The solid medium deformation apparatus – reloaded

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EGU2020-9859 - Abstract

Rock deformation experiments are used to compile mechanical data sets for minerals and rocks and to study microstructure and texture development.

The Griggs apparatus, a solid medium piston cylinder machine was designed about 60 years ago to investigate rock deformation mechanisms and rheology at elevated confining pressures. In a typical experiment today, the confining medium is NaCl, with confining pressures up to 3 GPa, temperatures up to 1100°C, and displacement rates between 10-8 and 10-2 ms-1 (equivalent to strain rates of 10-7 to 10-3 s-1). In axial tests, the cylindrical samples are 12 to 15 mm long with a diameter of 0.625 mm. In shearing test, split cylinder assemblies are used with 0.5 to 1 mm thick samples introduced along the 45° pre-cut. Reasonable total strains are limited to 30% axial shortening or shear strains of gamma 4. (Higher strains can be attained but are difficult to analyse mechanically. Unlike for gas rigs, torsion is not available for solid medium machines).

As of now, the operational fleet of solid medium deformation apparatus comprises worldwide over 20 machines in different labs (mainly in Europe, U.S.A. and Japan), providing the scientific community with an ever-growing rheological and microstructural data base.

Participants of the Orléans Workshop on Experimental Solid Medium Rock Deformation, January 30-31, 2020

In view of numerous developments in experimental design, as well as improvements of hardware and software for data acquisition and processing, the experimental community was recently invited to a two-day workshop, hosted by the experimental group of Orléans University.

The main goal was to discuss the following points:
- how to further improve the apparatus, increase its scope and improve calibrations;
- how to further improve data processing, and the precision and reliability of the results;
- how to maintain consistency among the labs and through time (backwards compatibility); - how ensure compatibility of results from axial and shearing experiments;
- how to make the data available to the community.

EGU2020-9859 - Display

This display focuses on the software used for converting the recorded experimental data to stress-strain curves. In particular, on the choices that have to be made on the way and how they influence the results. It is proposed to make every step transparent such hat different labs publish coherent results.

abstract | overview | >

converting experimental data to stress-strain curves ...

... in a transparent fashion

general procedure

go to software – rigP(prepare), rigC(for axial) and rigS(for shear)

pre-process – create input file

go to prepare raw data from experimental record

run program with explicit options:

option A go to crop data for analysis – select hitpoint

option B go to perform 'friction' correction ... if you must

option C go to 'area corrections' – for axial and shear experiments

option D go to σ_3 during the experiment – the salt correction

option E go to σ_1 and σ_3 at the start of the experiment

go to summary of options – corrections and calculations

software – rigP(prepare), rigC(for axial) and rigS(for shear)

prepare input

rigP

necessary input:

- run record (machine data)
- metadata
  - apparatus
  - experimental conditions
  - sample geometry
  - sample assembly

output:

- raw data file of complete run (SI units)
- input file for rigC and rigS:
  reduced file length (max = 1000 pts)
  (smoothing of data not yet implemented)
  includes both hitpoints (classical and 'lead')

analyze data

rigC – for axial experiments

1. open input file
2. asks for options
   - A choice of hitpoint
   - B 'friction correction' – Y/N
   - C area correction
   - D confining pressure correction
   - E set starting values for σ_1 and σ_3
3. data is read from hitpoint to end (option A)
4. stiffness correction of d ⟶ dc
5. strains and strain rates are calculated
6. friction' correction of F ⟶ Fc (option B)
7. calculate cross sectional area (option C)
8. define σ_3 and slope (ΔMPa/mm) (option D)
9. calculate $\Delta\sigma$ = (Fc - Fc(0)) / area
10. determine $\sigma_1(0)$ and $\sigma_3(0)$ at start (option E)
11. derive $\sigma_1 = \sigma_3(0) + \Delta\sigma$
12. calculate mean stresses
13. calculate equivalent viscosity
14. create output file

rigS – for shear experiments

1. open input file
2. asks for options
   - A choice of hitpoint
   - B 'friction correction' – Y/N
   - C area correction
   - D confining pressure correction
   - E set starting values for σ_1 and σ_3
3. data is read from hitpoint to end (option A)
4. stiffness correction of d ⟶ dc
5. shear strains, shear strain rates are calculated
6. friction' correction of F ⟶ Fc (option B)
7. calculate overlap area (option C)
8. define σ_3 and slope (ΔMPa/mm) (option D)
9. calculate $\Delta\sigma$ = (Fc - Fc(0)) / area
10. determine $\sigma_1(0)$ and $\sigma_3(0)$ at start (option E)
11. derive $\sigma_1 = \sigma_3(0) + \Delta\sigma$ (inside shear zone)
12. calculate mean stresses
13. calculate τ and σ_n (inside shear zone)
14. calculate equivalent viscosity
15. create output file

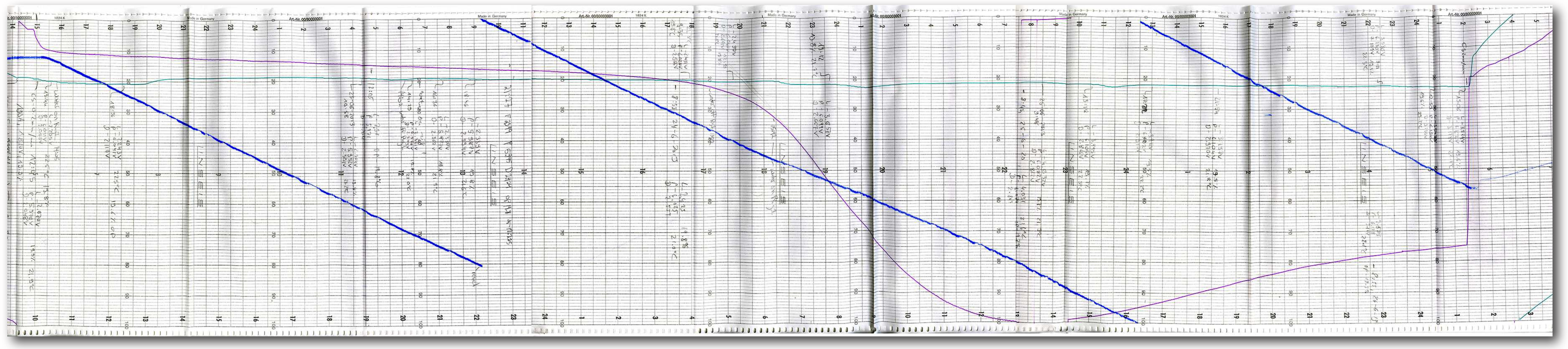
explicit options

```
*--- A-select hitpoint

        write(6,'(a)') 'Select hitpoint (1=classical, 2=new(=lead)) '
        read(5,*) ioptionHITP

*--- B-select friction correction

        write(6,'(a)') 'Friction correction for F ? (1=yes  0=no)'
        read(5,*) ioptionFRIC

*--- C-choice of area correction (Poisson correction)

        write(6,'(a)') 'Options for area correction'
        write(6,'(a)') '0: No area correction'
        write(6,'(a)') '1: Homogeneous shortening of sample '
        write(6,'(a)') '2: Barreling of sample'
        read(5,*) ioptionAREA

*--- D-definition of sig3

        ioptionSALT=0
        write(6,'(a)') 'Definition of sig3(t)'
        write(6,'(a)') '1: sig3(t) = Pc(0) at start'
        write(6,'(a)') '2: sig3(t) = Pc(0) + SALT correction'
        write(6,'(a)') '3: sig3(t) = Pc(t) as measured'
        read(5,*) ioptionSIG3

*------ salt correction is only possible for ioptionSIG3=2
        if(ioptionSIG3.eq.2) ioptionSALT=1


*--- E-definition of sig1 and sig3 at start of experiment (time=0)

        write(6,'(a)') 'Defining sig1(0) and sig3(0) at time=0'
        write(6,'(a)') '1: sig1(0)=sig3(0)=pc(0)'
        write(6,'(a)') '2: sig1(0)=sig3(0)=1/16*F(0)/A(0)+15/16*pc(0)'
        write(6,'(a)') '3: sig1(0)=F(0)/A(0) and sig3(0)=pc(0)'
        read(5,*) ioptionSTART

```

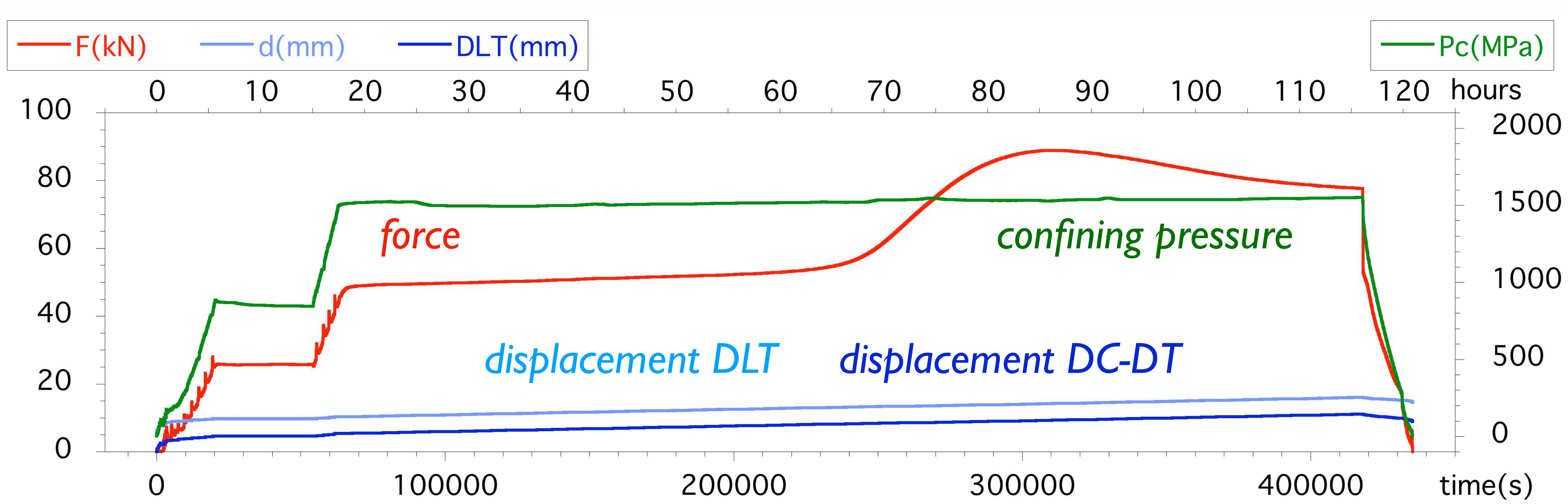
Fortran sources are available from https://micro.earth.unibas.ch/ or at renee.heilbronner@unibas.ch

prepare raw data from experimental record

run record



raw data = run record converted to SI units



why it is important

Open data is generally agreed to be beneficial for science and scientists.
a) published experimental data can be re-anaylized and compared to new data, in a coherent fashion, i.e. using the same options
b) through the citation, the experimentalist is honoured if his or her data is re-evaluated.

meta data

apparatus	distortion, run-in slope ('friction'), ...
experiment	Pc, T, displacement rate, ...
assembly	confining medium, piston diameter, ...
sample	axial: length, diameter ...
	shear: initial / final thickness, angle of pre-cut, ...

