

Dissolution-precipitation creep in polymineralic granitoid shear zones in experiments I: Strain localization mechanisms

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Abstract. Dissolution-precipitation creep (DPC) is considered as one of the main processes accommodating localized strain in polymineralic shear zones of the Earth's crust. Extensive field evidence for DPC in natural shear zones supports the importance of this process. The spatio-temporal evolution and the level of compositional heterogeneity that facilitate the nucleation of such polymineralic shear zones remain poorly understood. A series of new experiments was conducted on a
15 granitoid fine-grained (average starting grain size $\sim 15\ \mu\text{m}$) ultramylonite to different strains at 650°C , 1.2 GPa with strain rates varying from 10^{-3}s^{-1} to 10^{-6}s^{-1} . In Type I experiments, a fracture was induced (prior to reaching the P,T-conditions), whereas in Type II experiments, no initial fracture was induced. Consequently, in the Type I experiments viscous deformation localized strictly within the previous fracture in a $\sim 20\ \mu\text{m}$ wide zone, with grain sizes being reduced to 150-10 nm. In the Type II experiments, viscous deformation was distributed in the sample, with grain size being reduced locally to 200-50 nm. This
20 study supports two different hypotheses for shear zone nucleation in nature. In brittly induced strain localization, DPC will be activated and lead to a rapid and strong strain localization, producing a very weak and fast deforming high strain zone. In viscously induced strain localization (without main fracture), deformation concentrates in zones distributed through the sample, requiring higher shear strains to reach mechanical and microstructural steady state at slower deformation rates compared to brittle-induced strain localization. In both end-member strain localization scenarios, the dominant viscous deformation
25 mechanism in the shear zones is grain boundary sliding combined with pinning-assisted DPC. Our experiments indicate that chemical potentials in the microstructures in combination with different strain localization types may explain the often-observed concentration of strain in fine-grained polymineralic mylonites such as in granitoids but also other polymineralic rocks (e.g. peridotites, granulites etc.) in nature.

Strain localization in the viscous part of the lithosphere is important for regional and global scale deformation, from local shear zones to mountain building scale processes (e.g. Bercovici and Karato, 2002; Ebert et al., 2007; Goncalves et al., 2016; Nibourel et al., 2021; White et al., 1980). Strain localization is critical for the initialization of plate boundaries (e.g. Bercovici and Ricard, 2014). The processes underlying the localization are still debated and many models exist to explain them (e.g. Bercovici and Karato, 2002; Bercovici and Skemer, 2017; Gueydan et al., 2003; Platt and Behr, 2011; Rutter and Brodie, 1988; Schmid, 1982; Tullis and Yund, 1985; Wehrens et al., 2017). One basis for their understanding is the rheology of the rocks hosting the shear zones. The Earth's lithosphere consists mainly of polymineralic rocks (e.g. granitoid, peridotite, gabbro). While the rheology of monomineralic rocks is reasonably well understood, the rheology becomes increasingly difficult to understand with more mineral phases in the system.

For viscous deformation two main end-member deformation mechanisms can be distinguished: diffusion- and dislocation creep. In this study, we extend the term diffusion creep to include dissolution precipitation creep (DPC), where nucleation ("neo-crystallization") and growth of new grains may occur. This appears to be the most commonly observed deformation mechanism in polymineralic shear zones (e.g. Fitz Gerald and Stünitz, 1993; Herwegh and Jenni, 2001; Hippertt, 1998; Imon et al., 2002; Kilian et al., 2011; Mansard et al., 2018; Okamoto and Hiraga, 2022; Stünitz and Fitz Gerald, 1993).

While dislocation creep is a grain size insensitive process, a fine-grained material favors deformation through grain size sensitive diffusion creep due to the increase of grain and interphase surface areas. As diffusion creep implies lower stresses or faster strain rates of the deformation in comparison to dislocation creep, a switch in deformation mechanism from dislocation to diffusion creep has been considered as an important process (e.g. Rutter and Brodie, 1988; Schmid, 1982), because the effect is a weaker deformational behavior that could explain strain localization in Earth's crust. Depending on the starting grain size, different deformation mechanisms may operate (Ranalli, 1982; Rybacki and Dresen, 2004). In coarse grained (granitic or gabbro/peridotite) rocks, dislocation creep probably is the dominating deformation mechanism. The processes reducing the grain size and thereby potentially causing a transition from dislocation to diffusion creep still are a matter of debate and many models exist to understand this transition. The main models include: (1) initially reduced grain size through natural inhomogeneities or brittle fracturing (Mancktelow and Pennacchioni, 2005; Pennacchioni and Mancktelow, 2007; Wehrens et al., 2016); (2) reactions and formation of new phases e.g. symplectites or phase transformations (Ceccato et al., 2018; Fitz Gerald and Stünitz, 1993; Menegon et al., 2008; Stünitz and Fitz Gerald, 1993; Stünitz and Tullis, 2001); (3) a mixture of different deformation mechanisms: initially dynamic recrystallization switching to diffusion creep by subsequent phase mixing and inhibited grain growth through pinning (Bercovici and Skemer, 2017; Ebert et al., 2008; Herwegh et al., 2011; Herwegh and Jenni, 2001; Linckens et al., 2011, 2015; Olgaard and Evans, 1986; Ricard and Bercovici, 2009; Wehrens et al., 2017).

