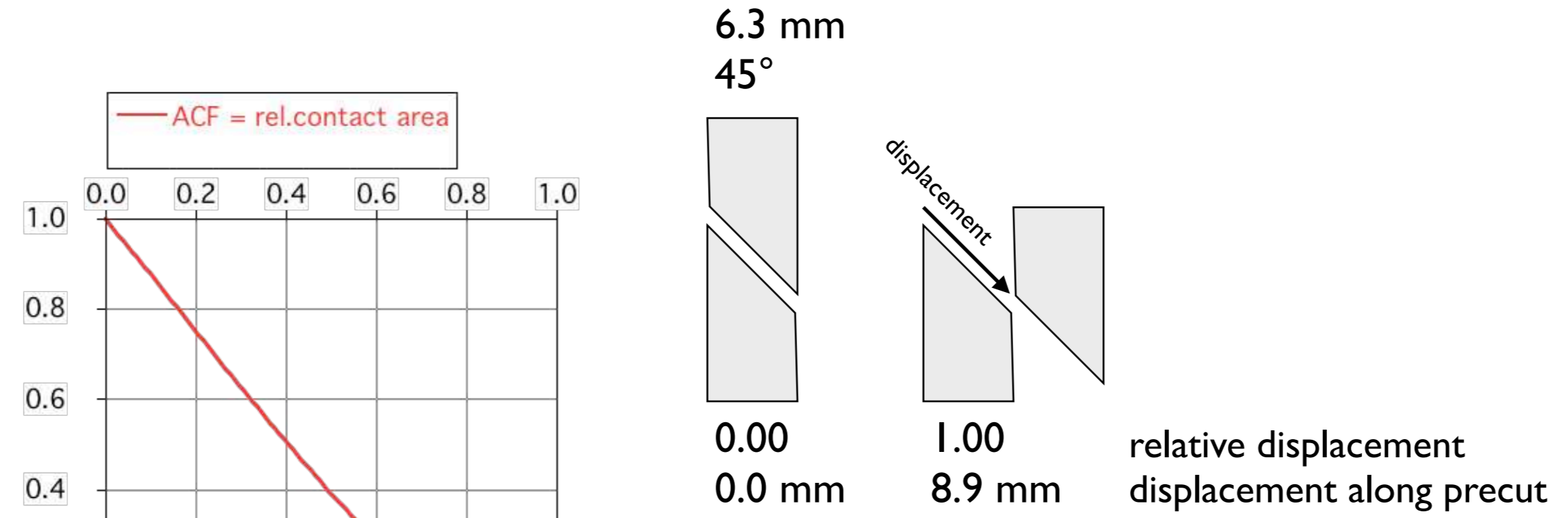
for coaxial experiments

- Two types of area correction:
- 1 = classical (Poisson): volume conserving homogeneous widening of sample
- 2 straight-sided cylinder deforms to barrel shape

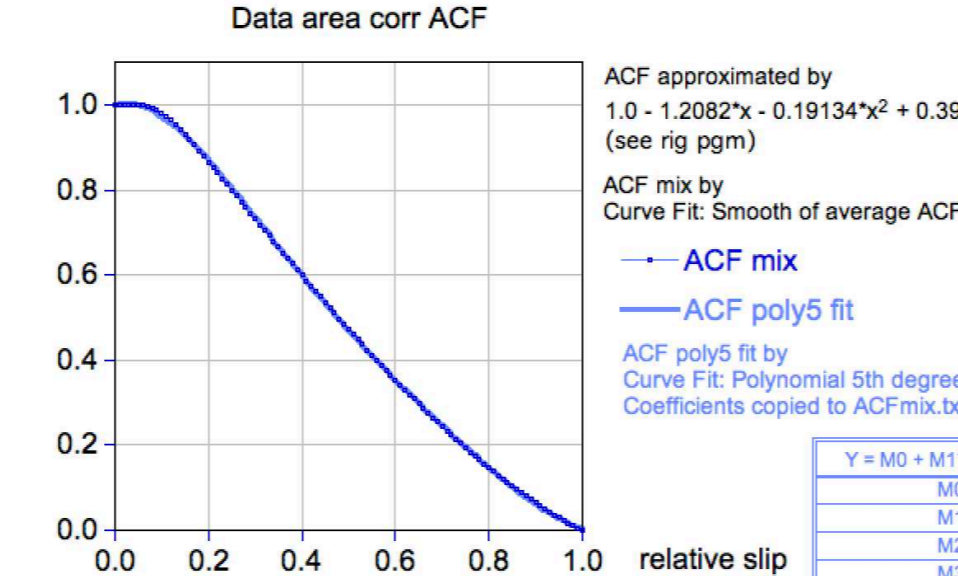
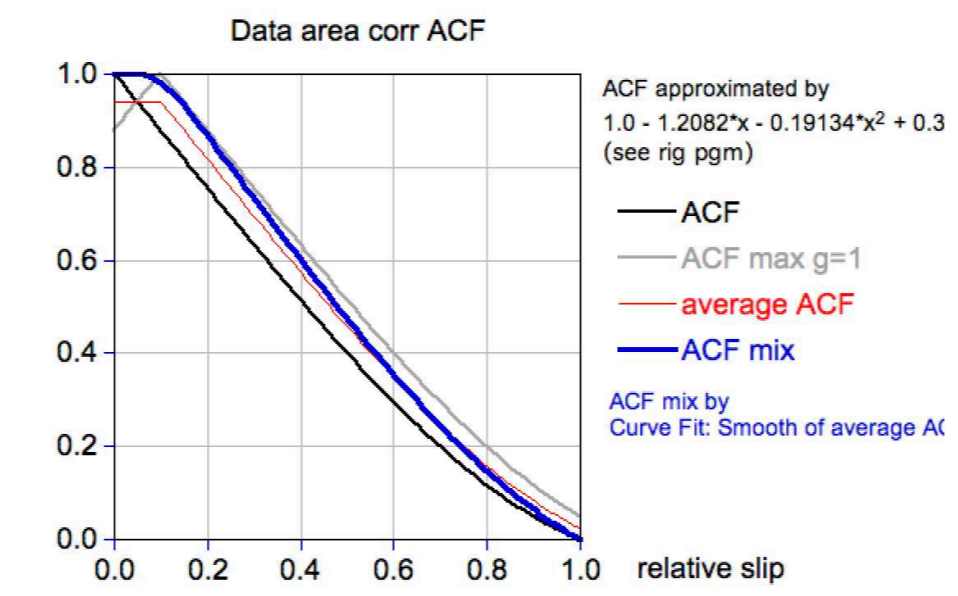


In coaxial set-ups, the net effect of the area correction is a displacement-dependent weakening.

for shearing experiments

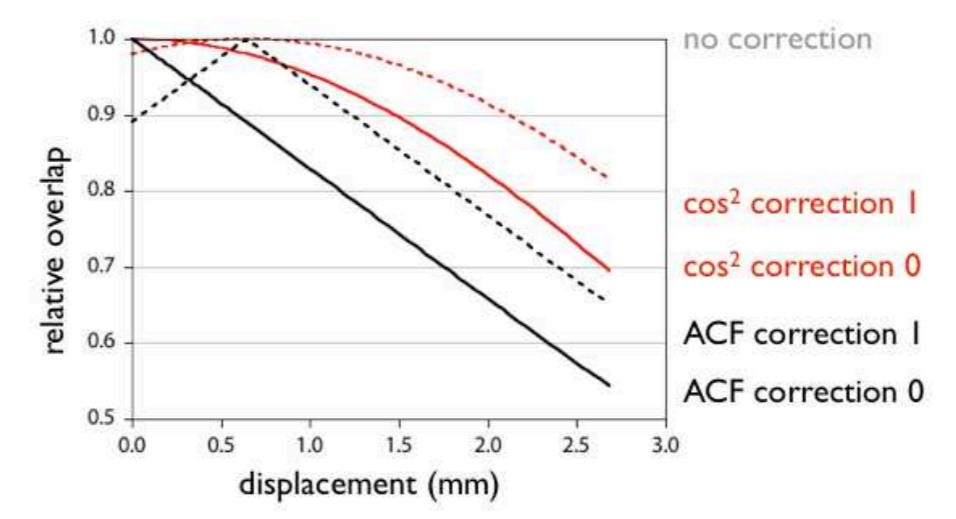
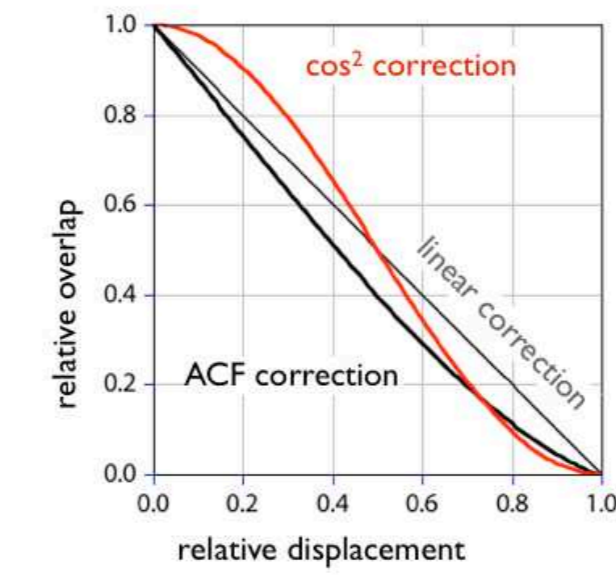
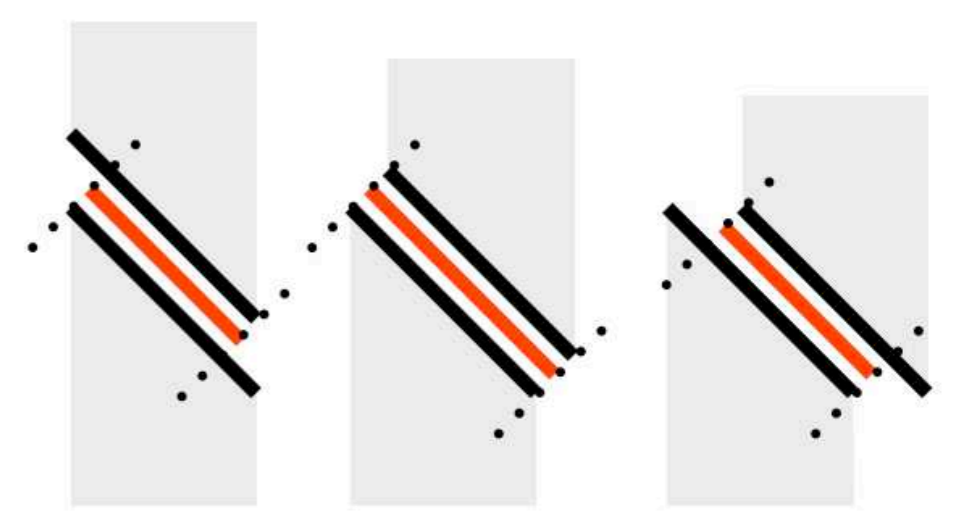
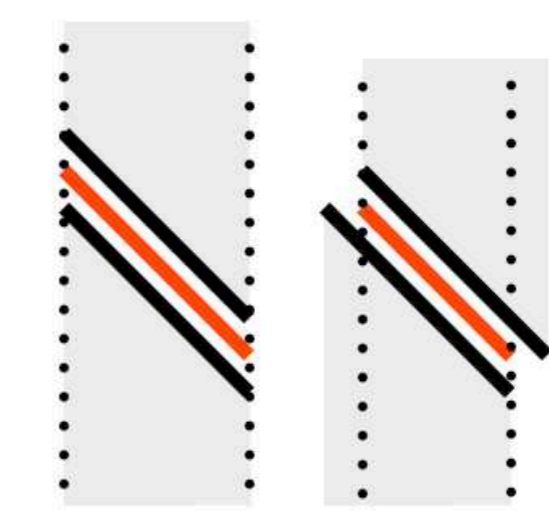