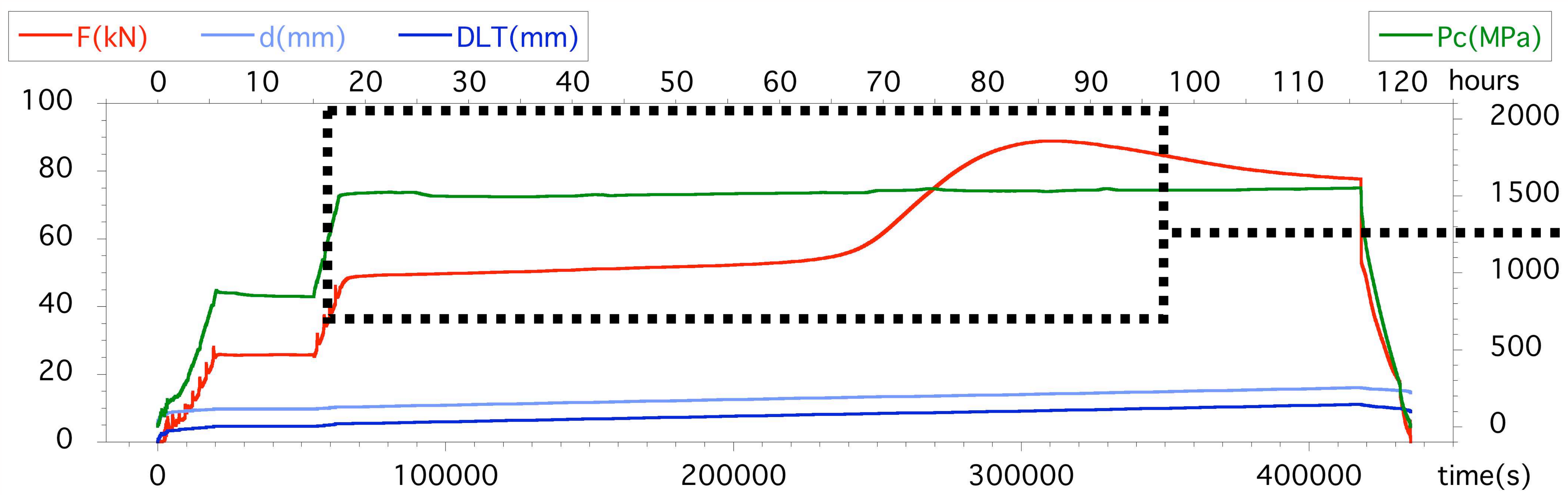
⇩

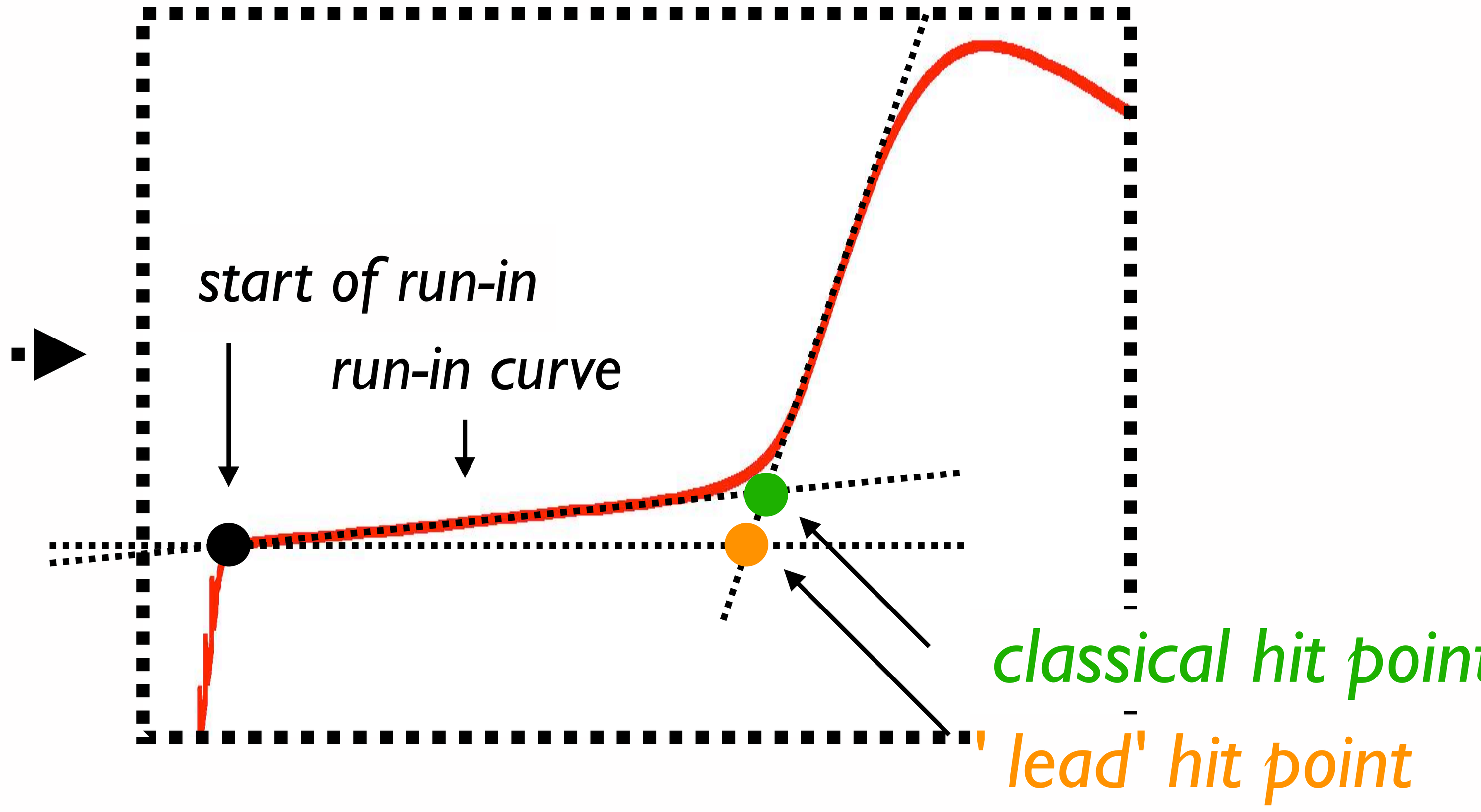
meta data (as used in header of input file for rigS2020)

```
name of experiment                   383BR

apparatus                            Tromsø 2
distortion(mm/N)                     0.80000E-05
friction(N/mm)                       0.13000E+04

nominal Pc(MPa)                      1500
nominal T(°C)                         700
displacement rate(ms-1)              10-8
inner outer sleeve (1=NaCl 2=KI)     11      (all NaCl)

diameter(mm)                         6.33
length(mm)                           0       (NA for shear)
alfa(deg)                            45

th0(mm)                              0.90
thFinal(mm)                          0.58
pre-experimental slip(mm)            0.35
gamma meas                           0       (not measured)
```

crop data for analysis – select hitpoint

raw data file

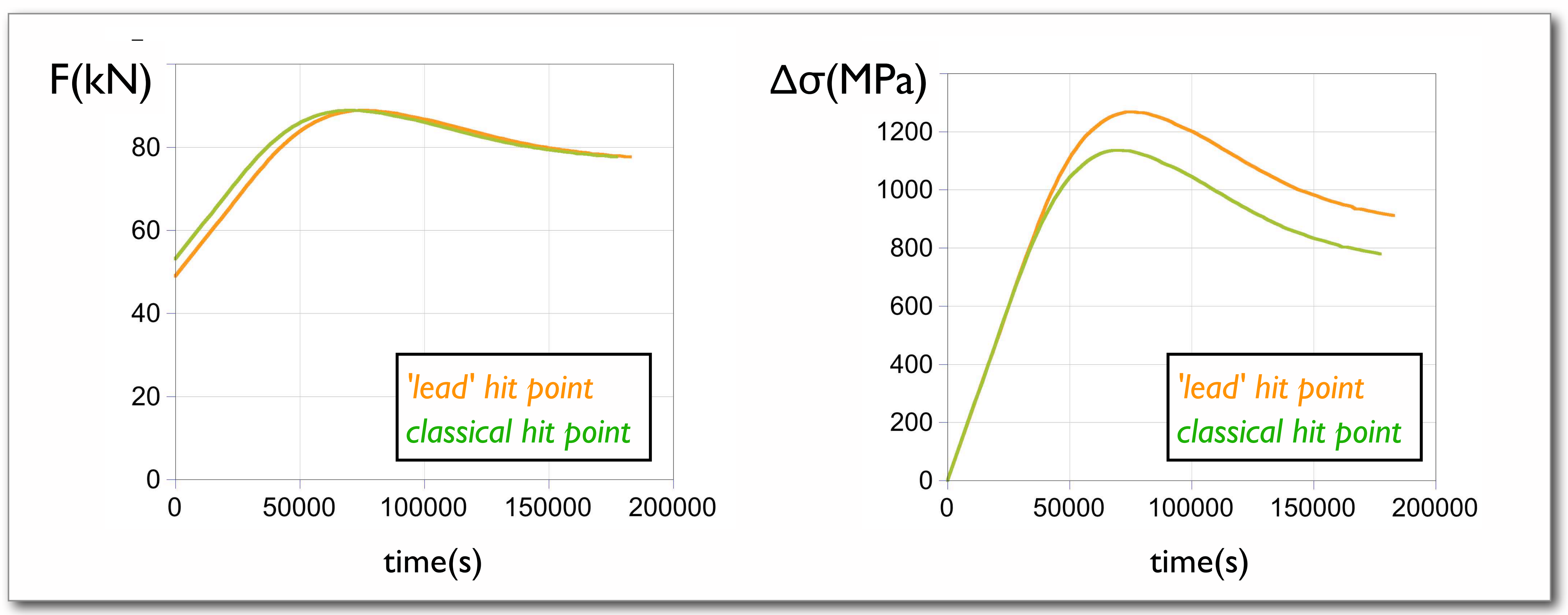




options A

1: using classical hitpoint
2: using (new) lead hitpoint

effect of choice



why it is important

Selection decides if the load accumulated between the start and the end of the run-in curve is 'felt' by the sample as differential stress, i.e., if the load at hitpoint = load at 'lead hit', or if the load at hitpoint = load extrapolated from the run-in curve.