The phase mixing and pinning processes are key to constrain DPC in all three models and are most efficient in polymineralic rocks – highlighting the importance of polymineralic rocks for localized shear zones in the crust.

We performed experiments on natural fine-grained granitoid rocks in two series of experiments. A naturally banded ultramylonite with and without an initially induced fracture was deformed at 650°C and 1.2GPa confining pressure. Although coarse grained granitic or monomineralic samples of quartz and feldspar at the same temperatures, pressures, and similar strain rates deform in a brittle to semi-brittle manner (e.g. Hirth and Tullis, 1994; Tullis and Yund, 1977), our granitoid ultramylonites deform viscously and are mechanically weak. We use samples with properties very close to what is found in nature: (1) low porosity as compared to gouge samples (e.g. Pec et al., 2016), (2) induced inhomogeneity as fracture in a closed system (Tullis et al., 1990), (3) natural inhomogeneity in terms of mylonitic bands (versus e.g. aplite or granite studies by Tullis (2002), Tullis et al. (1990), Tullis and Yund (1977)) and (4) relatively low experimental temperature (compilation of experiments in Ghosh et al. (2022)). This allows to study the role of different phases and phase mixing on the onset of viscous deformation in polymineralic rocks, estimate the strength of polymineralic shear zones at amphibolite facies metamorphic conditions and infer different strain localization mechanisms in nature.

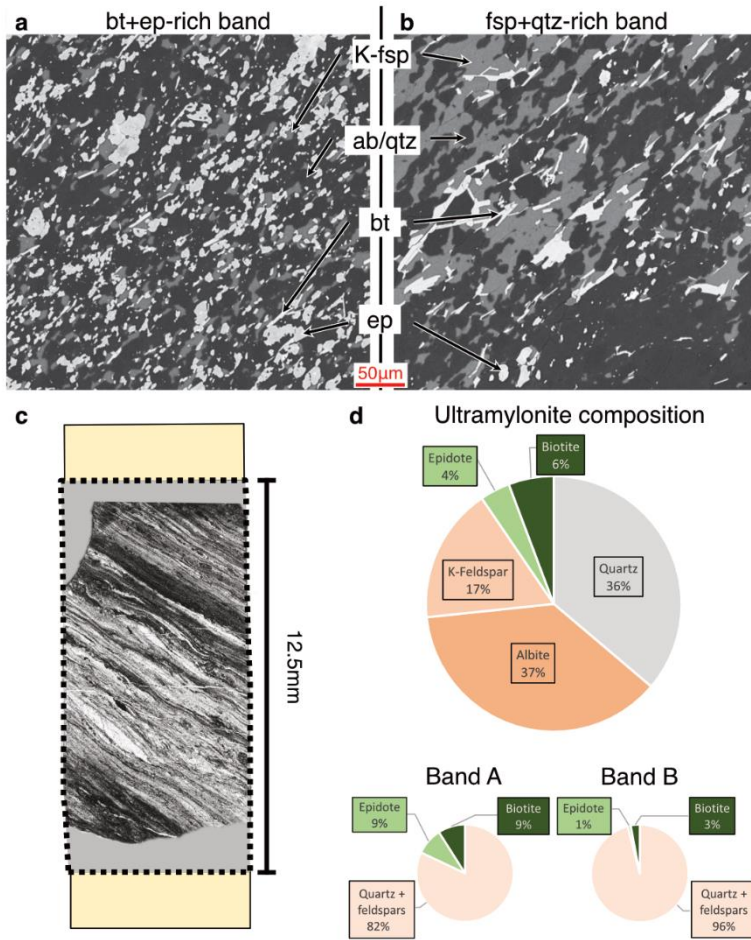


Figure 1: Starting material characterization. Ultramylonite from the Central Aar granite from Grimsel Test site underground rock laboratory. (a, b) Two endmember mylonitic bands. (a) is an ep/bt-rich band, very fine grained ($<15\ \mu\text{m}$), displaying well-mixed phases. (b) is a feldspar(+qtz)-rich coarser band, where less phase mixing occurs. (c) shows foliation and mylonitic bands in experiments. Thin section PPL image of example from 634NN, loaded to experimental conditions and unloaded without imposing any additional strain to the mylonite. Zone (a) appears dark, (b) appears bright in PPL. (d) Bulk composition of the ultramylonite, measured by XRD from milled sample. py+/-wm+/-zircon appear as trace minerals. Compositions of the two band types from SEM images. More details see text and Wehrens et al. 2016 for detailed microstructural investigation of the starting material.