ACF approximation in Kaleidagraph (function ACF in rigS6)
 $c_2 = 1.00 - 1.2082 \cdot c_0 - 0.19134 \cdot c_0^2 + 0.39461 \cdot c_0^3$



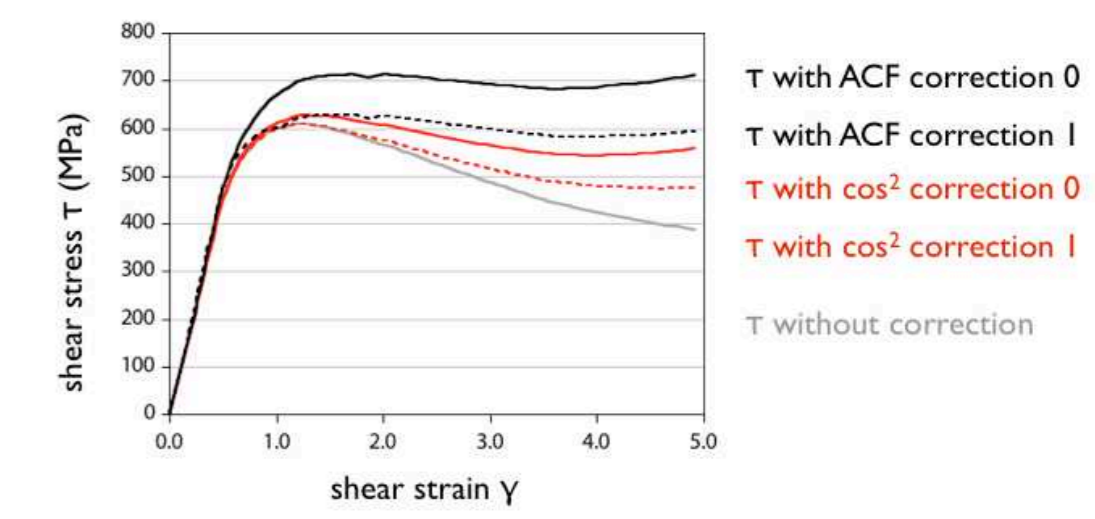
$Y = M_0 + M_1 \cdot x + \dots + M_5 \cdot x^5 + M_6 \cdot x^6$	
M0	1.0008
M1	0.33616
M2	-7.396
M3	14.545
M4	-12.895
M5	4.415
R	0.99996

In shearing set-ups, the net effect of the area correction is a displacement-dependent strengthening.



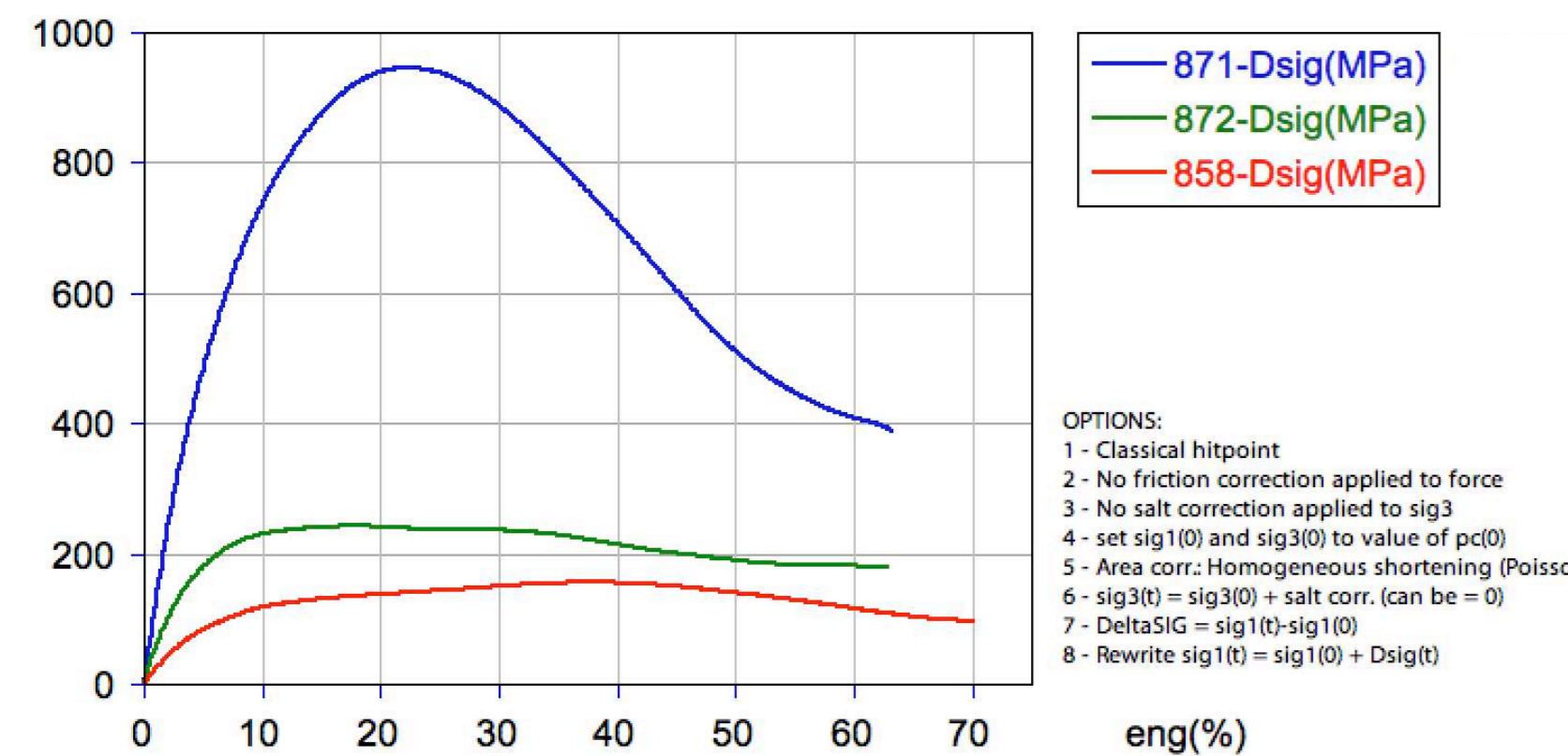
The alumina forcing blocks are assumed to be undeformed. The area correction applies only to the sample in the 45° pre-cut.

The correct function for the overlap is the autocorrelation function (ACF). However, to avoid the discontinuity at $\gamma = 1$, the ACF function is approximated by a \cos^2 - function.



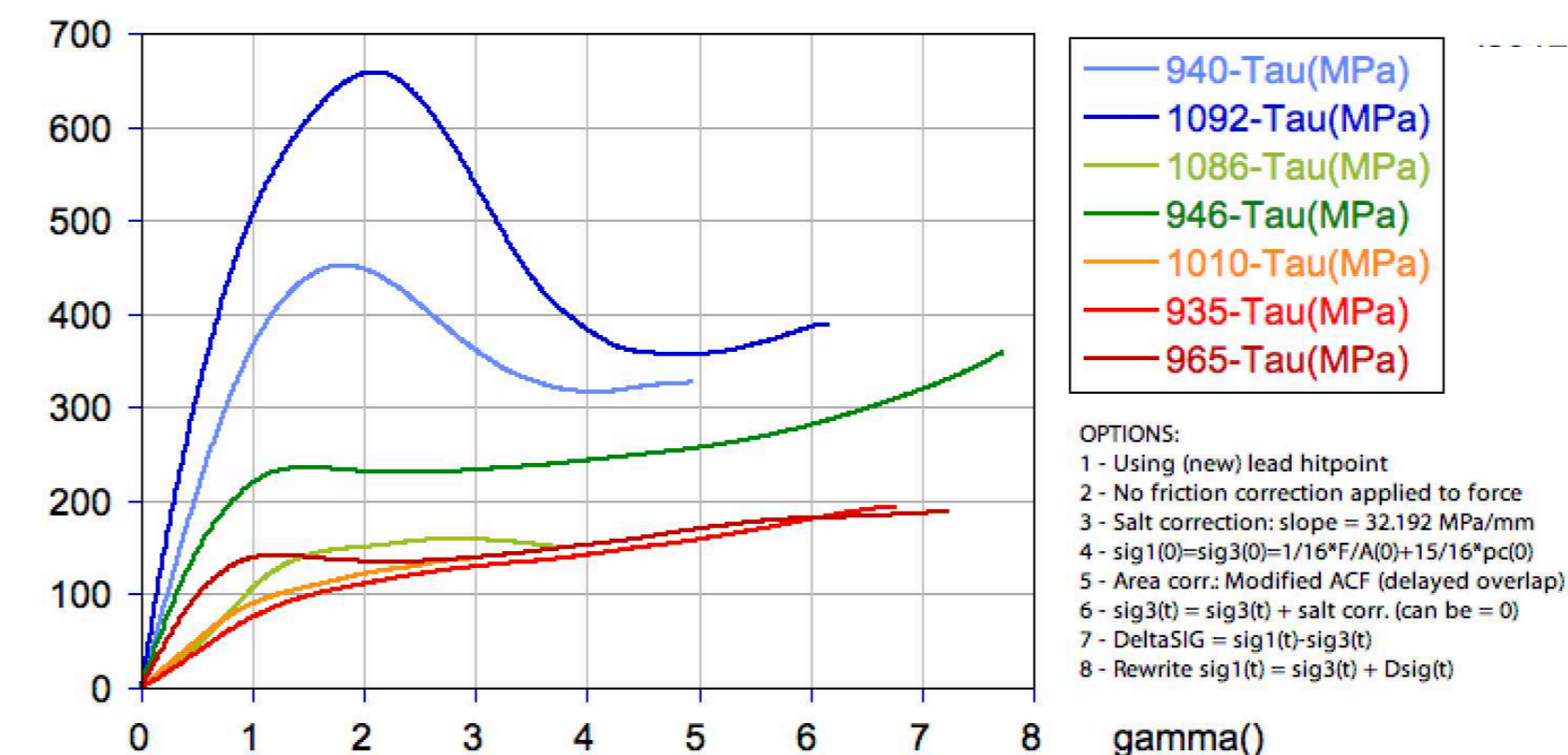
stress strain plots for different run options