input file

header

```
Run record = 383BR.txt

...etc.
```

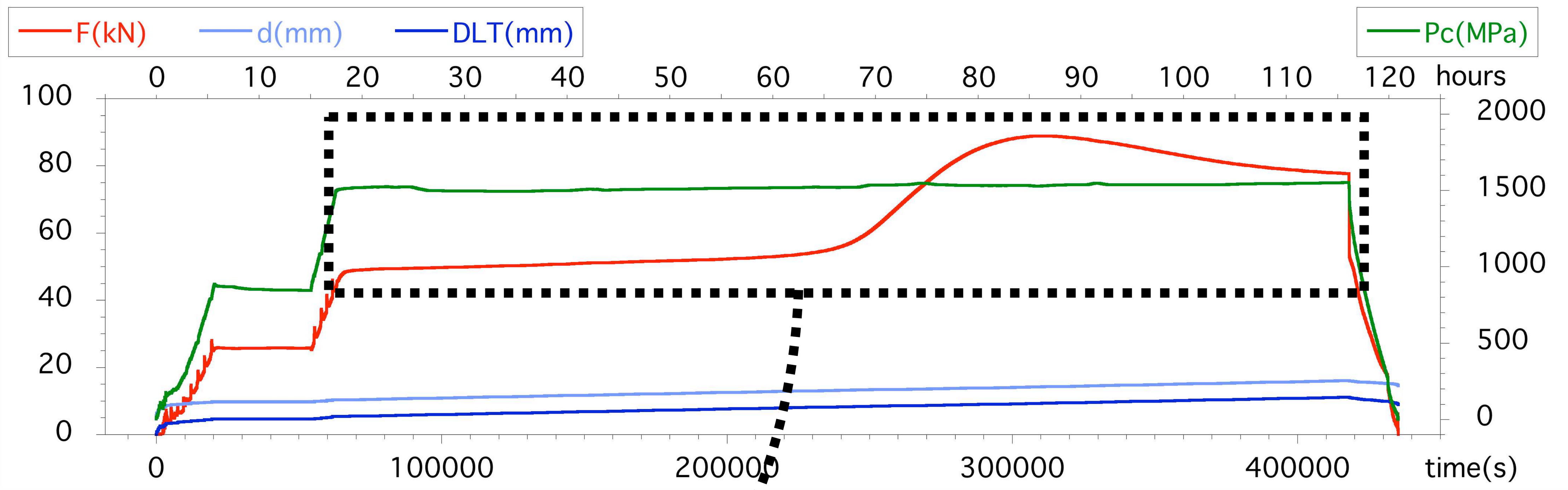
data

	t(s)	F(kN)	Pc(MPa)	DLT(mm)	d(mm)
'lead' hit point	0.0000	48.9604378	1520.09253	-0.00000000	0.000000000
classical hit point	5506.0000	53.1029053	1519.33765	0.085999995	0.085733011
	16695.0000	61.5200462	1533.93213	0.250000000	0.252155900
	18407.0000	62.7319412	1534.43542	0.273099989	0.267285258
	20119.0000	63.9878998	1534.18384	0.296499997	0.282414615
	21831.0000	65.2879333	1533.93213	0.319599986	0.322759569
	23543.0000	66.6099930	1533.68054	0.342899978	0.342932045
	25255.0000	67.9320602	1535.19031	0.366099983	0.347975165
	26967.0000	69.2541199	1537.20337	0.389399976	0.393363237
	28679.0000	70.5761871	1538.20984	0.412499994	0.408492565
	30391.0000	71.8541794	1540.72620	0.435999990	0.433708161
	32103.0000	73.0660706	1543.99731	0.459499985	0.448837519
	33815.0000	74.2779694	1545.75879	0.482999980	0.469009995
	35527.0000	75.4898605	1543.49414	0.506500006	0.499268711

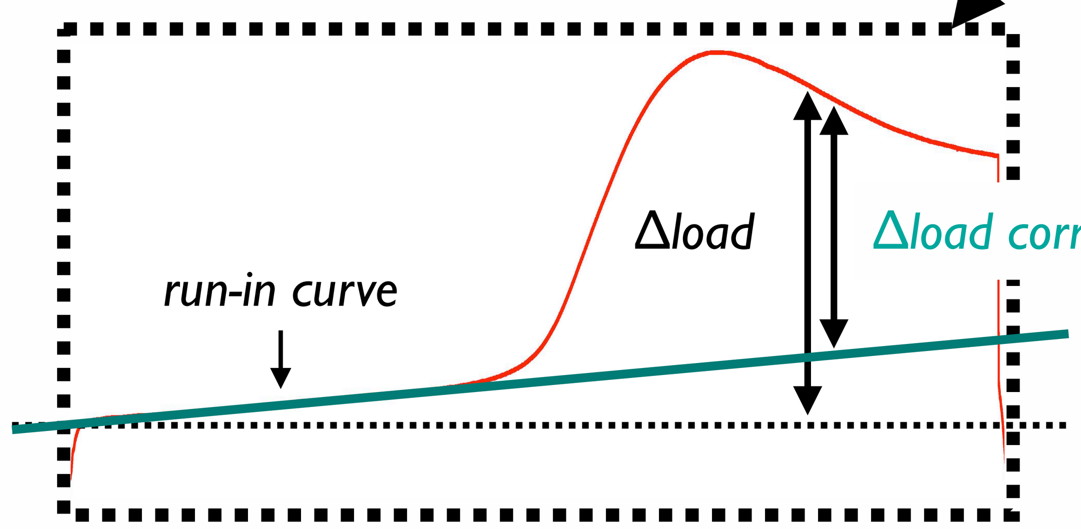
... etc.

perform 'friction' correction ... if you must

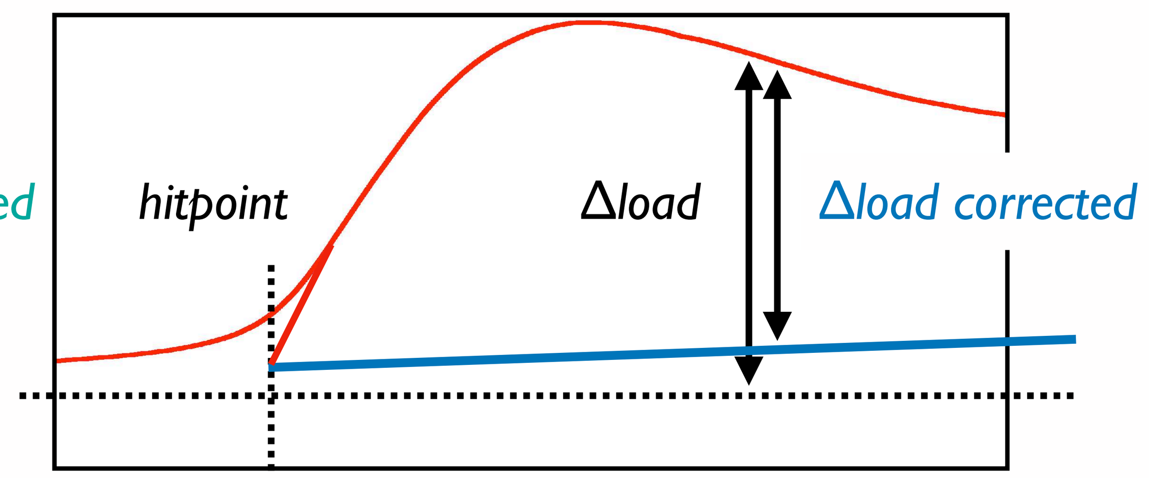
raw data file



differential load



original 'friction correction'
determined from tangent to run-in curve

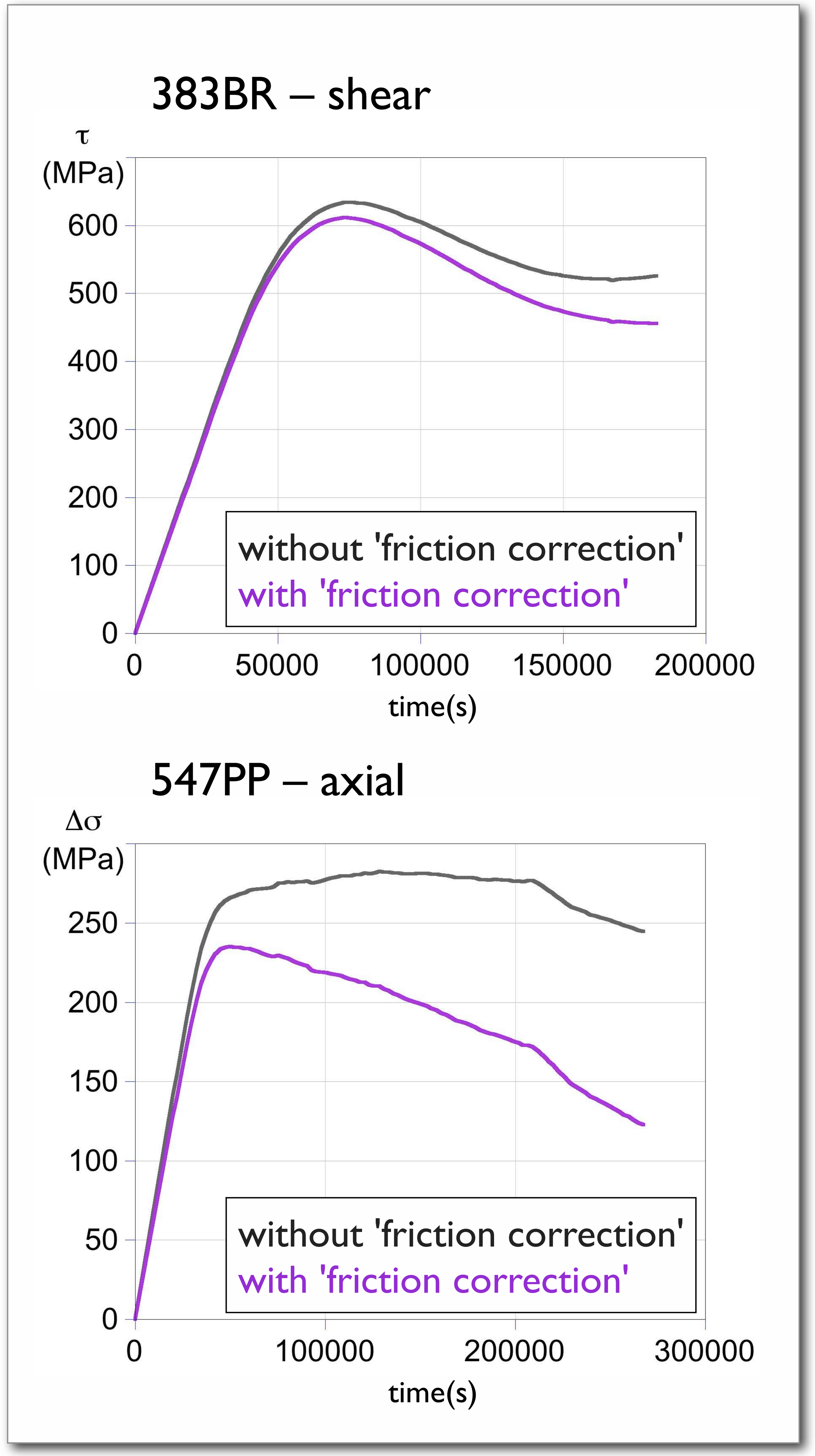


recent versions of the 'friction correction'
determined from the analysis of run-in curves of many different experiments

why is there a 'friction correction'