2 Materials and methods

2.1 Starting material characterization

The starting material is a fine-grained granitoid ultramylonite from the Central Aar granite (Fig. 1) that was cored within the Grimsel test site, an underground rock laboratory in the Central Alps. Microstructures of this ultramylonite formed during Alpine peak metamorphic conditions at 450°C and 600MPa (Challandes et al., 2008; Goncalves et al., 2012) and were not overprinted afterwards, preserving the original microstructures. Additionally, the underground laboratory also allows for coring of fresh samples without any traces of weathering. Sample characterization was performed macroscopically, and through optical as well as scanning electron microscopy (SEM) (Fig. 1). The bulk mineral composition (Fig. 1d) was measured by X-ray diffraction (XRD) from a milled representative portion of the sample and further local chemistry from grey value thresholding of SEM backscatter images (Fig. 1d bottom).

The bulk mylonite consists of quartz (qtz) 36 wt%, albite (ab) 37 wt%, K-feldspar (K-fsp) 17 wt%, biotite (bt) 6 wt %, epidote (ep) 4 wt%, and traces of pyrite (py) and minor accessory minerals. It is foliated, and the bands are dominated by different minerals and grain sizes. Their widths range between ~100 µm and ~400 µm. From these mylonitic bands we distinguish two end-member bands based on their grain size and composition. The most fine-grained (down to 2 µm) band is bt/ep-rich and the phases are very well mixed (Fig. 1a). The other end-member band is coarser, with grains >30 µm and contains more fsp and qtz (Fig. 1b). The bt/ep-rich bands appear macroscopically dark green and microscopically dark and pleochroic (Fig. 1c).

Based on these optical characteristics, most fine-grained and homogeneous areas were targeted for the experiments such that the median grain size of the samples is ~15 µm.

For the experiments, the ultramylonite was cored with a 6.33 mm drill bit such that the foliation is inclined 45° to the core axis. Then each potential sample was scanned by X-ray computed micro-tomography (XRCT) in the Bruker SkyScan 1273 with 6 µm pixel size to validate that no drilling-induced fractures exist prior to the deformation. One sample (634NN) was brought to experimental conditions over-night (>8h) to check whether no grain growth or changes of microstructures of the starting material occur before deformation starts. The microstructures in this sample did not differ significantly from the starting ultramylonite (see Supplementary).

The grain sizes of various zones were measured by manual segmentation and calculation of the area equivalent circle diameter

$d = 2 \cdot \sqrt{A/\pi}$. A statistically relevant number of grains or a representative area could not always be measured due to

constraints of resolution and grain size distributions. Therefore, usually a median grain size is given, with an additional range of measured grain sizes. The median is plotted with a number-weighted density distribution and correlates well with the peak of the curve in all samples, serving as a reasonable approximation for the grain sizes in the samples