coaxial experiments



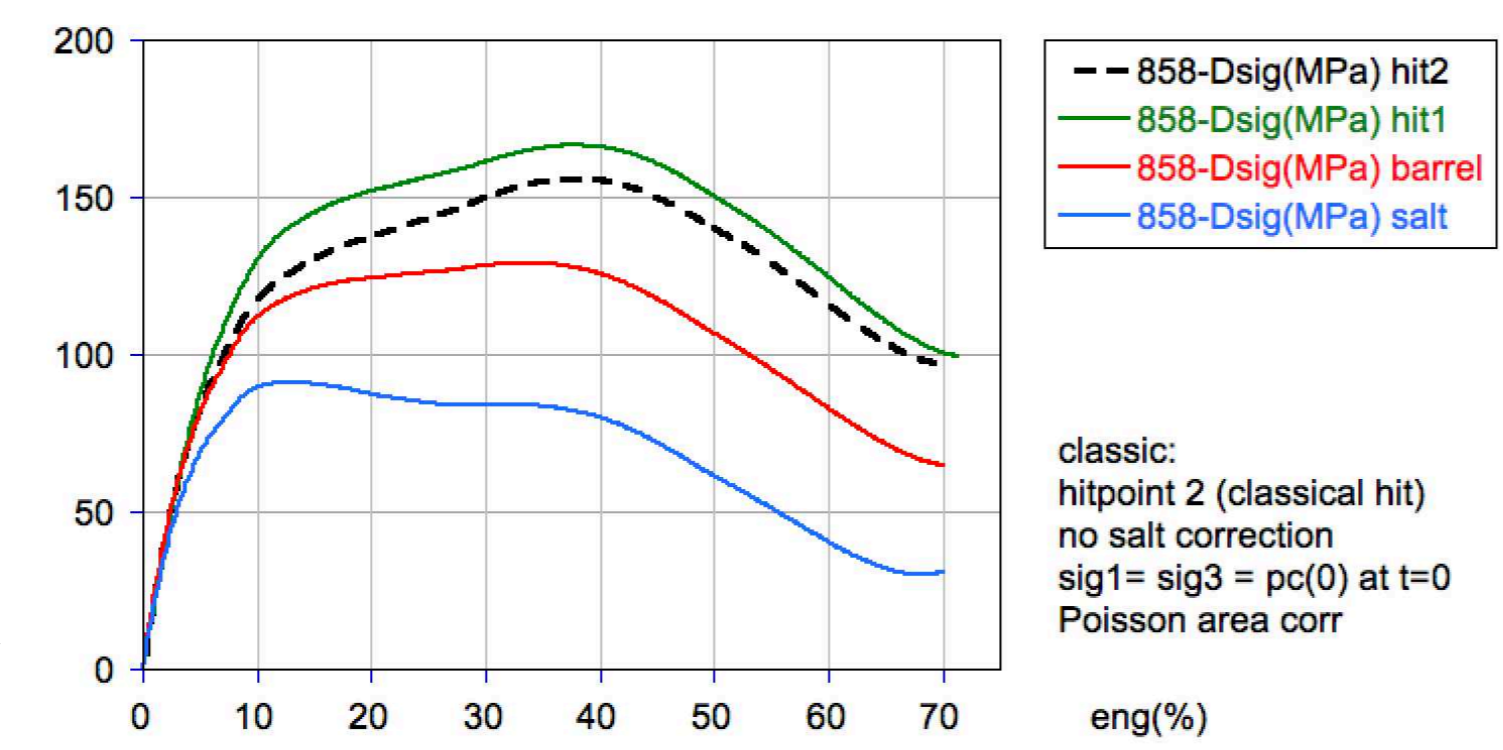
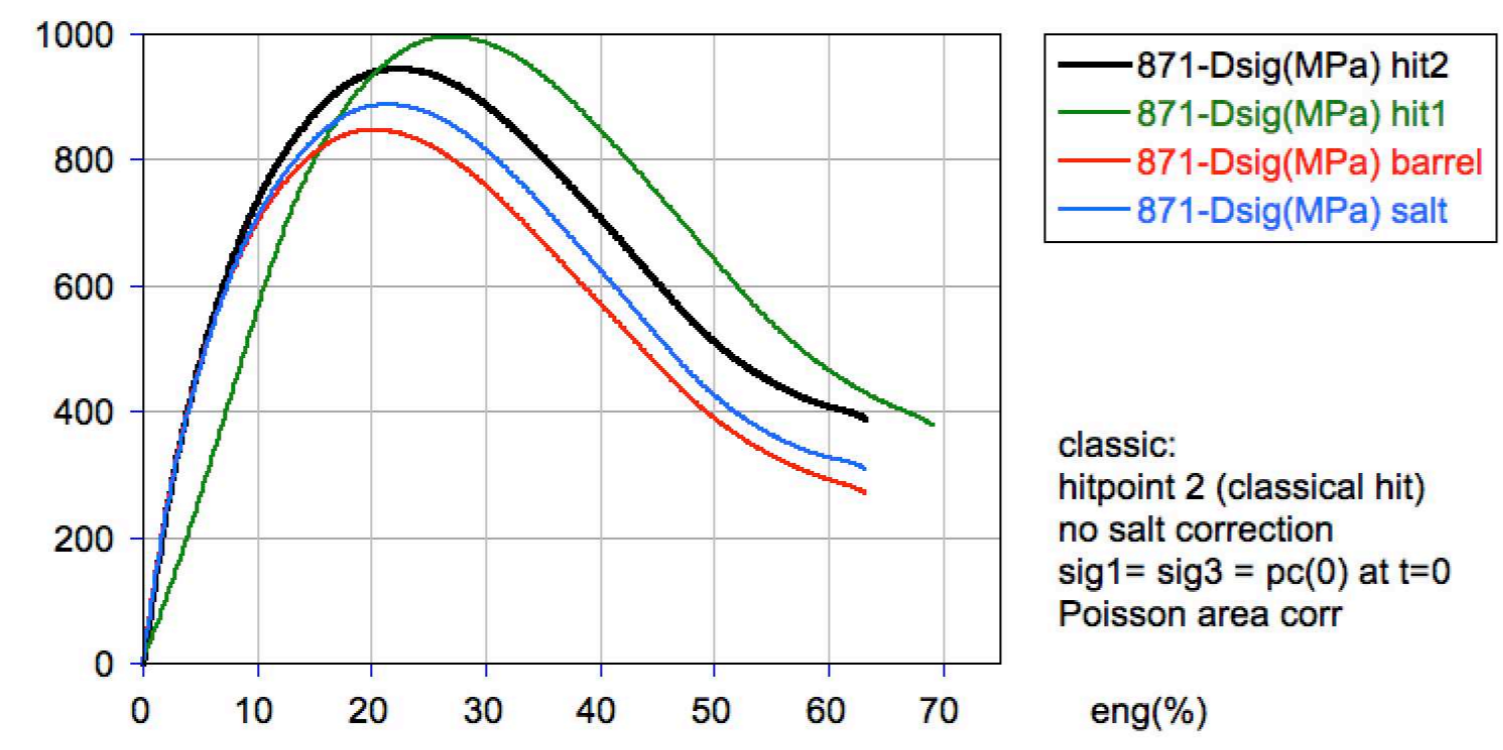
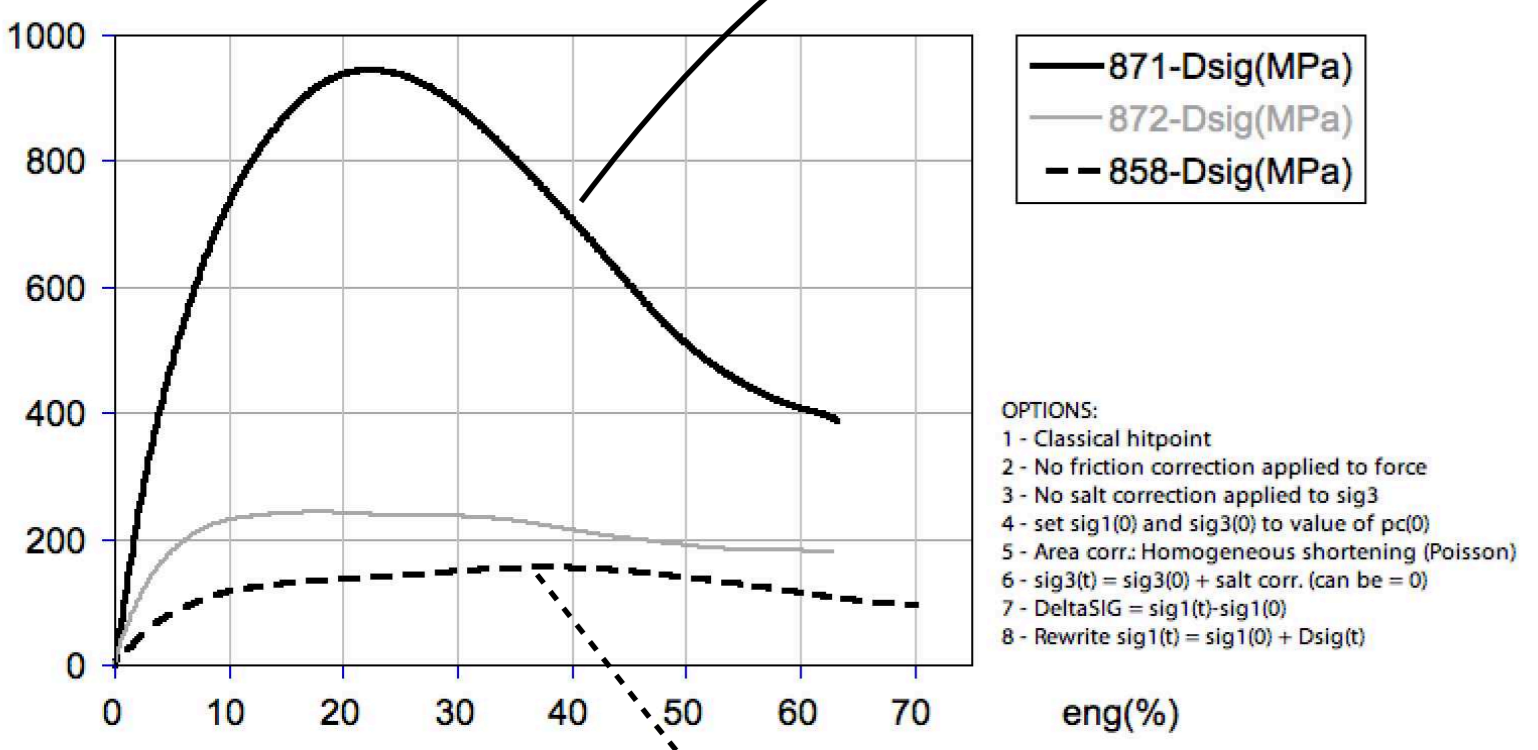
- COAX CLASSIC OPTIONS: denoted:
- 1 - Classical hitpoint 2
 - 2 - No friction correction applied to force 0
 - 3 - No salt correction applied to sig3 0
 - 4 - set sig1(0) and sig3(0) to value of pc(0) 1
 - 5 - Area corr.: Homogeneous shortening (Poisson) 1
 - 6 - sig3(t) = sig3(0) + salt corr. (can be = 0) 1
 - 7 - DeltaSIG = sig1(t)-sig1(0) 1
 - 8 - Rewrite sig1(t) = sig1(0) + Dsig(t) 1