The 'friction correction' is still used occasionally to curb apparent strengthening of sample at high strains.
However, there is only an empirical basis for this correction – it certainly has nothing to do with friction – and in cases where the differential load is small (weak samples), the resulting load may even become negative...

effect of 'friction correction'



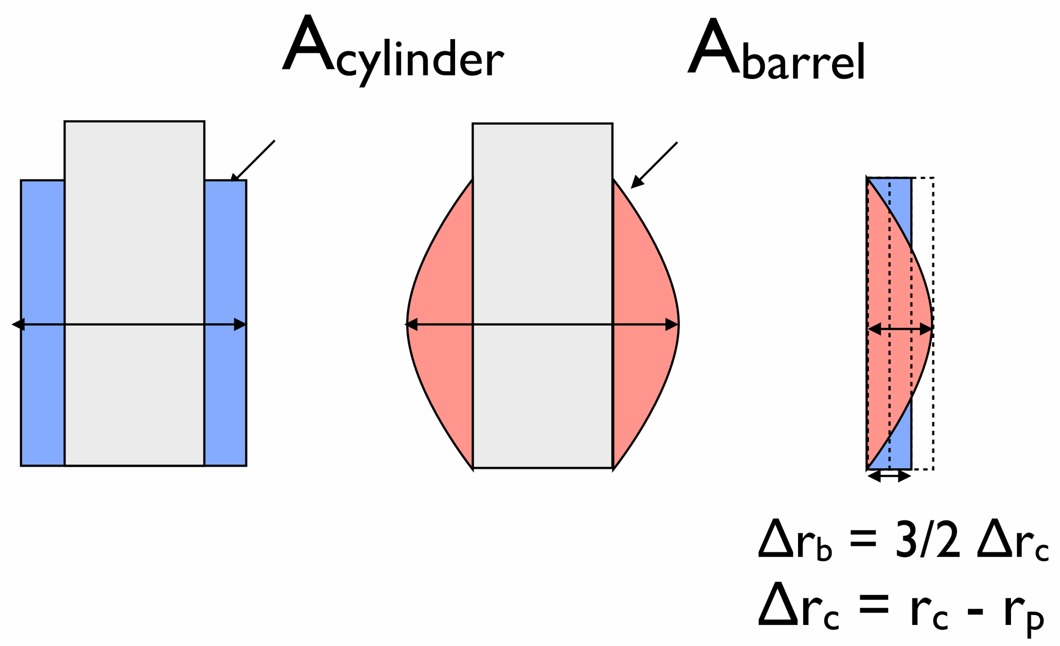
options B

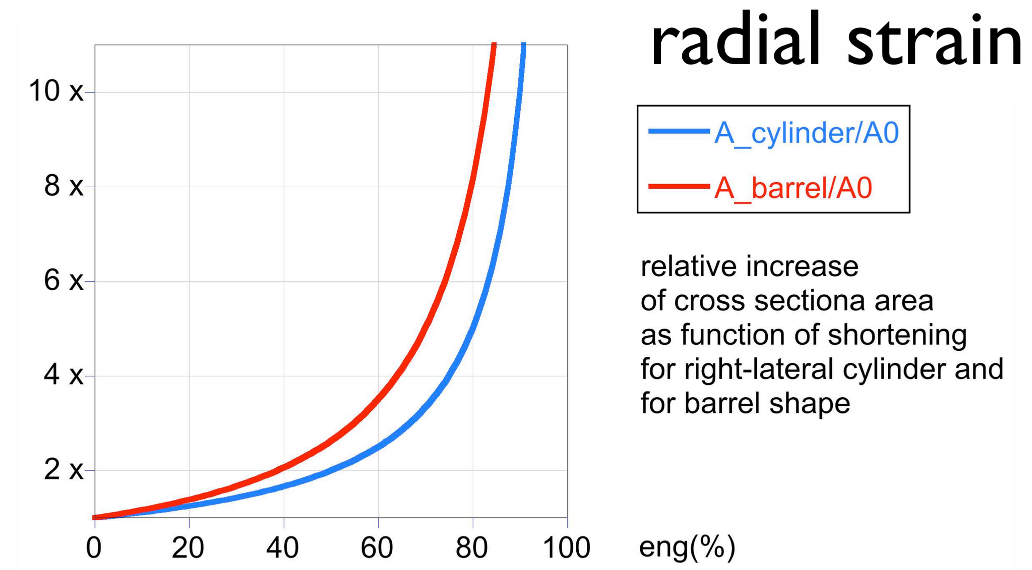
0: no 'friction? correction is applied
1: using 'friction? correction

Compression experiments are started by moving the σ_1-piston in order to bring it into contact with the sample (hitpoint). During the run-in, the piston moves through lead and the load increases as a function of displacement. One can think of the slope of the run-curve as the base line with respect to which the differential load has to be calculated. Because the slope was originally attributed to friction between the σ_1- and the σ_3-piston, this correction was called 'friction correction'.