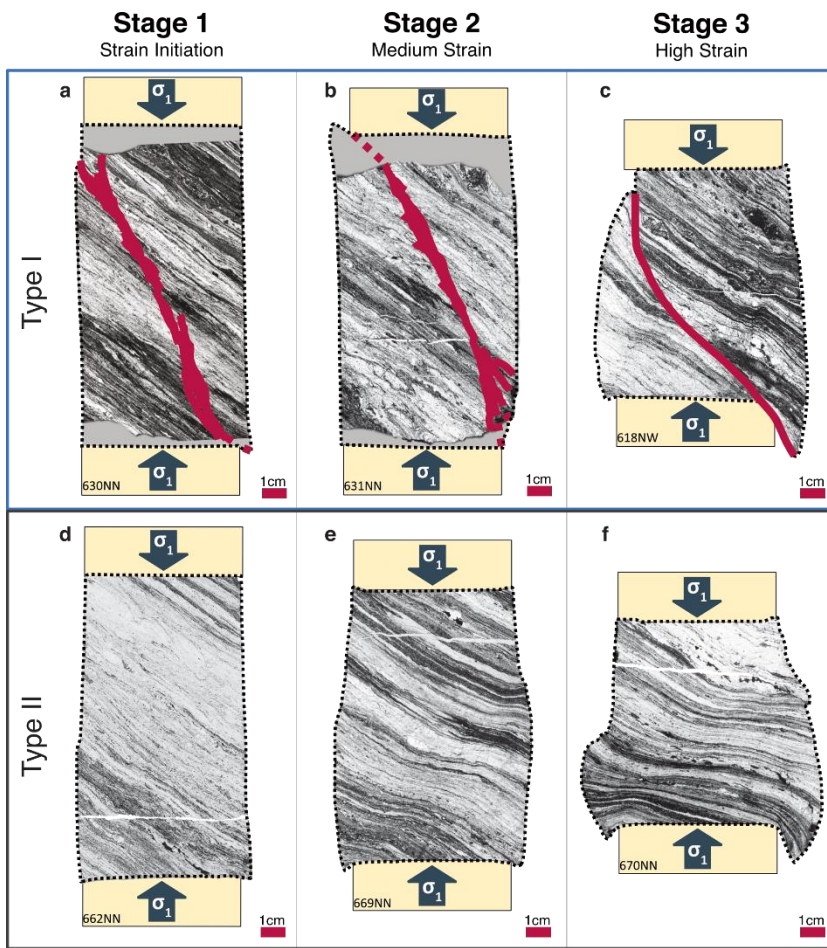


Figure 2: Strain evolution of the two main experiment types as observed from experiments to different strains. PPL microscope images with the final sample geometry drawn manually around the samples. Shear zone orientation and localization in Type I experiments (a-c) in red, note the $\sim 30^\circ$ angle in the initial localization crosscutting the initial foliation, but dipping in the same direction. In Type II experiments (d-f), deformation is distributed, but most widening occurs at the lower 1/3 of the sample. Some corner areas are missing due to damage during polishing (grey). More details in text.

2.2 Experimental setup

We used a Griggs type apparatus at the rock deformation lab at Orléans University, calibrated by Pec (2014). NaCl is used as confining medium for the inner and outer salt pieces. One K-type thermocouple is used to measure the temperature as input for the automated temperature control in the middle of the sample length. The samples were cut to 12 ± 0.5 mm length and polished plane-parallel, then dried at 125°C at least overnight and weighed dry. Welded Pt-capsules were used for all experiments. 0.025 mm thick Ni-foil was wrapped around 2-3 times, depending on the sample thickness inside the capsule. Then, a controlled amount of H_2O (see Table 1) was added at the bottom of the Pt-capsule before inserting the sample, thereby minimizing evaporation of water during closing and welding the capsule.

After reaching the targeted pressure and temperature conditions, the samples equilibrate for at least 1 h. Then the motor advancing the σ_1 piston is started at constant speed and moves through the top lead piece, thereby not deforming the sample significantly. The displacement, confining pressure, and load are recorded digitally in 1 s intervals and simultaneously recorded with an analogue chart recorder. The hit-point, when the piston is touching the sample and actual deformation is starting, is reached after ca. 12 h. The exact position is calculated from the intersection of two slopes before and after the hit-point.

We performed experiments at 650°C , 1.2 GPa and a constant piston displacement rate of 10^{-8} ms^{-1} (Table 1). We have two types of experiments (Fig. 2). For Type I experiments, a brittle fracture is induced during pressurization, prior to reaching the experimental P,T-conditions. For Type II experiments, pressurization of the sample to experimental P,T-conditions is performed more slowly and carefully, to avoid creating loading-induced fractures. Then, a series of samples for both experiment types are deformed to different finite strains (Fig. 2), monitored by the recording of the load (Fig. 3).

The experiments are performed until peak stress, medium strain (or high strain) is reached, to reconstruct the microstructural evolution. These stages refer to the different zones in the stress-strain curve (Fig. 3c): peak stress is the point of maximum stress, after initial sample loading and possible strain initiation; medium strain is where the sample underwent some strain but is not yet in the steady state stress; high strain is the microstructure that forms at steady state stress levels.