general shearing experiments

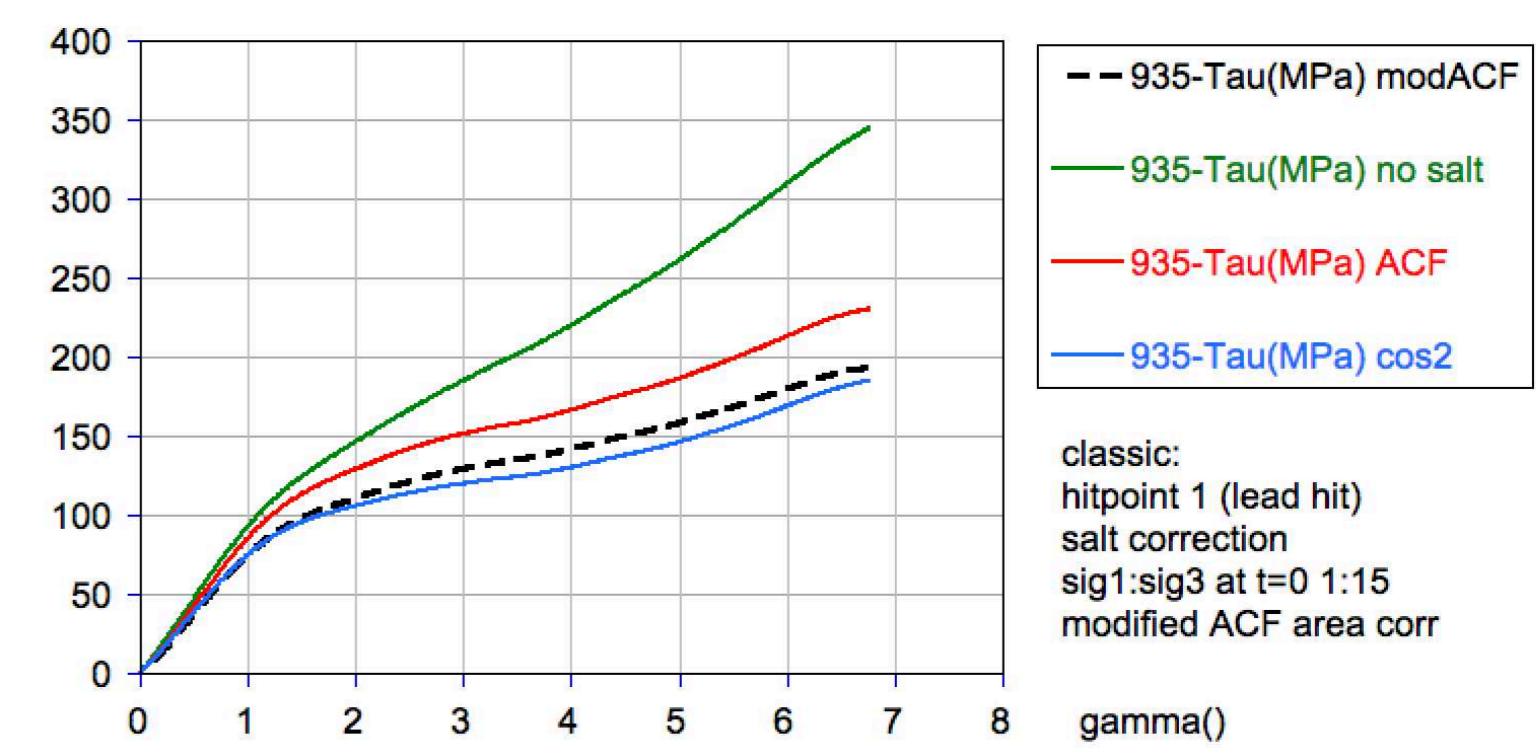
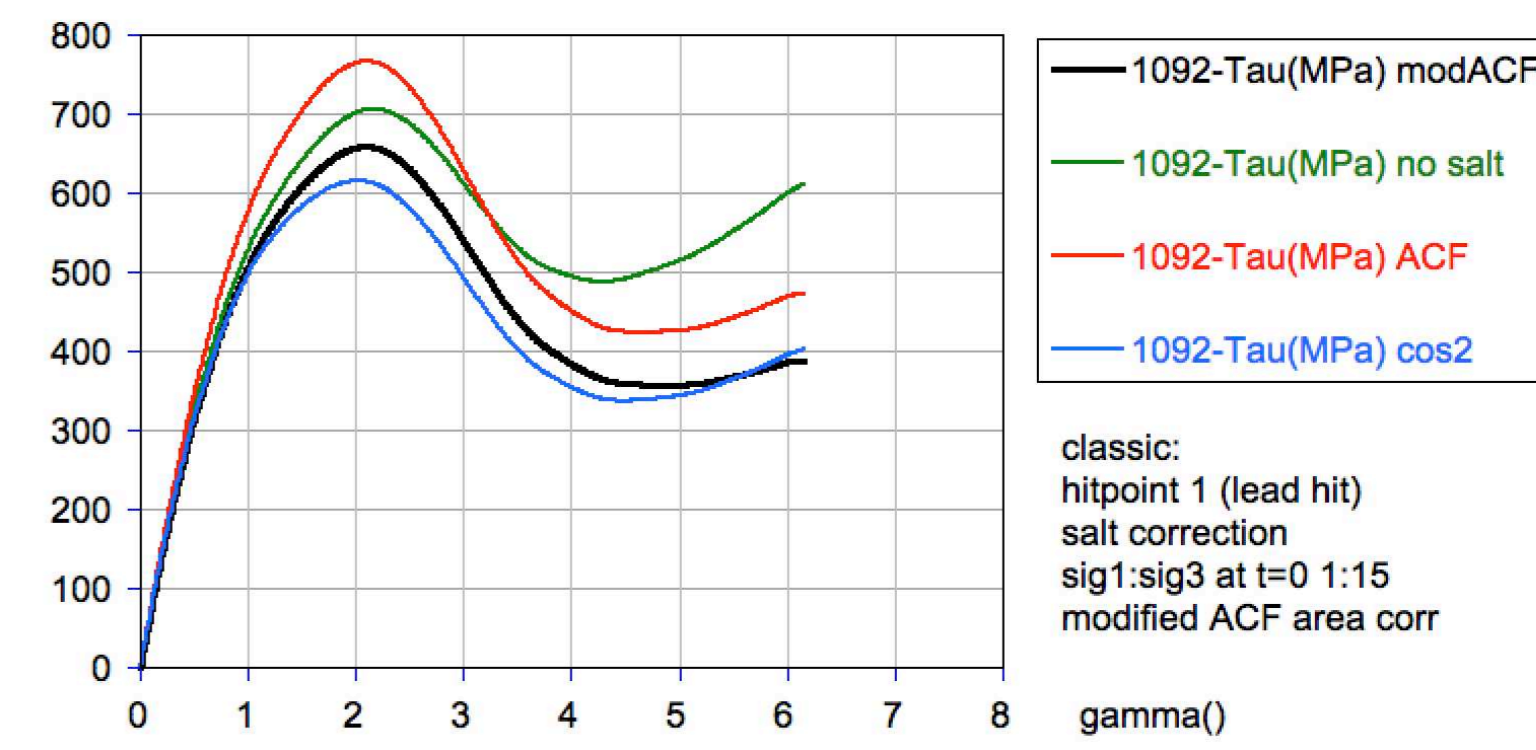
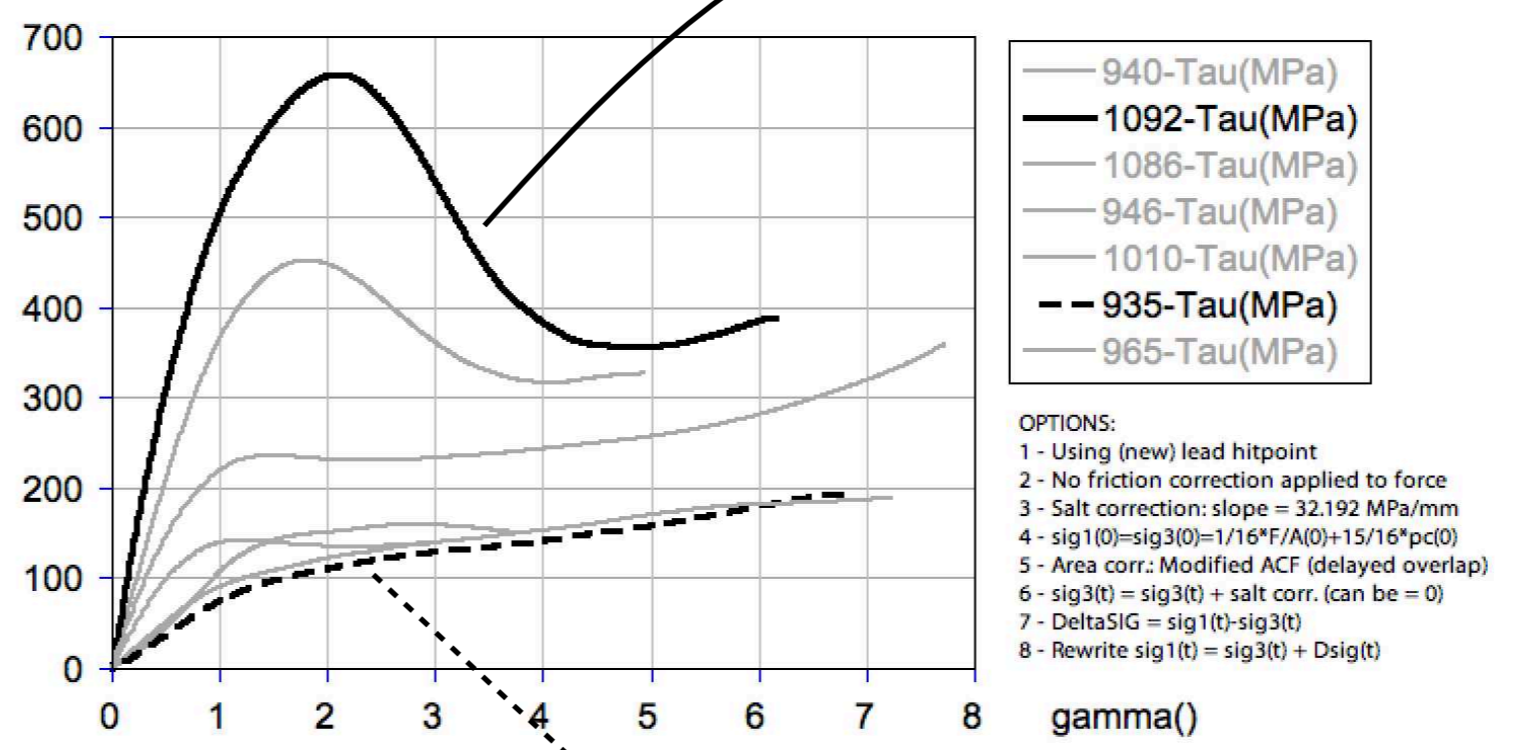