'area corrections' – for axial and shear experiments

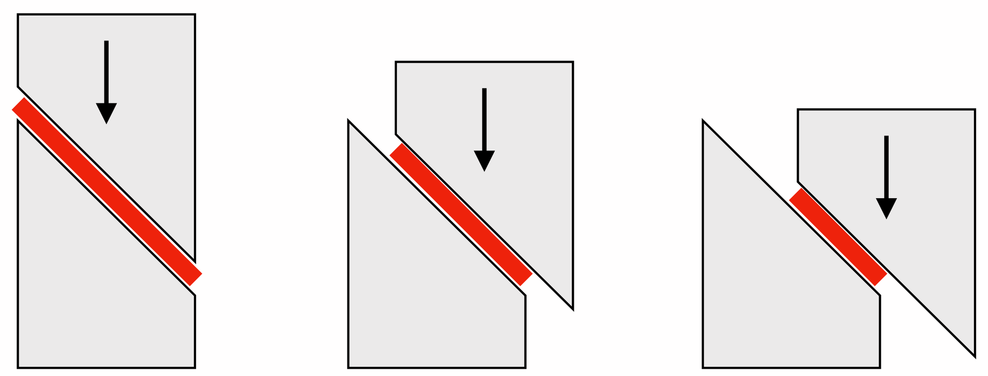
axial

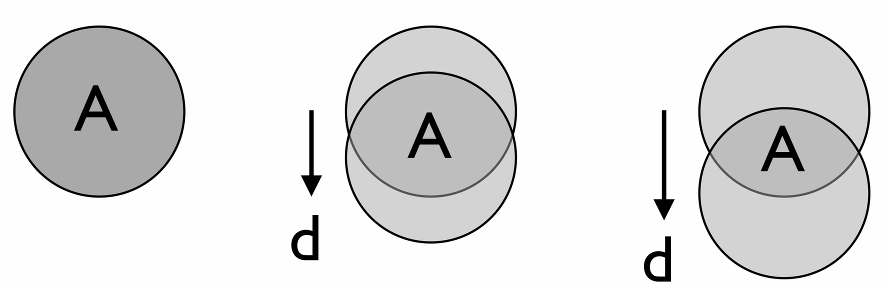


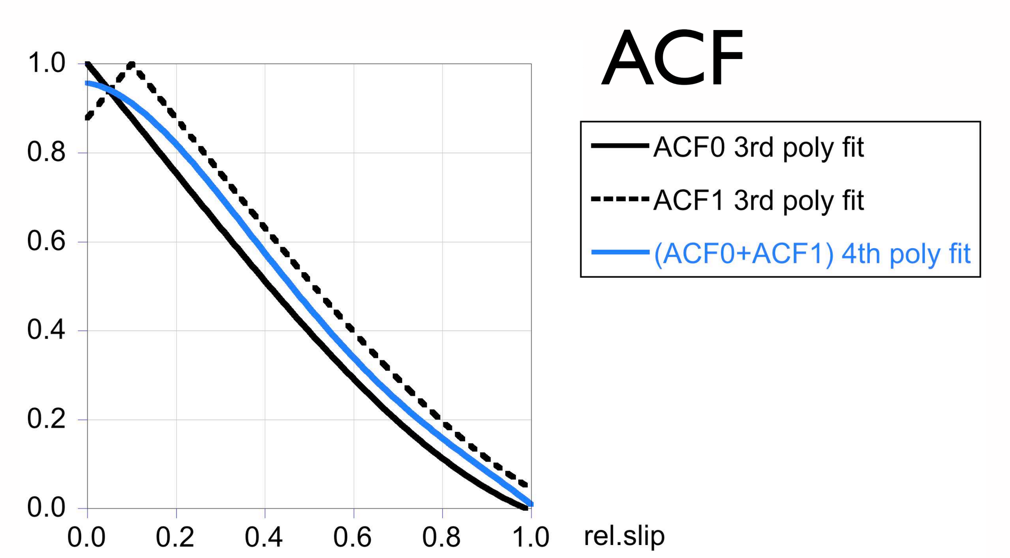


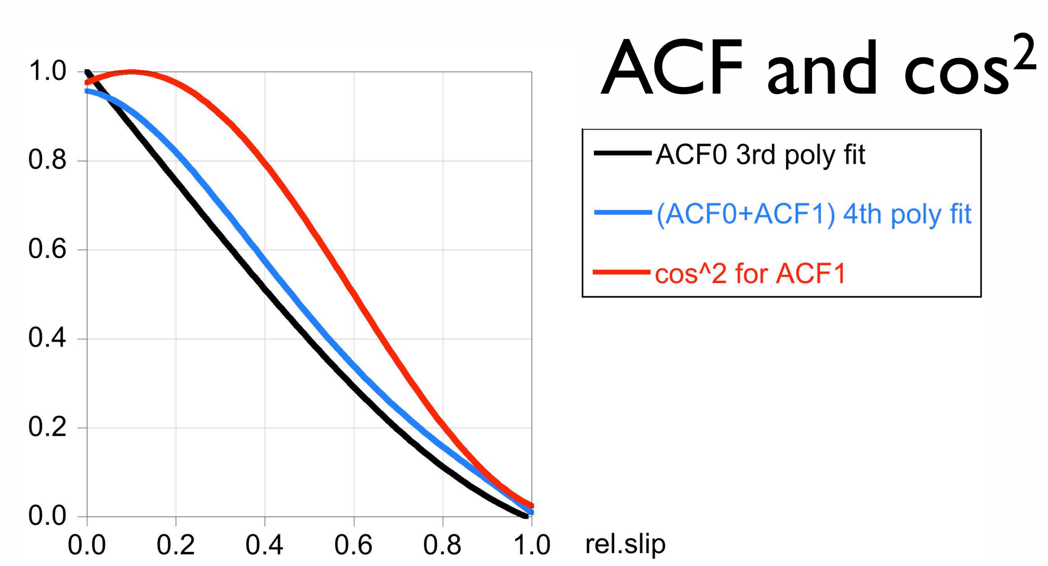
shear

$A = A_{overlap}$





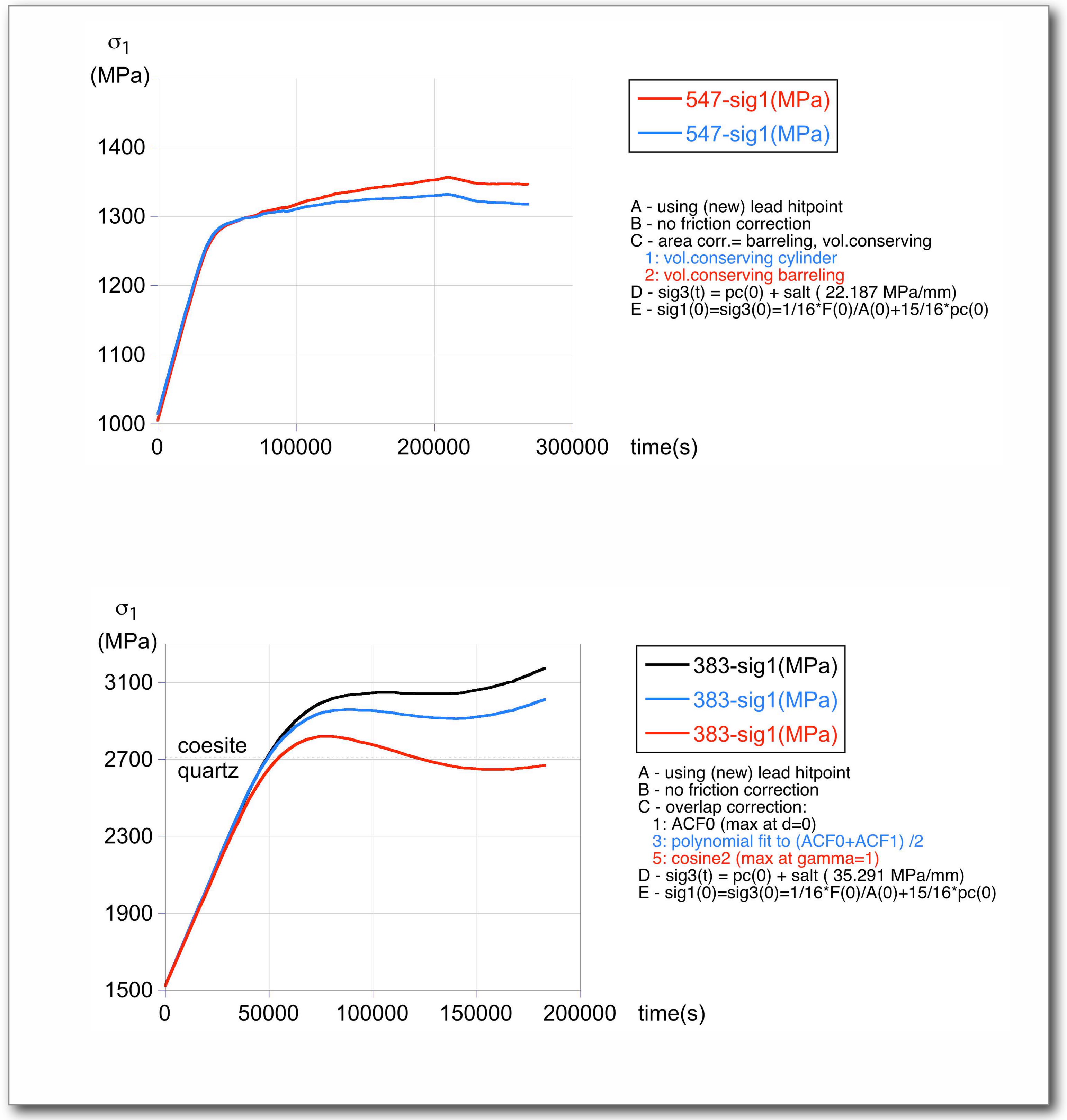


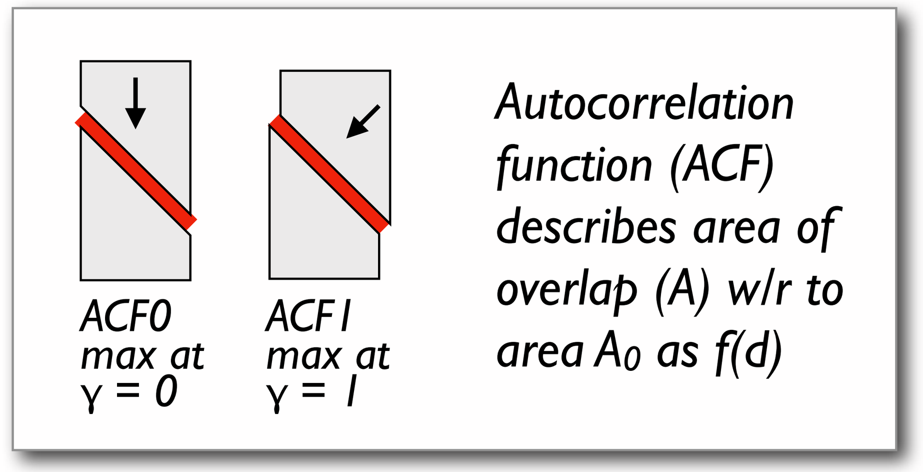


why is it important

Both area corrections have a strong influence on the stresses, not only w/r to their absolute values, but also w/r to the general behaviour, i.e., wether a sample displays weakening, strengthening or steady state flow.

effect of area corrections





options C– axial

0: no area correction
1: homogeneous shortening of sample
2: barreling of sample

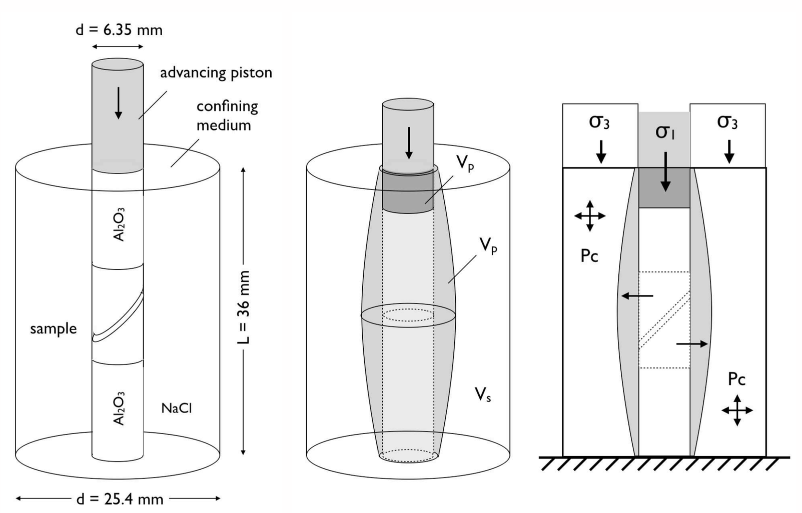
options C – shear

0: no area correction
1: ACF0 (max at d=0)
2: ACF1 (max at gamma=1)
3: polynomial fit to (ACF0+ACF1) /2
4: cosine2 (max at d=0)
5: cosine2 (max at gamma=1)
6: cosine (max at d=0)

The area correction targets two different 'areas'. In axial experiments it is the cross sectional area of the sample which grows as the sample is shortened. In the case of shear experiments, the area to be corrected is the area of overlap of the forcing blocks which decreases as the forcing blocks are offset with increasing shear.
Stresses in shear samples are notoriously difficult to assess. Sample 383BR (Richter et al., JGR, 2016 – which underwent both a qtz-to-coe and a coe-to-qtz transition) is used to evaluate the different options for the overlap correction.