Due to the different geometries evolving during the experiment, stresses acting on the deforming zones were corrected differently. In the Type I experiments (blue curves in Fig. 3), all the stress is assumed to concentrate on the shear zone, and the experiments are corrected as general shear experiments (mostly simple shear with a small pure shear component). These samples are deforming three orders of magnitude faster than Type II experiments. The angle and width of the shear zone are considered for the calculation of shear stress and shear strain rate $\dot{\gamma}$. An equivalent (coaxial) strain rate $\dot{\epsilon}_{eq}$ from the shear strain rate is calculated as $\dot{\epsilon}_{eq} = \dot{\gamma} \frac{2\sqrt{3}}{3}$ (Sun and Pec, 2021). The acquired strain of these samples is calculated from the sample shortening as for coaxial shortening experiments, so that their finite strain is lower than that of type II samples despite their fast strain rates and high gamma values (Fig. 3). In Type II (grey curves in Fig. 3), without pre-fracture, stresses are assumed to be distributed throughout the whole cylindrical sample and are corrected as coaxial shortening experiments. A linear friction correction as in Pec, 2014 and Sun and Pec, 2021 was applied on all datasets. More details on the corrections are found in companion paper 2.

2.3 Imaging

Grain sizes ranging from $>20\mu\text{m}$ down to $<10\text{nm}$ required systematic investigation of our experiments though different scales with suitable instruments. Imaging of the samples was performed through the light microscope first, resolving grain scales of mm to μm . Scanning electron microscope (SEM) analyses were performed at a Zeiss EVO50 SEM, using the backscatter (BSE) detector and energy dispersive X-ray spectroscopy (EDX) with the EDAX TEAM™ software, mostly at working distances of 8-9 mm and acceleration voltages of 10-20 kV, allowing to resolve grains on μm scale. Subsequently a high-resolution field emission gun (FEG)- Gemini SEM 450 was used at $\text{WD}=5\text{ mm}$, acceleration voltages of 5-8 kV, probe currents of 70-80 pA to resolve grains $<1\text{ }\mu\text{m}$.

After careful area selection in the FEG-SEM, (scanning) transmission electron microscopy (STEM) was finally used to resolve nm scale grains with the Thermo Fisher/FEI Helios Nanolab G3 (operator at Utrecht University, access through the EXCITE Network), requiring thin foils of selected sample areas. Two methods were used to cut the foils: classical cut with foils vertical to the thin section surface, and pyramidal cut (subsequently thinned) with the foils parallel to the thin section surface. Thereby, three-dimensional information was added to the grain orientation, shapes, and phase mixing. The highly strained and fine-grained material was difficult to prepare and broke out or bent during polishing. To avoid amorphization during ion beam treatment, the polishing was stopped, even though the target thickness of 100 nm on top and $<300\text{ nm}$ in the bottom was not reached. Note that under these circumstances, this led to an overlapping effect of minerals with grain sizes below the thickness of the samples.

3 Results

3.1 Mechanical data

The data shown in Fig. 3 compares the stress-coaxial strain evolution of both experiment types and shows the force data before correcting for sample geometries. Both experiments have a very similar peak stress considering the force on the cylindrical samples. After correction for the different experimental geometries, the localized strain zones in pre-fractured Type I experiments are three orders of magnitudes faster than the coaxial shortening experiments (Type I $\dot{\epsilon}_{eq} = 10^{-3}\text{ s}^{-1}$, Type II $\dot{\epsilon} = 10^{-6}\text{ s}^{-1}$, also see Table 1). The differential stresses at peak stress are $<700\text{ MPa}$ in Type I, $<600\text{ MPa}$ in Type II experiments. In Type I experiments, weakening occurs after peak stress at coaxial strains of $\sim 7\%$ and then an almost steady state stress of $\sim 500\text{ MPa}$ is reached after $>10\%$ coaxial strain. In experiment Type II, continuous weakening is observed with increasing strain to $<200\text{ MPa}$ at 40% shortening. The resulting strains in experiment Type I are up to $\gamma=200$, with slight variations due to varying localized shear zone widths (see Table 1). This is an advantage of this experimental setup, because such large strains are commonly geometrically not feasible in Griggs type experiments but are often necessary to reach a steady-state

170 microstructure. Only torsion experiments in solid medium apparatus could reach large strains to study phase mixing and strain localization, such as in setups of e.g. Cross and Skemer (2017). Despite the complex geometries and sample inhomogeneities, there is a reasonable agreement between the experimental results.

Table 1: Summary of experimental parameters. All experiments were deformed at 650°C and 1.2GPa. The shear strain rates are calculated from final shear zone thicknesses after the experiment, as described in the Methods section.