- SHEAR CLASSIC OPTIONS: denoted:
- 1 - Using (new) lead hitpoint 1
 - 2 - No friction correction applied to force 0
 - 3 - Salt correction: slope = 33.071 MPa/mm 2
 - 4 - sig1(0)=sig3(0)=1/16*F/A(0)+15/16*pc(0) 2
 - 5 - Area corr.: Modified ACF (delayed overlap) 2
 - 6 - sig3(t) = sig3(t) + salt corr. (can be = 0) 2
 - 7 - DeltaSIG = sig1(t)-sig3(t) 2
 - 8 - Rewrite sig1(t) = sig3(t) + Dsig(t) 2

effect of selecting:
hitpoint 1
barrel area correction
salt correction

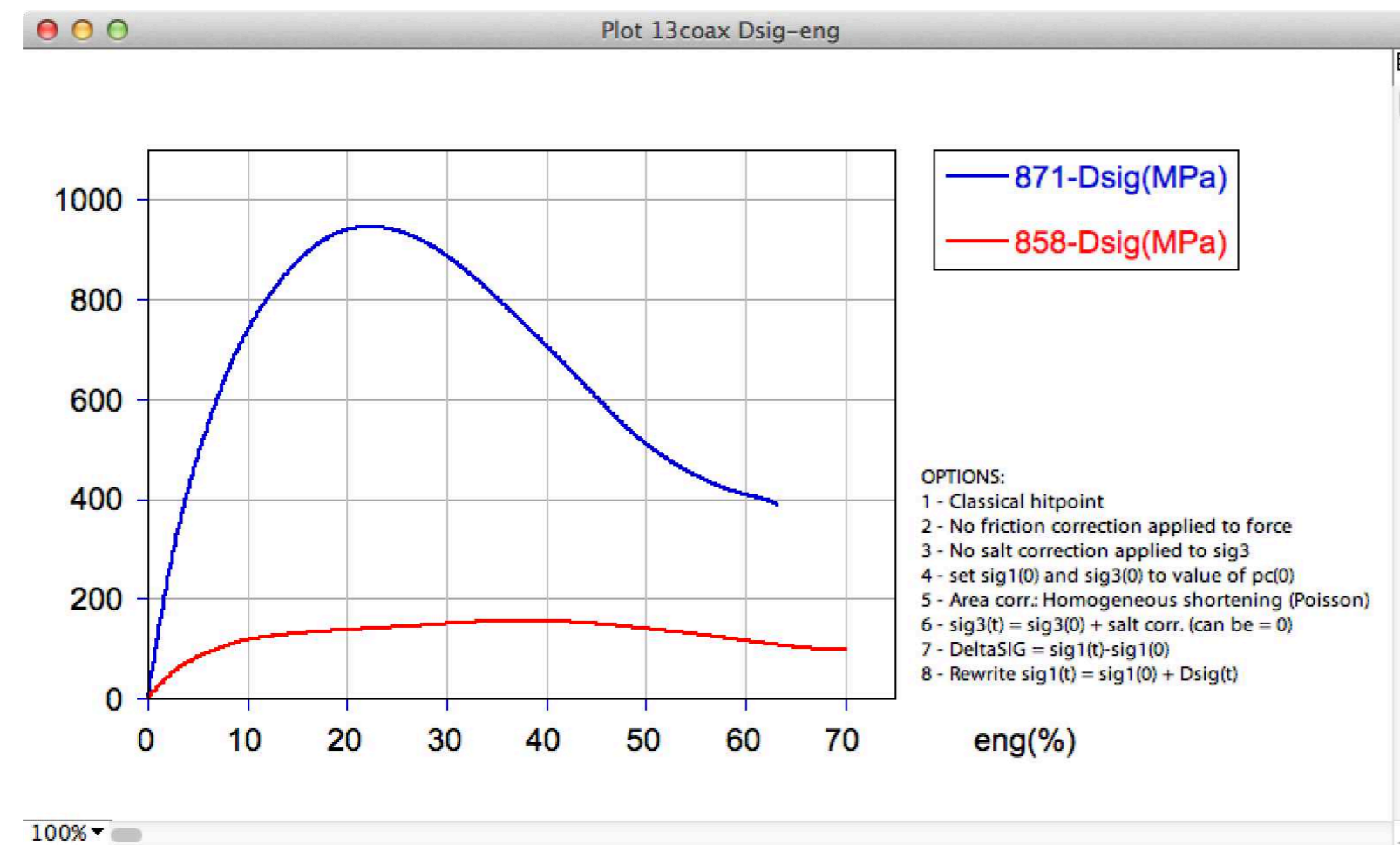


effect of selecting:
no salt correction
ACF area correction
cos² area correction

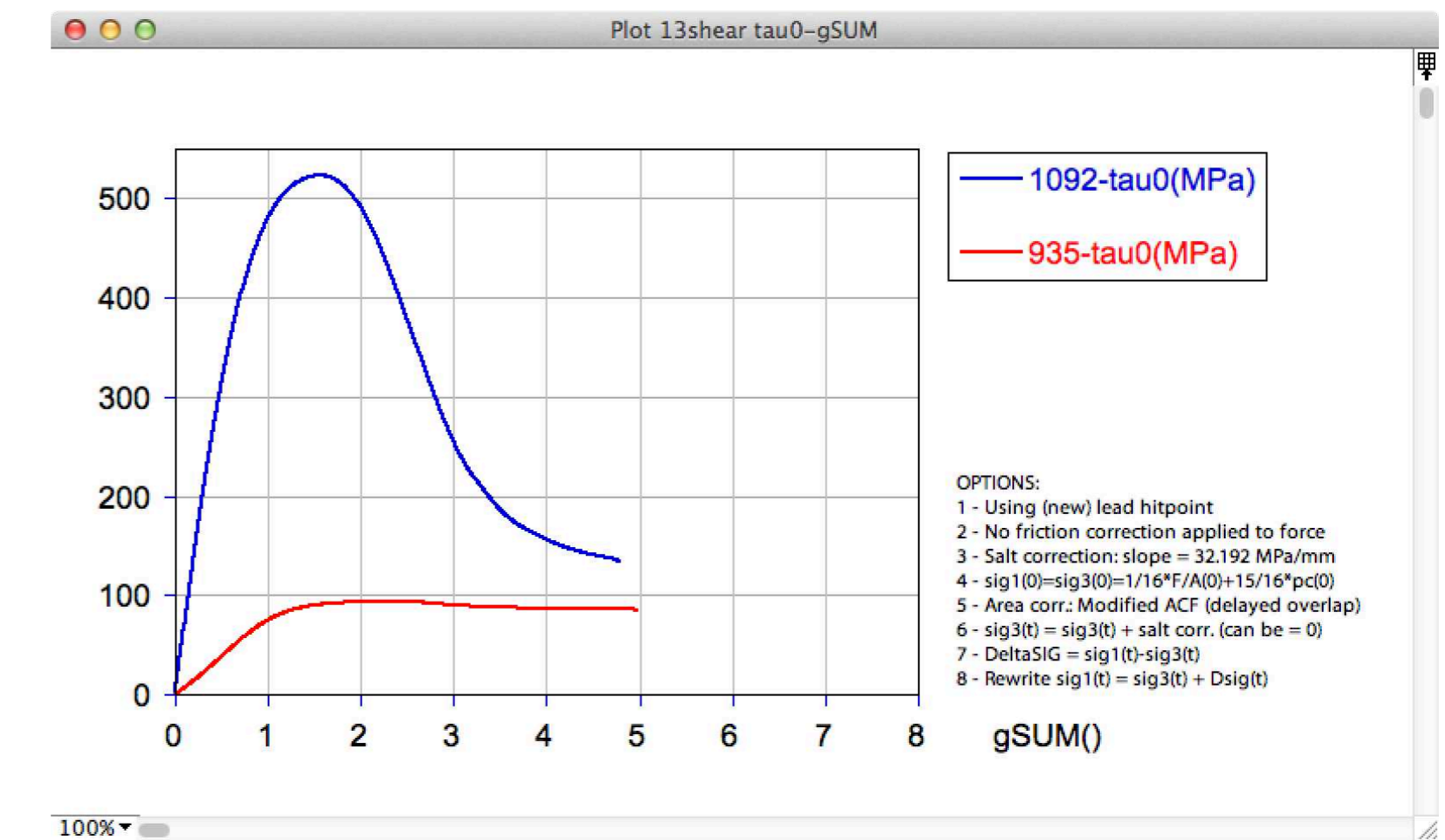
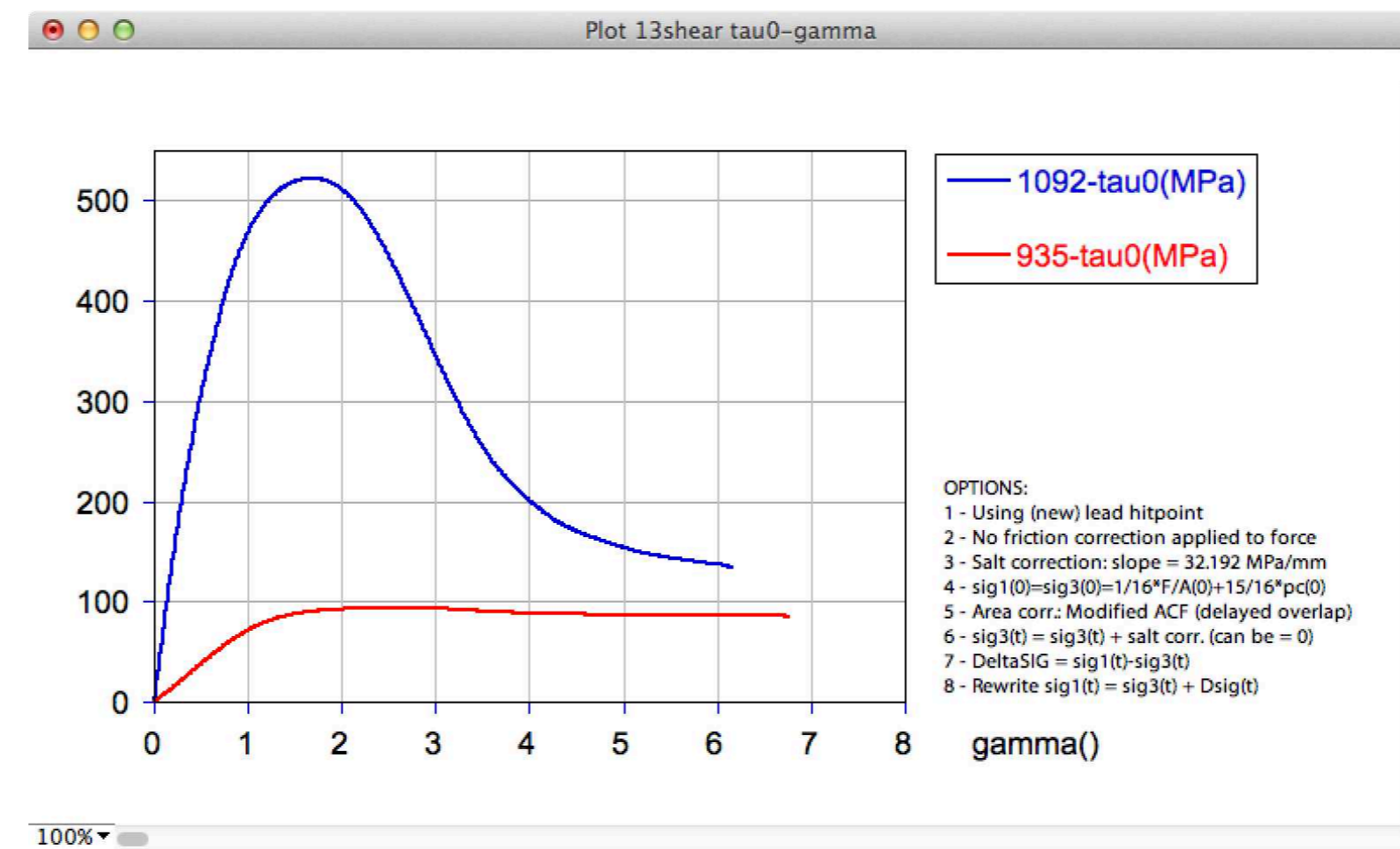