σ_3 during the experiment – the salt correction

confining pressure



ΔMPa/mm for NaCl, and KI

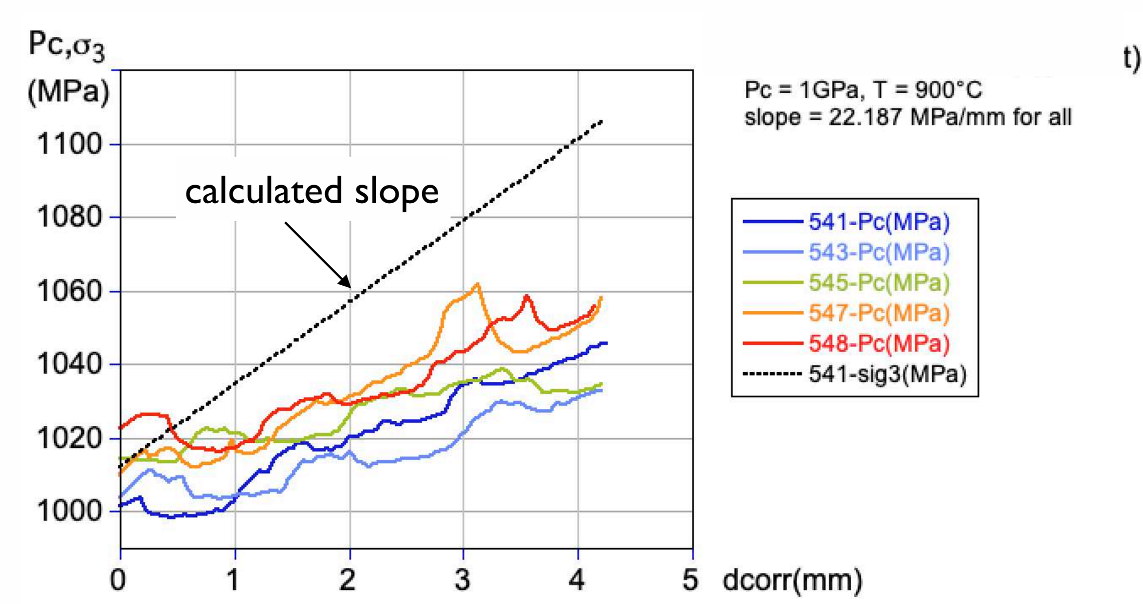
slopes ΔMPa / mm for NaCl							
T (°C)	0 kb	3 kb	5 kb	10 kb	15 kb	16 kb	20 kb
100	43.96	44.27	44.43	44.69			
200	42.13	42.75	43.08	43.69	44.1	44.158	44.36
300	39.73	40.63	41.13	42.11	42.8	42.912	43.29
400	37.12	38.21	38.85	40.14	41.1	41.255	41.8
500	34.65	35.81	36.51	38	39.16	39.357	40.05
600	32.62	33.69	34.37	35.9	37.17	37.39	38.19
700	31.12	32.02	32.61	34.03	35.29	35.519	36.35
800	30.06	30.81	31.3	32.52	33.66	33.879	34.69
900			30.34	31.38	32.36	32.549	33.27
1000				30.51	31.37	31.531	32.16
1100						30.737	31.3

slopes ΔMPa / mm for KI						
T (°C)	0 kb	3 kb	5 kb	10 kb	15 kb	16kb
100	21.368	21.685	21.82	22.005	22.07	22.074
200	20.125	20.678	20.93	21.305	21.448	21.457
300	18.661	19.397	19.753	20.306	20.529	20.543
400	17.238	18.05	18.466	19.148	19.434	19.452
500	16.066	16.827	17.247	17.974	18.295	18.316
600	15.237	15.861	16.23	16.915	17.234	17.255
700		15.179	15.479	16.059	16.344	16.363
800			14.941	15.428	15.666	15.682
900					15.168	15.182
1000						
1100						

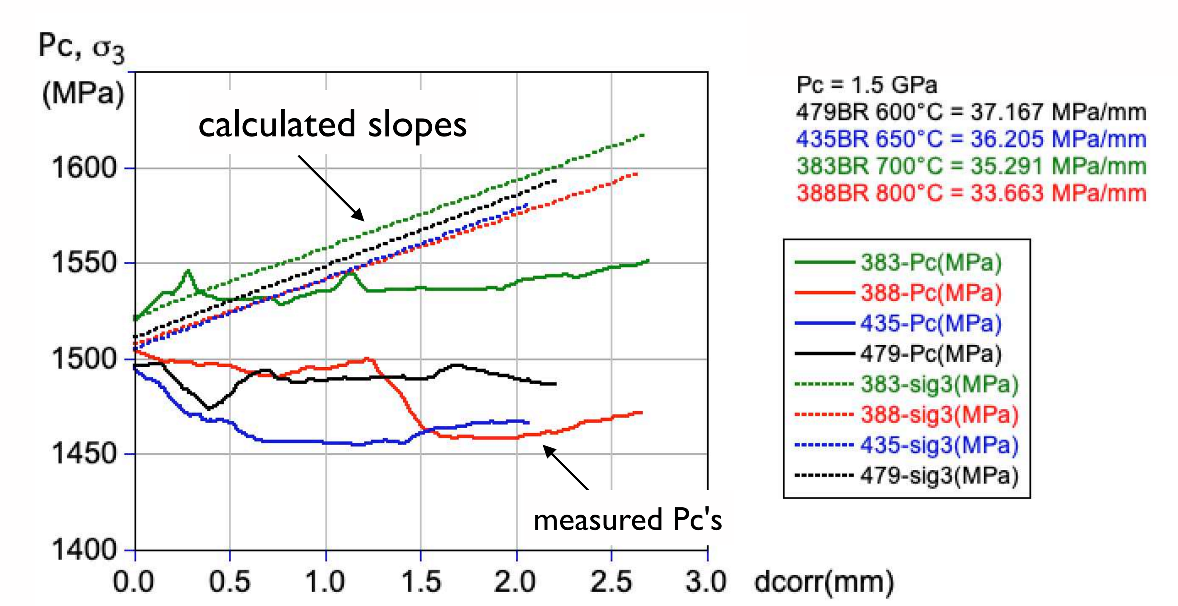
slopes ΔMPa / mm for KI & NaCl						
T (°C)	0 kb	3 kb	5 kb	10 kb	15 kb	16kb
100	31.981	32.338	32.497	32.732		
200	30.367	31.019	31.332	31.839	32.09	32.118
300	28.377	29.275	29.73	30.505	30.916	30.964
400	26.349	27.377	27.926	28.909	29.461	29.528
500	24.578	25.584	26.157	27.243	27.889	27.971
600	23.229	24.094	24.619	25.685	26.363	26.453
700		22.984	23.425	24.368	25.016	25.105
800			22.553	23.352	23.925	24.008
900					23.087	23.161
1000						
1100						

measured and calculated confining pressure

axial



shear

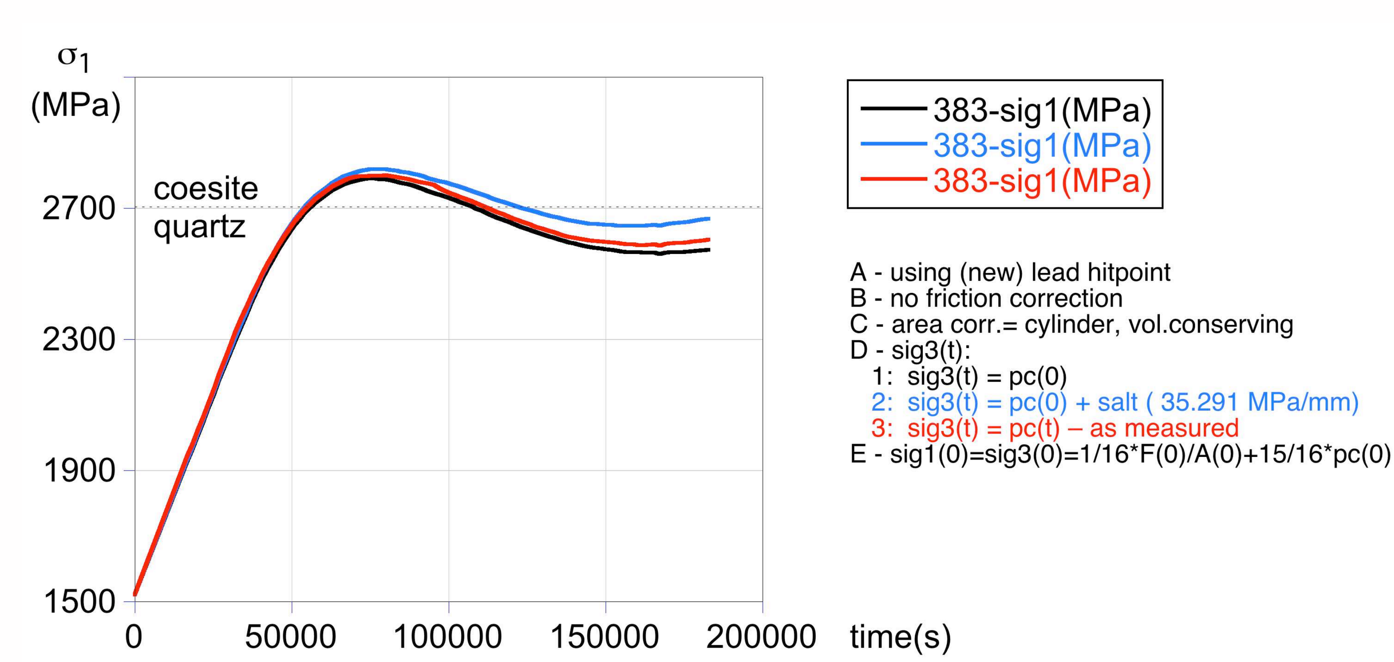


options D

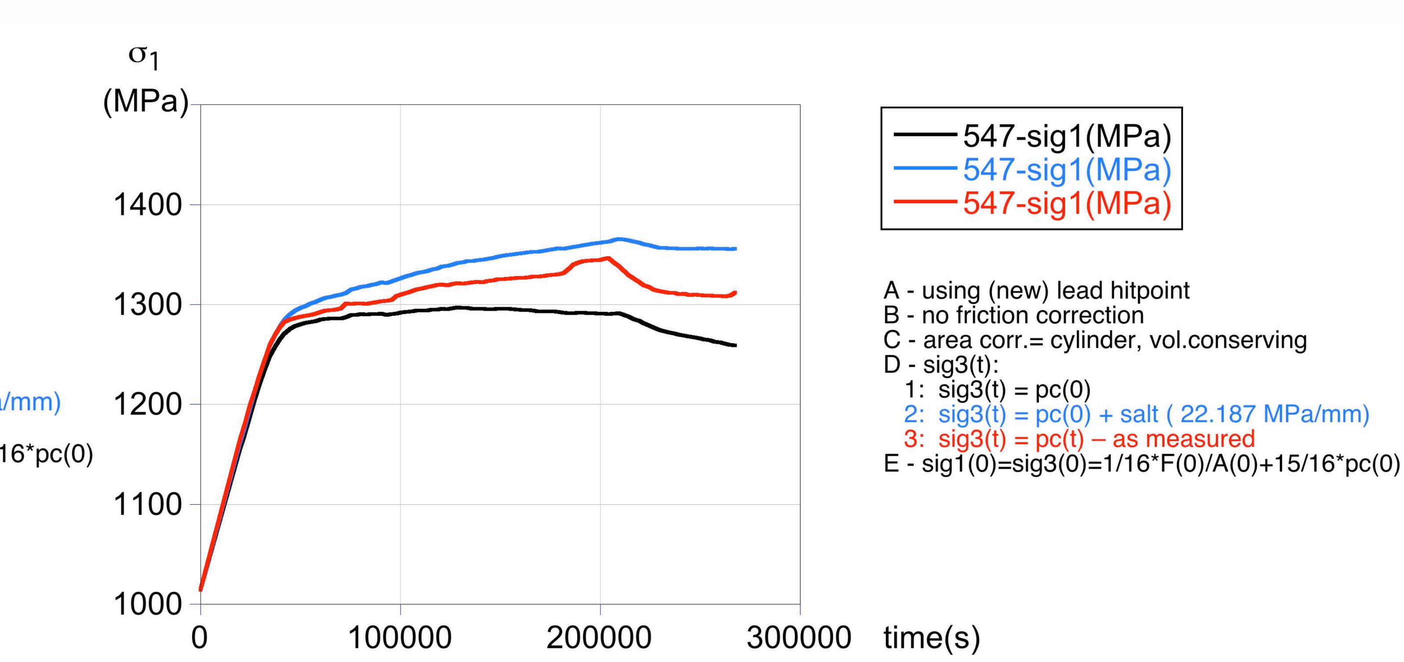
1: sig3(t) = Pc(0) at start
2: sig3(t) = Pc(0) + SALT correction
3: sig3(t) = Pc(t) as measured