Exp. No.	Type	Water (H ₂ O)	Coaxial Strain ϵ (%)	Shear strain γ (final)	Final shear zone thickness (mm)	Shear zone angle (°)	Piston displacement rate (m s ⁻¹)	(Equivalent) strain rate (s ⁻¹)
633NN	I	0.2wt%	7.0%	103	0.010	35	$1.01 \cdot 10^{-08}$	$1.55 \cdot 10^{-03}$
631NN	I	0.2wt%	5.0%	75	0.010	35	$1.20 \cdot 10^{-08}$	$1.80 \cdot 10^{-03}$
618NW	I	0.2wt%	27.5%	191	0.020	42	$1.54 \cdot 10^{-08}$	$1.23 \cdot 10^{-03}$
630NN*	I	0.0wt%	0.0%	—	—	—	—	—
634NN	II	0.2wt%	0.0%	—	—	—	—	—
670NN	II	0.2wt%	40.0%	—	—	—	$1.61 \cdot 10^{-08}$	$1.34 \cdot 10^{-06}$
669NN	II	0.2wt%	5.0%	—	—	—	$1.55 \cdot 10^{-08}$	$1.26 \cdot 10^{-06}$
658NN	II	0.2wt%	6.8%	—	—	—	$1.36 \cdot 10^{-08}$	$1.13 \cdot 10^{-06}$
662NN	II	0.2wt%	8.0%	—	—	—	$1.33 \cdot 10^{-08}$	$1.11 \cdot 10^{-06}$

175 Last column: For Type I experiments an equivalent (coaxial) strain rate is given, for Type II experiments a coaxial strain rate. All experiments at 650°C and Pc=1.2GPa.

*Exception: Was only loaded 1.5kbar at 100°C without reaching the final experimental P,T-conditions.

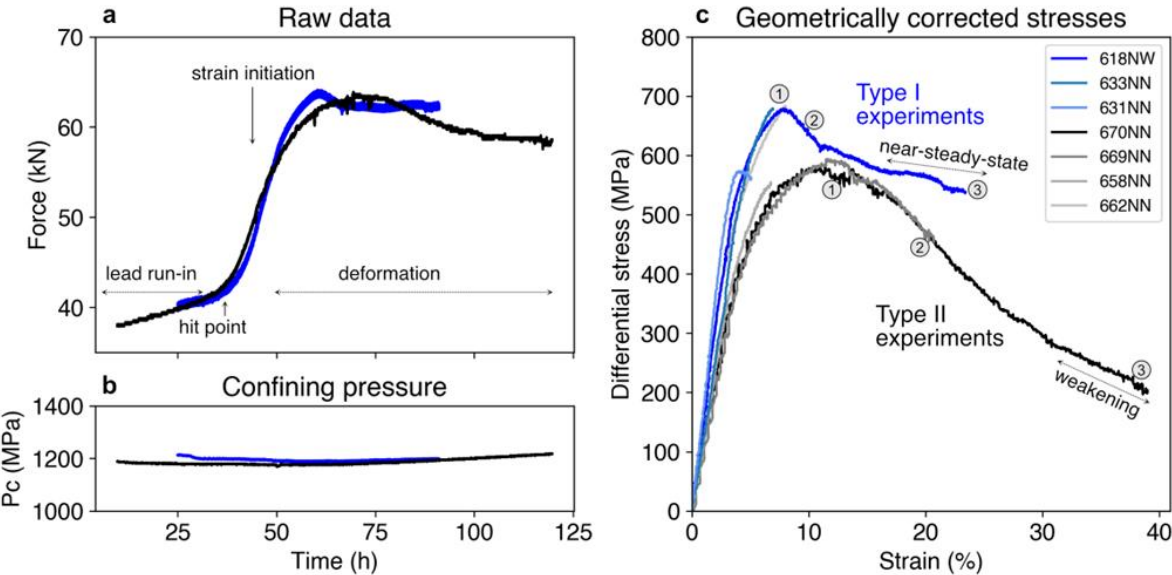


Figure 3: Mechanical data of the presented samples. We show here the raw data of the high strain samples, that represents the force acting onto the experimental samples with the similar initial cylindrical cross-section (a) and the confining pressure (b). The corrected data for different sample geometries is displayed in (c). The onset of deformation in Griggs type experiments initiates during the slope after hit point (a). In (c), the three deformation stages as used in this study are labelled with numbers: 1 is peak stress, 2 is medium strain, 3 is high strain.