which 'strain' ?

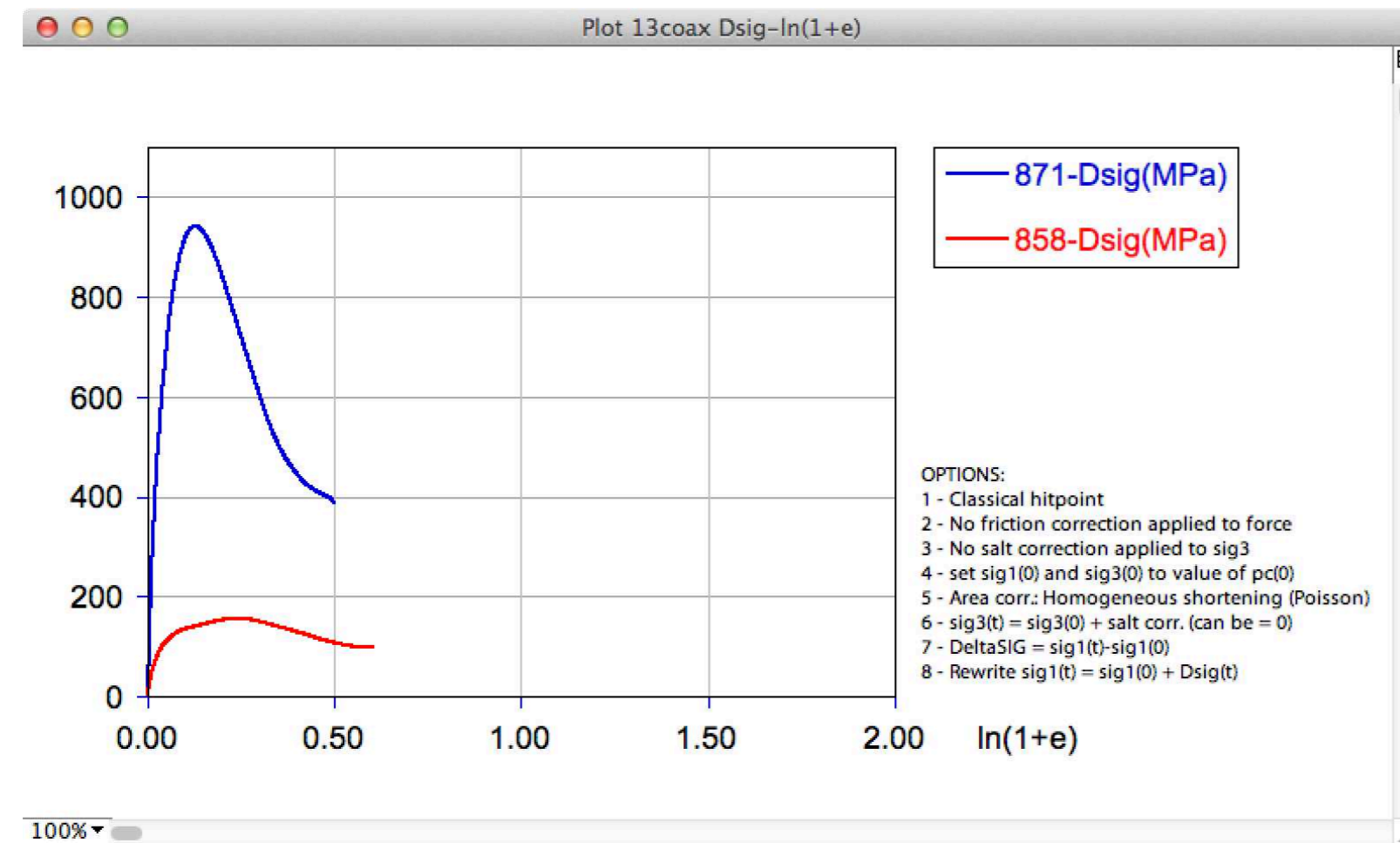
engineering strain



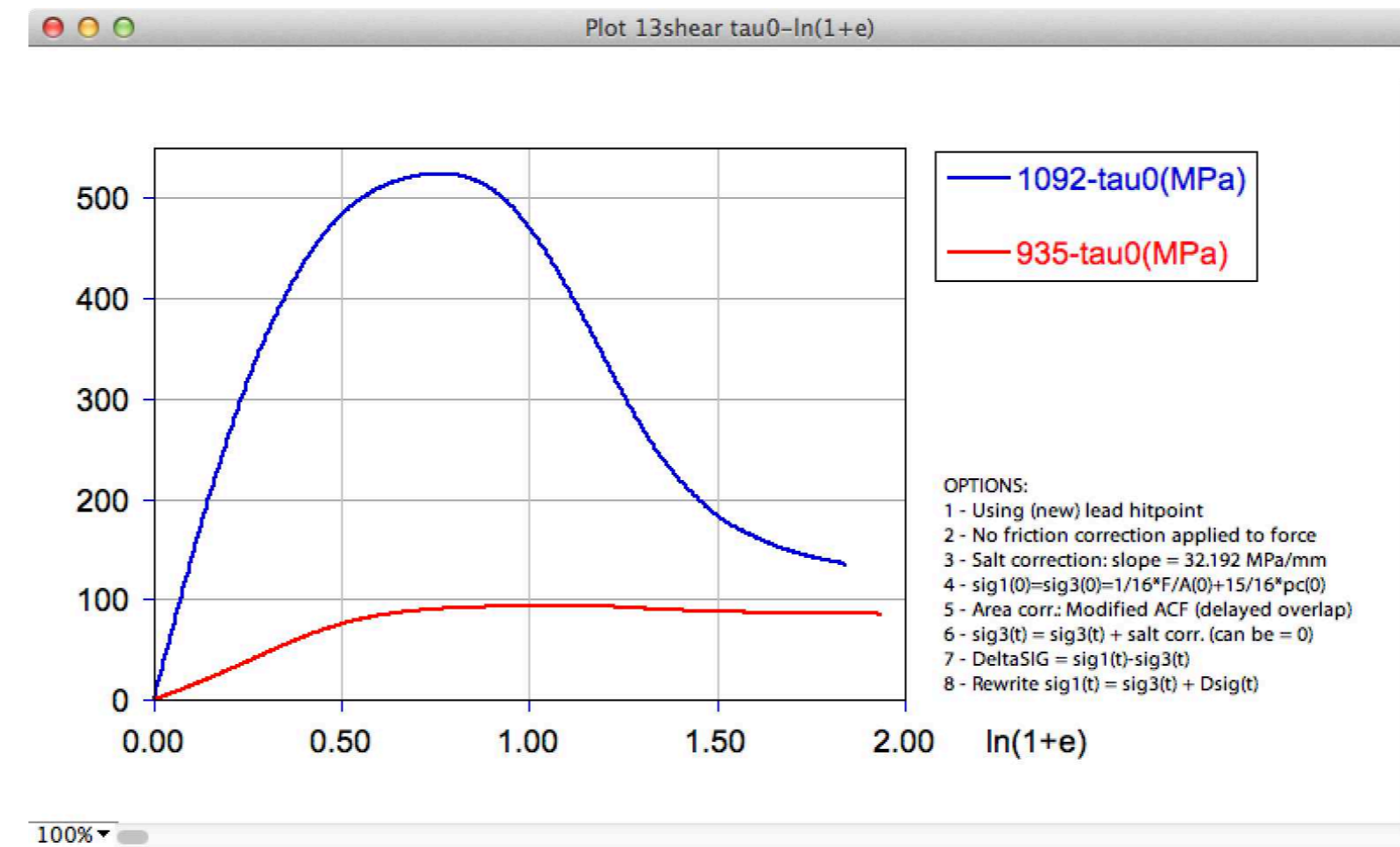
shear strain



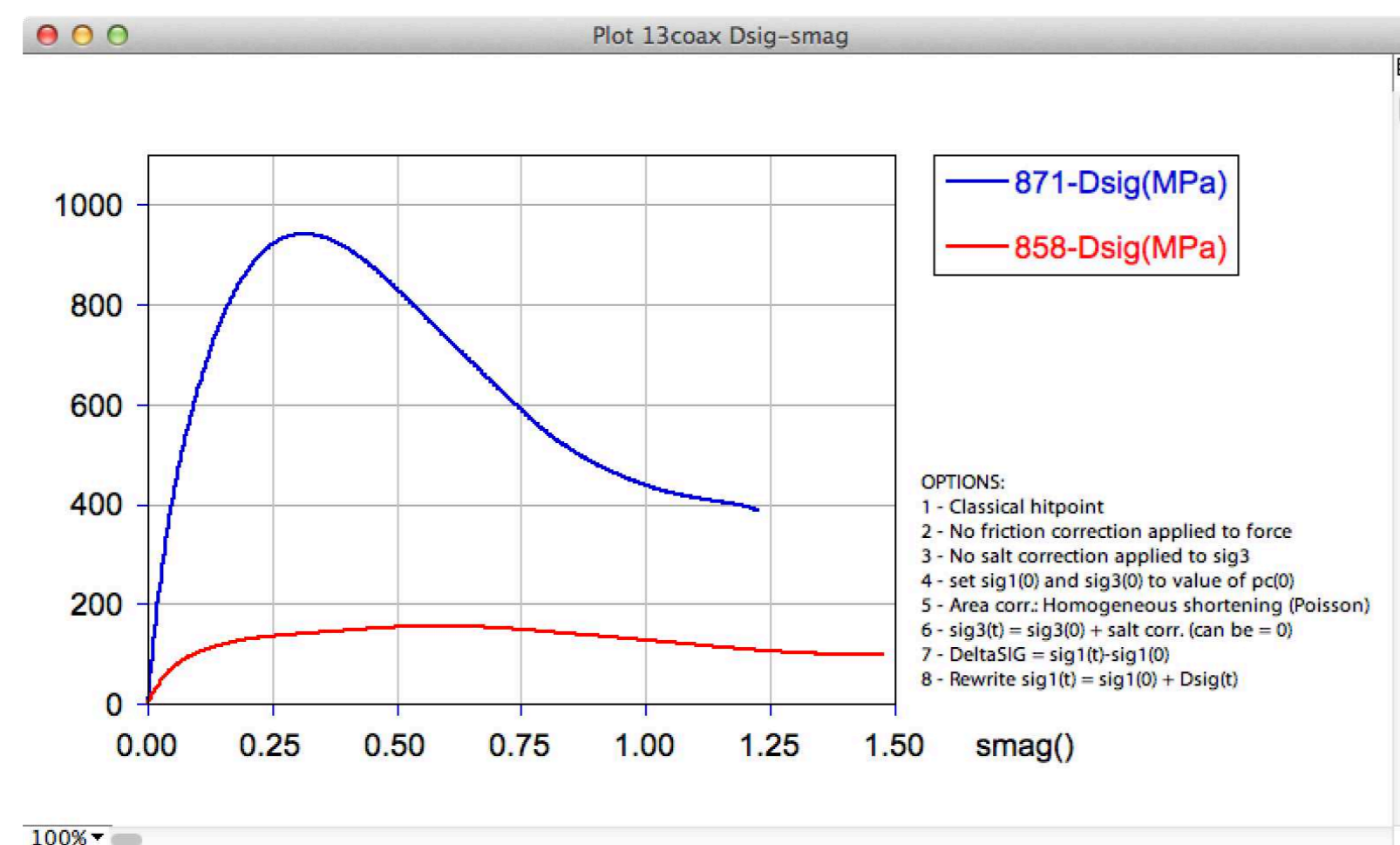
natural strain



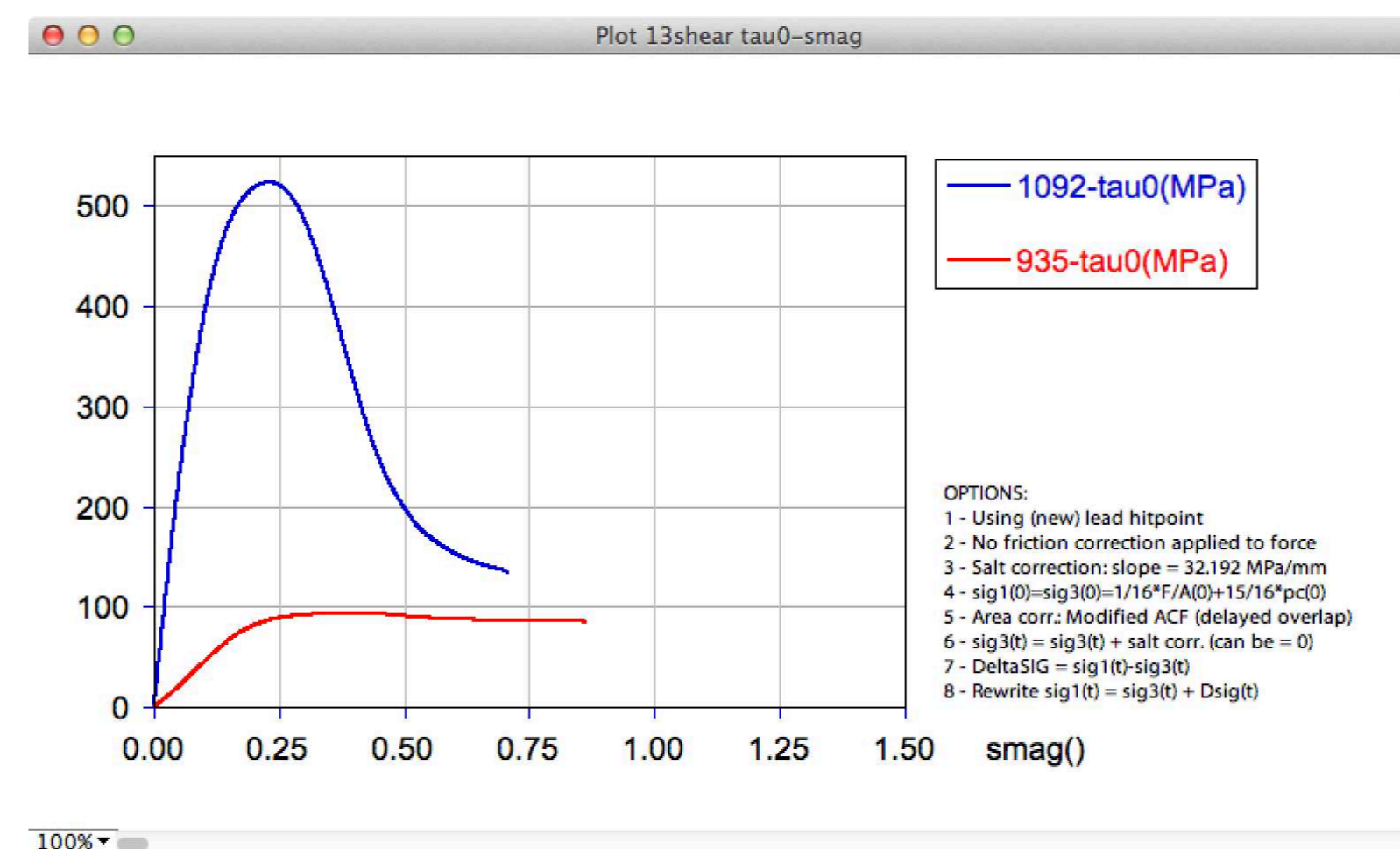
natural strain



strain magnitude



strain magnitude

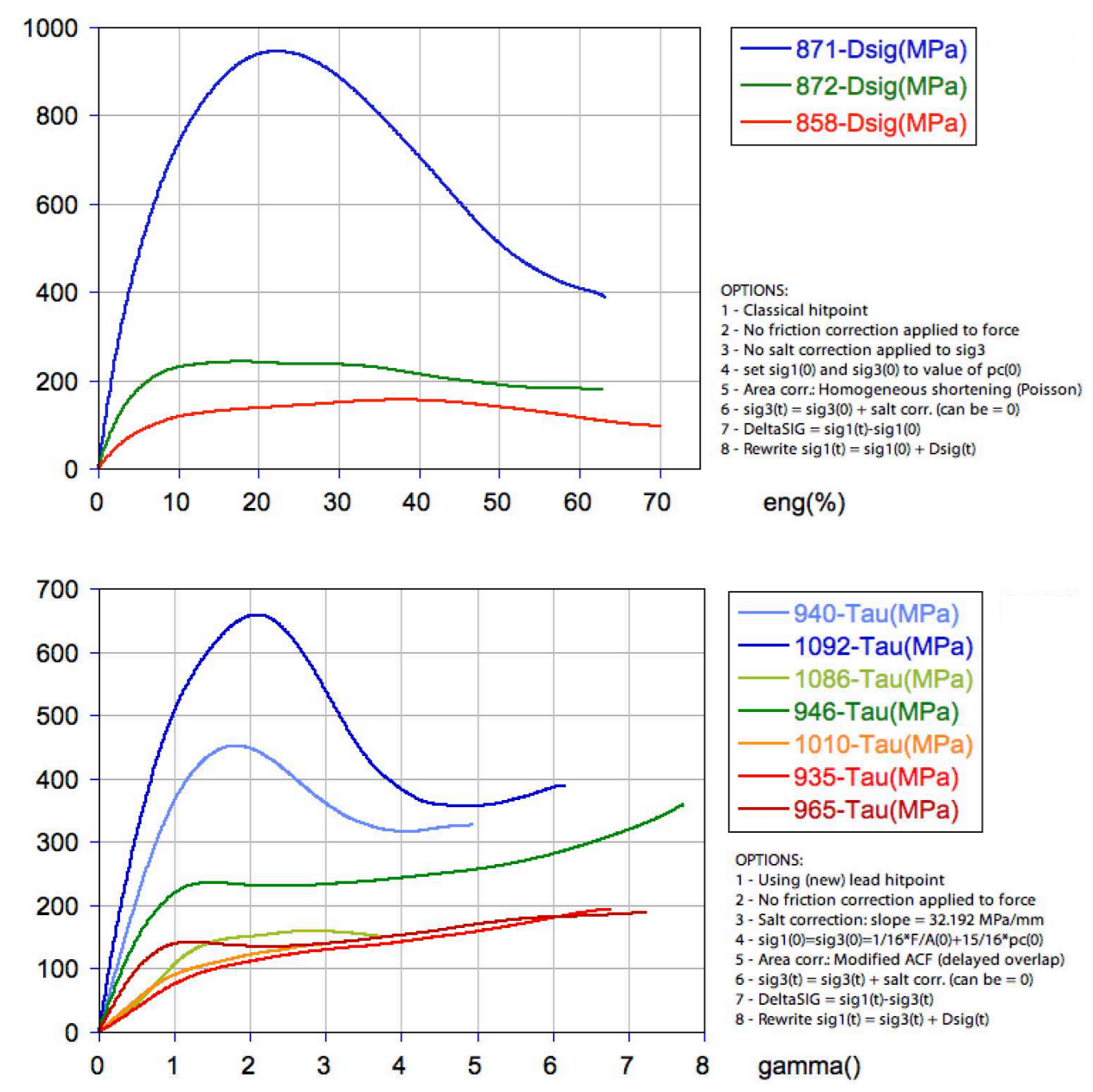


Changing the strain definition does not alter the calculated stress, but it severely changes the shape of the stress-strain curve.

When comparing coaxial and general shearing data, the choice of the 'equivalent' strain is important.