effect of salt correction

axial



shear



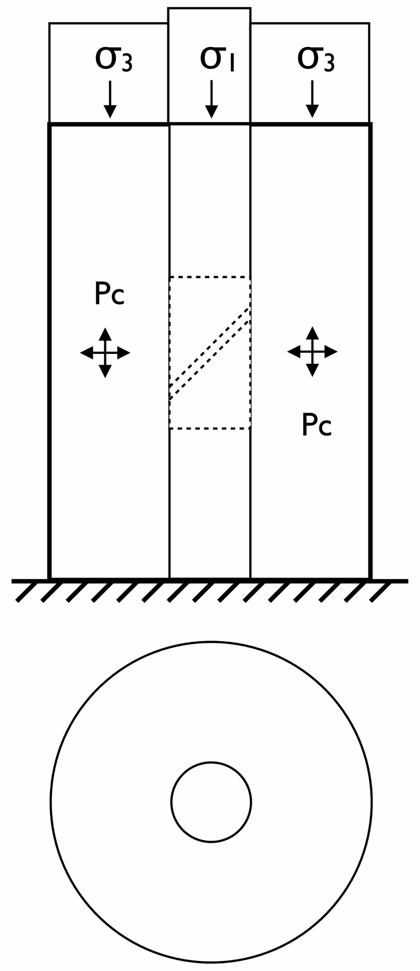
Traditionally the confining pressure was not measured during the experiment. Therefore $\sigma_3(t)$ was set to the value of the confining pressure at the start.
Today we have the option of monitoring pc(t), however, these measurements are not all too reliable because we cannot measure the confing pressure directly. What is measured is the oil pressure of the hydraulic ram and it is not clear if this pressure is fully transmitted to the confining medium.
In addition it is to be expected that the pressure inside the vessel increases as the loading piston advances, thus introducing additional material into the fixed volume of the pressure vessel. This pressure increase only stops once the σ_3-piston starts to retreat.

why it is important

$\sigma_1(t)$ cannot be calculated directly, but is found as the sum of the confining pressure and the differential stress: $\sigma_1(t) = \sigma_3(t) + \Delta\sigma(t)$.

σ_1 and σ_3 at the start of the experiment

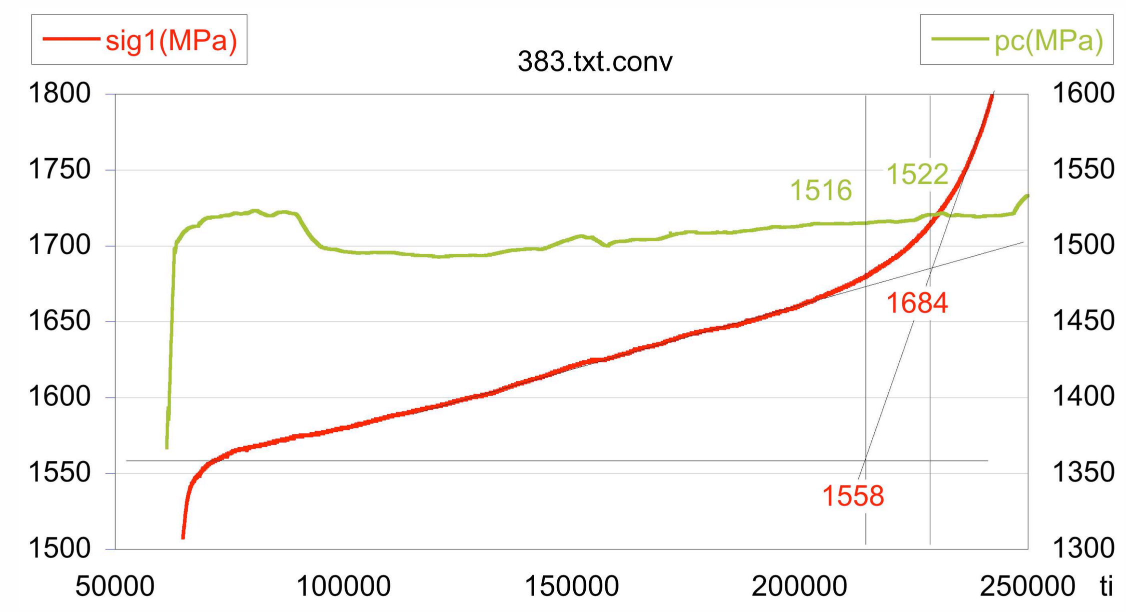
confining pressure



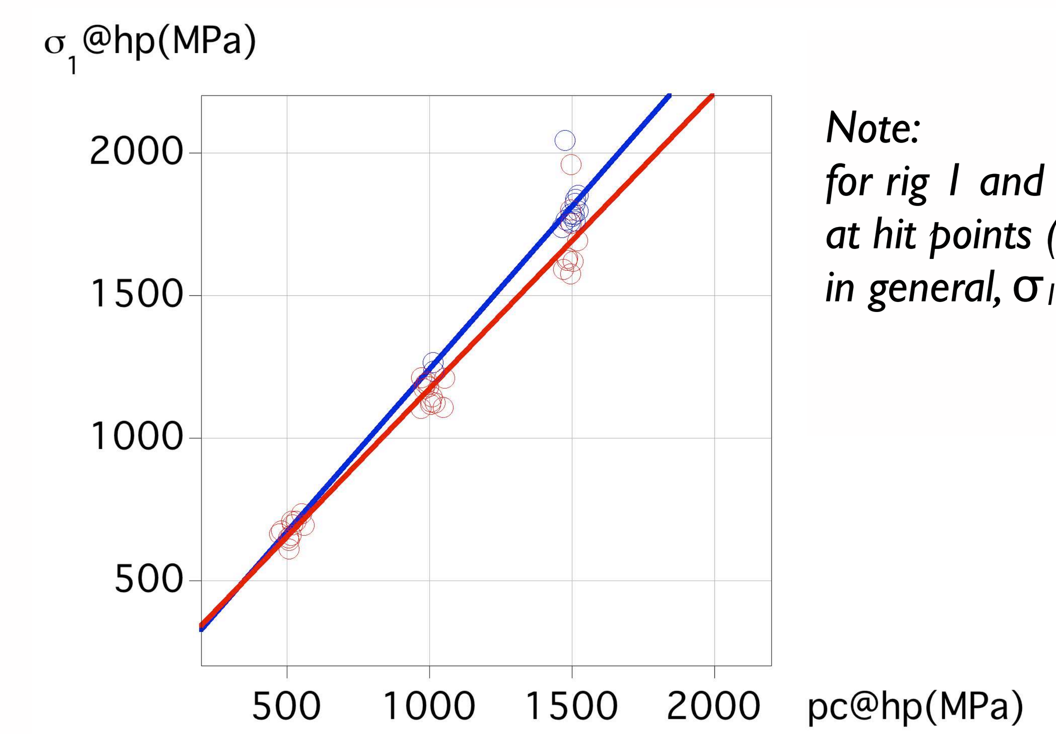
diameter $\sigma_1 : \sigma_3$ = 1 : 4
area $\sigma_1 : \sigma_3$ = 1 : 15

At the start of the experiment, the confing pressure is applied through the ring-shaped σ_1- piston and the so-called σ_1- or load piston. In general, σ_1 (= load/(area of σ_1- piston)) is not the same as σ_3 (= oil pressure of hydraulic ram that actives the σ_3- psiton.

measured s1 and s3 at start of experiment

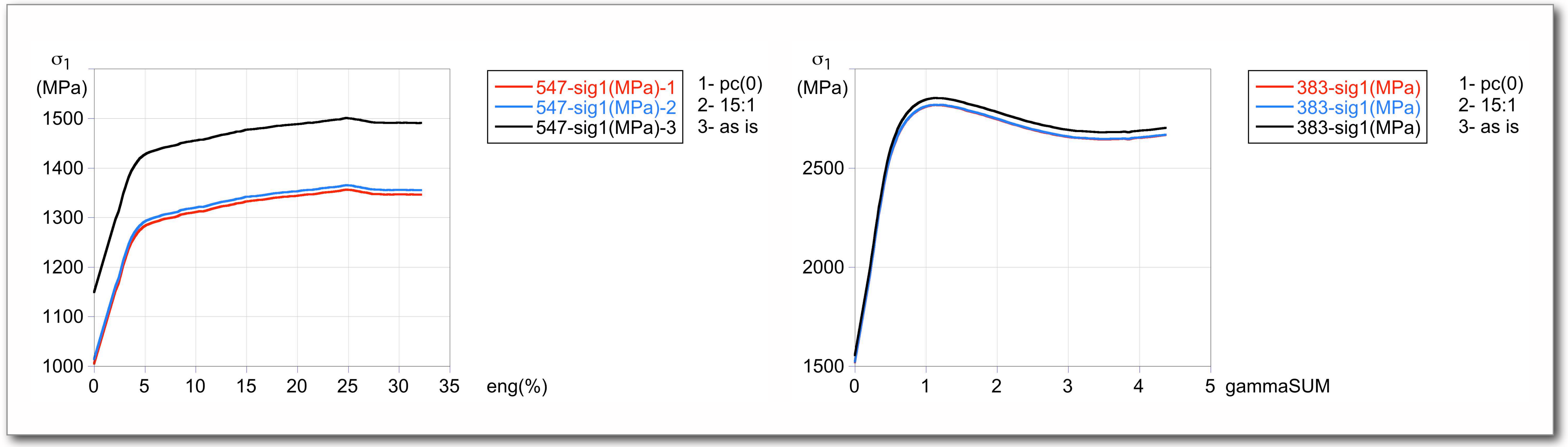


	@classical hitp	@'lead' hitp
σ_3 = Pc(MPa)	1522	1516
σ_1 = F/A(MPa)	1684	1558
difference	162	42
$\sigma_3 : \sigma_1$ = 15:1	1532	1519