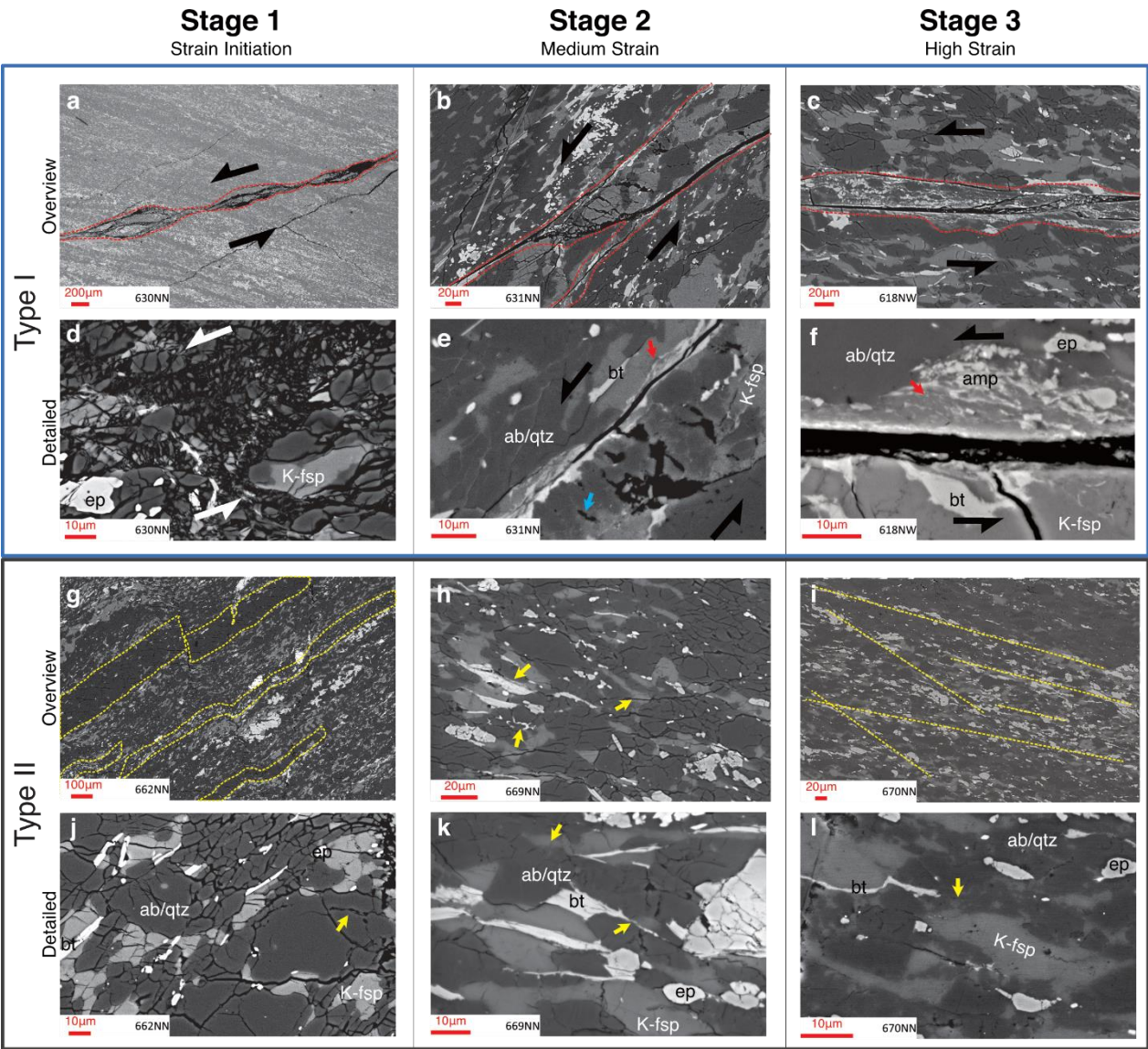


Figure 4: Differences in strain evolution of the two main experiment types as observed from experiments to different finite strains. SEM images at low magnification to highlight the different geometries of the shear zone in Type I (a, b, c) and II (g, h, i) experiments and the details in high magnification SEM images below corresponding overview. Blue arrow in (e) points at open porosity, red arrows in (e) and (f) point at newly neo-crystallized grains. Yellow arrow in (j) shows rounded pores at a grain boundary. Yellow arrows in (h, k, l) point at newly formed grains at phase boundaries. The shear sense in Type I experiment images is indicated by arrows, for the shear zone angle please refer to Fig. 2. Compression direction is vertical in Type II experiment images. Black openings in exp. 662NN (j) occur during unloading and are relaxation cracks indicating stress concentration. Note that strain initiation in Type I experiments (a, d) occurs before the final experimental P,T-conditions are reached, while strain initiation in Type II experiments (g, j) is at peak stress in the stress-strain evolution of the sample.