When using natural strain, shear experiments accumulate more strain, when using the strain magnitude (derived from the octahedral shear strain), the coax experiments achieve higher strains...

results !

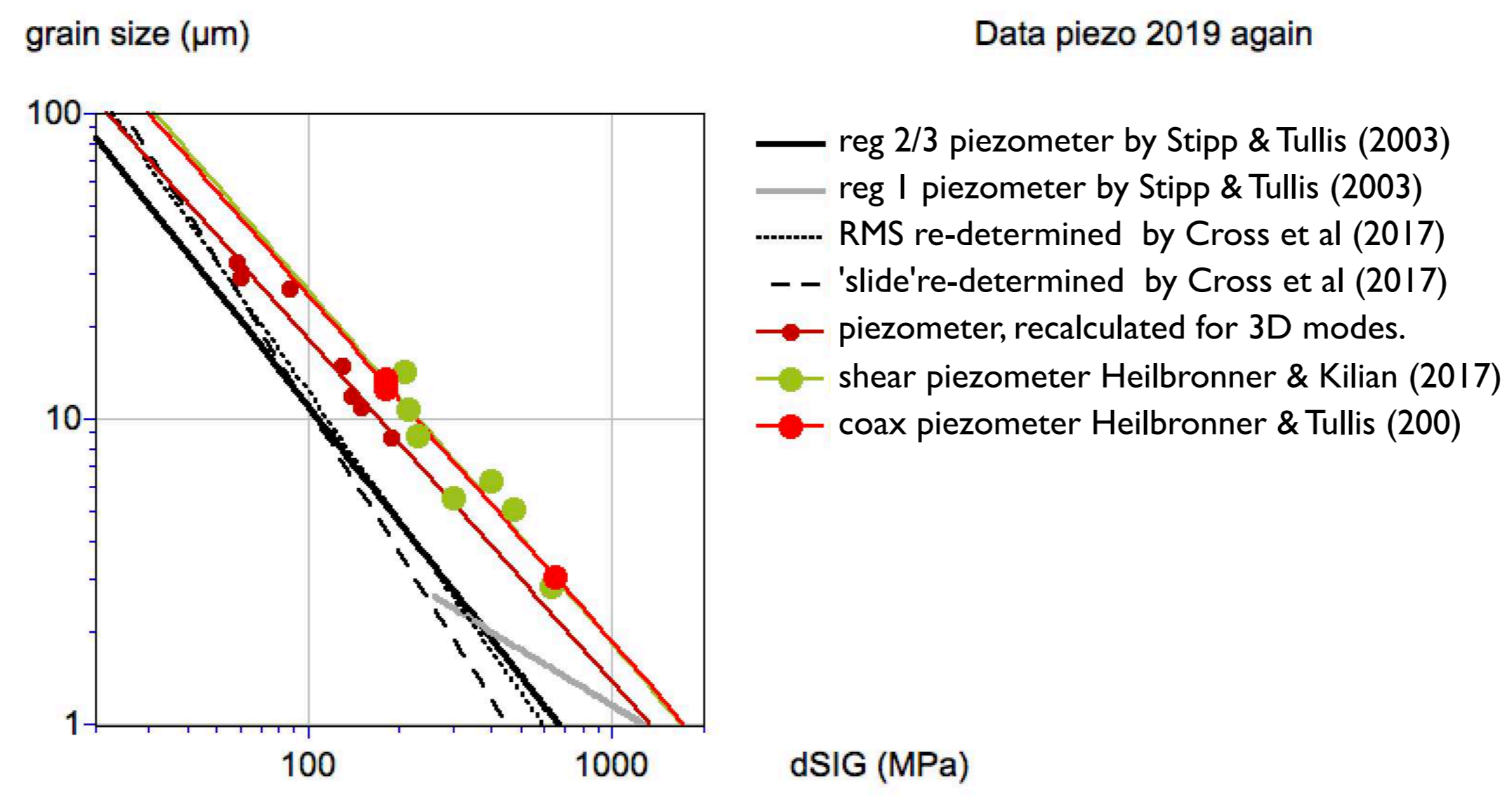


how to choose the flow stress ?

new run records	mode v(D) μm	$\Delta\sigma$ (2017)	$\Delta\sigma$ (2019)
1-w871			400
2-w872			240
3-w858			160
1a-w940	5.048	476	638
1b-w1092	3.843	628	712
2a-w1986	5.521	300	466
2b-w946	6.278	402	328
3a-w1010	8.752	230	286
3b-w935	14.182	206	274