Note:
for rig 1 and rig 2 at Tromsø:
at hit points (1 and 2):
in general, σ_1 > Pc

effect of choices



why it is important

In the context of phase transformations, for example, it may be critical to know the absolute stress levels of σ_1 or σ_{mean}. And because absolute stress levels depend on the starting values, $\sigma_1(0)$ and $\sigma_3(0)$ we should determine these values as correctly as possible.

options E

1: sig1(0)=sig3(0)=pc(0)
2: sig1(0)=sig3(0)= 1/16*F(0)/A(0)+15/16*pc(0)
3: sig1(0)=F(0)/A(0) and sig3(0)=pc(0)

The measured confining pressure pc(0) at the start of the experiment and the value of $\sigma_1(0)$ seldom coincide.
$\sigma_1(0)$ = load at start divided by cross sectional area of the loading piston = F(0)/A0. Generally, $\sigma_1(0)$ is not evaluated, and the assumption is that $\sigma_1(0) = \sigma_3(0)$ = pc(0).
However, since the pressure inside the vessel is affected through the σ_1- and the σ_1-piston, and assuming that any differences between the load on these to pistons evens out, the 'average pressure' inside the vessel can be figured out. It depends on the relative cross sectional areas of the pistons through which the pressure (actually the load) is applied. The diameter of the σ_1-piston = 1/4", the outer diameter of the σ_3-piston = 1", the ratio is 1:15.

summary of options – corrections and calculations

option

(-) stiffness correction subtracts the elastic distortion of the apparatus (Δmm) from d as a function of F:
dc = d - 'distortion'(F) — typical value for 'distortion' ≈ 10 μm/kN

A definition of the hitpoint

B friction correction subtracts some force (ΔF) from the F as a function of d:
Fc = F - 'friction'(d) — typical value for 'friction' ≈ 1000 N/mm

C salt correction adds confining pressure (ΔMPa) to medium inside vessel as a function of d, Pc and T:
Pc = Pc + 'slope' — typical value for 'slope' ≈ 35 MPa/mm for NaCl, 17 MPa for KI at Pc=1GPa, T=600°C, increasing with Pc, decreasing with T

D area correction (not really a correction) calculates the (non-linear) relative change of A as a function of dc:
axial experiments: sample cross section: $A(dc)/A_0 > 1$ — typically: $A(dc)/A_0 = L_0 / (L_0 - L)$
shear experiments: piston overlap: $A(dc)/A_0 < 1$ — typically: $A(dc)/A_0$ = type ACF, cos, $\cos^2$

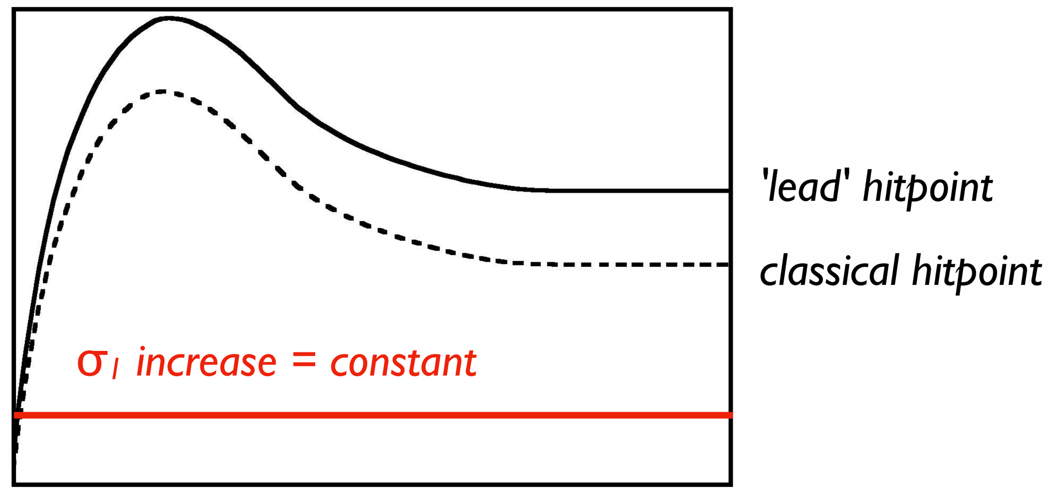
E definition of the starting values for σ_1 and σ_3

d = axial displacement of Δσ piston
dc = displacement of piston inside vessel – 'experienced by sample' (shortening of sample)
F = applied load
Fc = load applied to sample inside vessel – 'felt by sample' (loading of sample)
Pc = confining pressure
T = temperature
A = cross sectional area of sample (axial) of piston overlap (shear)
L = length of sample

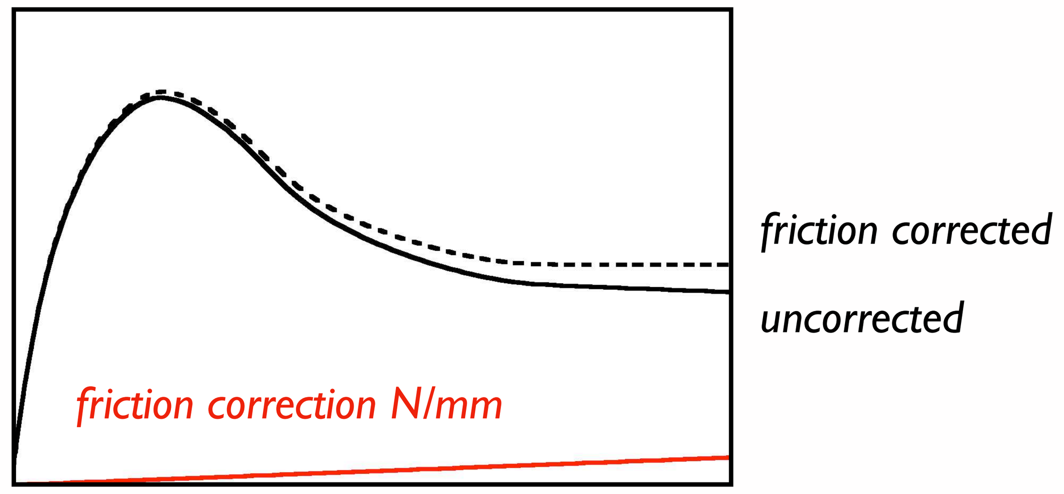
σ_1 piston should be called delta Δσ piston or load piston
σ_3 piston should be called confining pressure piston

effect of options

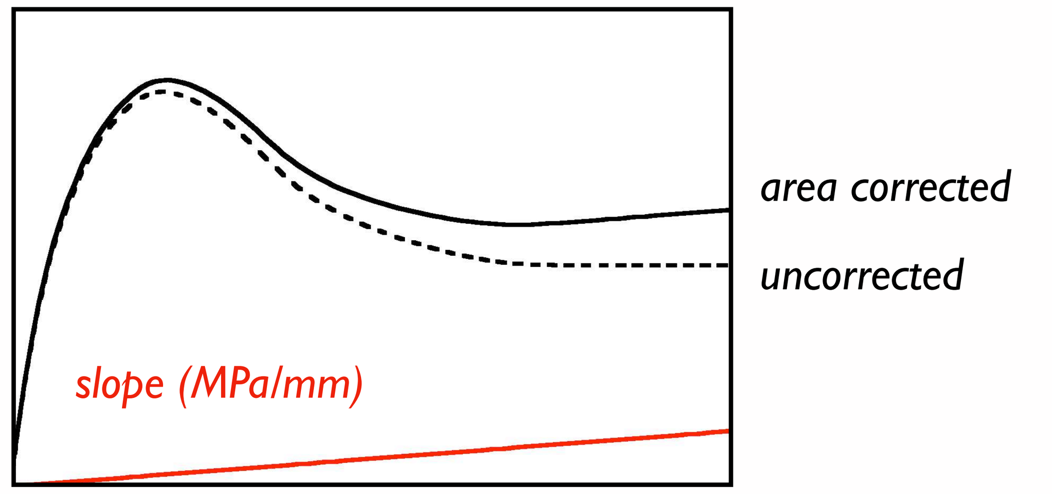
hitpoint
σ_1 + constant



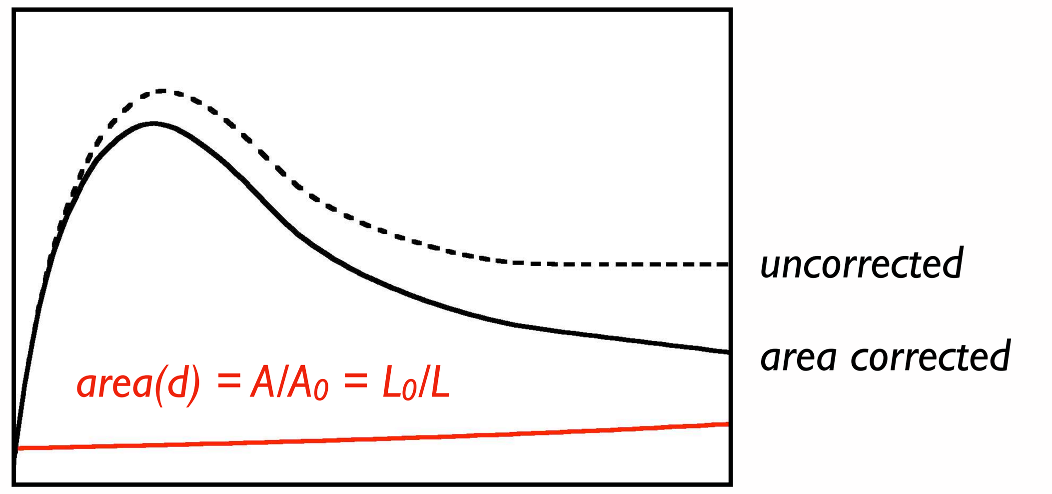
friction
σ_1 - friction(d)



salt
σ_1 + slope(d,Pc,T)



area axial
σ_1 / area(d)



area shear
σ_1 / area(d)