3c-w965	10.714	214	274

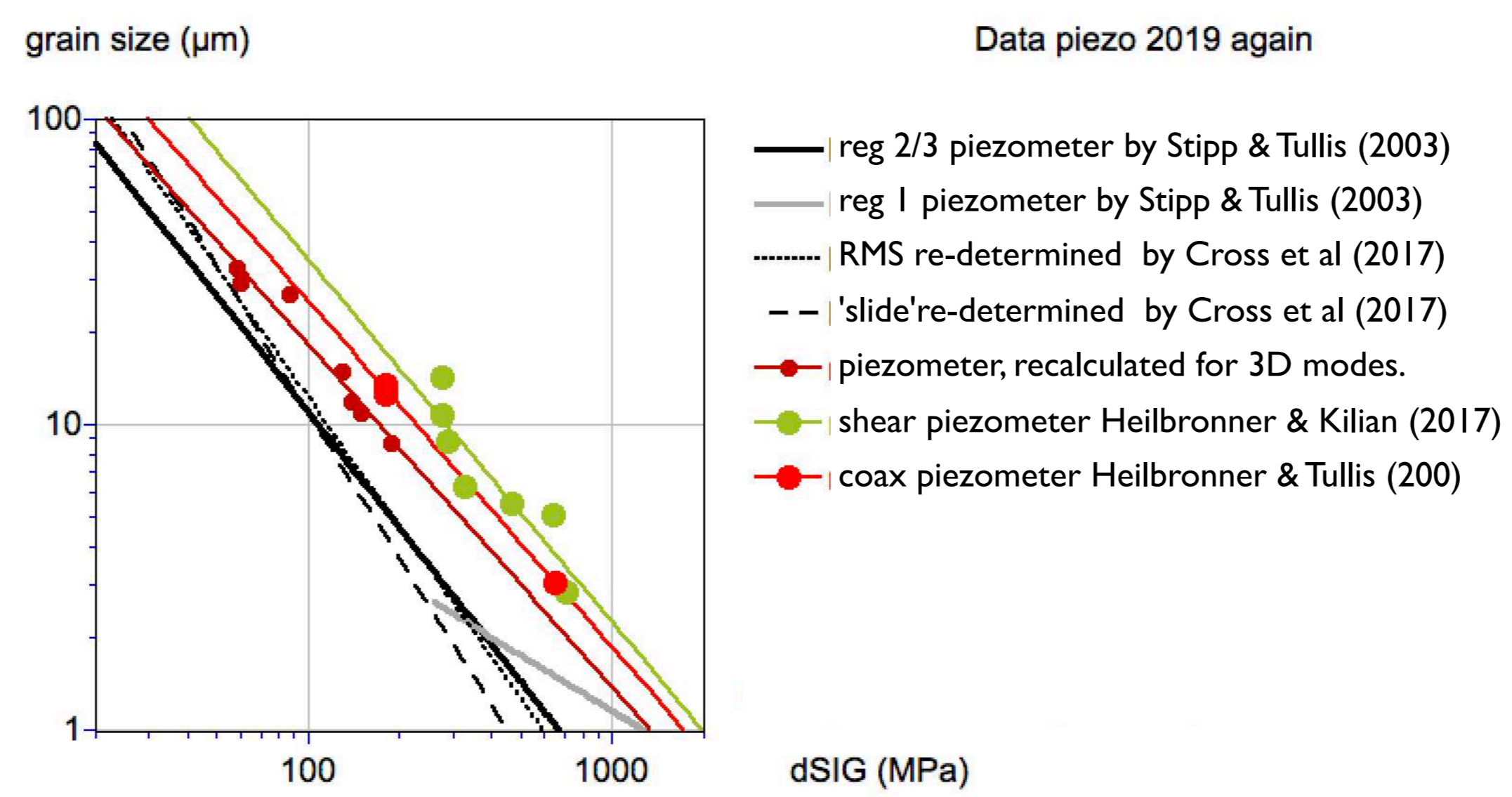
scanning effort by Leif Tökle, Brown University is gratefully acknowledged

2019 coax SSA added



published gs data shear (2017)
 + new gs w871 and w858
 + published stresses (2017, 2002)
 => in solid medium coax = shear
 => coax stronger in solid than molten

2019 stresses recalculated



published gs data shear (2017)
 + new gs w871 and w858
 + stresses from run record
 + recalculated for standard coax and standard shear
 => shear even stronger !
 => coax solidmedium same as molten