3.2 Microstructures – Type I experiments

In Type I experiments, the grain sizes are at first reduced in a localized fracture down to $<0.5\ \mu\text{m}$, already before the experimental pressure and temperature (P,T)-conditions are reached. The fracture crosscuts the initial foliation but follows the foliation direction separated by steps. Some portions are located along the foliation and other portions are more vertical connecting to the next inclined step (Fig. 4a,d). The resulting brittle zone is undulating, and its width varies between 10 to 100 μm (Fig. 4a). This zone has a cataclastic microstructure and high porosity (Fig. 4a,d). These basic geometric and mechanical features are reproducible and are observed in all four experiments (Table 1, Fig. 3).

Subsequently, viscous deformation localizes in these fine-grained, originally cataclastic, zones. Once the P,T-conditions are reached and 5-7% shortening are applied (exp. 631NN), the viscous shear zone has a dramatically reduced porosity (Fig. 4b) and its width narrows down to $\sim 20\ \mu\text{m}$. Grains are flattened and stretched, grain boundaries are more irregular, and a larger volume fraction of very fine grains develops (Fig. 4e). The surrounding host rock microstructures are identical to the starting material (e.g. slightly angular, same grain size; Fig. 4b). These microstructures indicate that the onset of viscous deformation was initiated within the fracture rather than in the unaffected host.

With increasing shear strain (exp. 618NW), porosity decreases further and evolving microstructures lead to further grain size reduction in such narrow zones (Fig. 4c,f). A through-going unloading crack (black) forms directly adjacent to the most highly localized deformation zone during decompression of the sample after deformation (Fig. 4f). In the viscous very high strain zone, deformation has localized in a $\sim 2\ \mu\text{m}$ to 200 nm wide zone (Figs. 4c,f and 5a,d).

On SEM scale, we find elongated biotite/ amphibole (bt/amp) grains in incompletely filled voids, indicating nucleation and growth (Fig. 5b). Rounded pores are observed at grain boundaries and triple junctions, often within albite (ab), K-feldspar (K-fsp) and indicate dissolution or intergranular sliding processes (Fig. 5c, see Discussion). Feldspars display chemical changes through reaction (nucleation and growth) and thereby formation of smaller grains with different compositions adjacent to each other. By nucleation the grain size is reduced and the feldspars become more mixed compared to the starting material (Fig. 5c). In the following, secondary minerals that grew during the experiments are indicated with a 2 after the abbreviation, e.g. secondary K-feldspar = K-fsp2 in contrast to primary K-feldspar = K-fsp1.

In many areas, reaction microstructures of primary biotites are observed (e.g. Fig. 5d,e), leading to reduced grain sizes and nucleating new grains (as verified by TEM). Primary epidotes (and biotites) are overgrown with phases having a higher Z-contrast in BSE images. These new phases can be characterized by Fe/Mg Ca-silicate with low Al-content. These are most likely amphiboles, which cannot be identified with certainty due to their small grain sizes compared to the electron beam interaction volume (Fig. 5f), so that mixed analyses are likely. They will be referred to as amphiboles (amp) below. No amphibole was contained in the starting material, and these new minerals must have formed during the experiment.

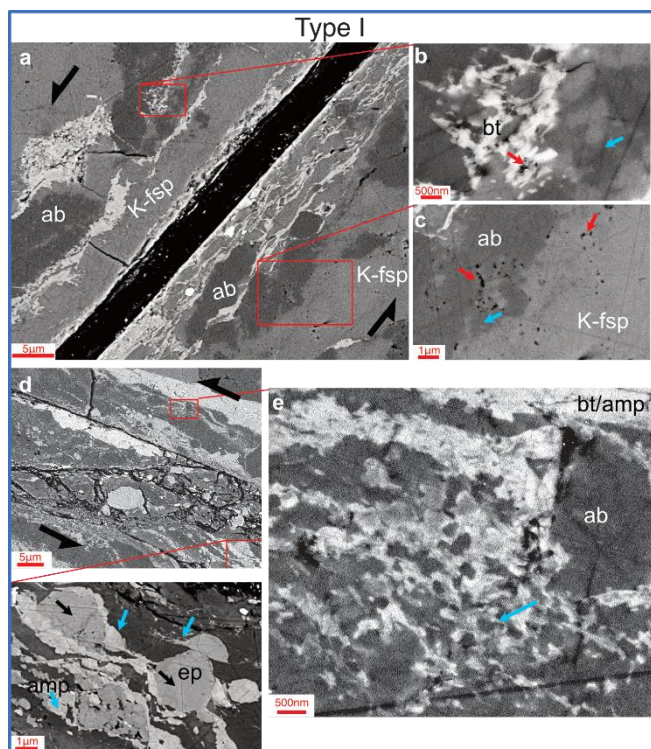


Figure 5: Deformation microstructures in pre-cracked experiment Type I. SEM images from experiment 618nw. Overview (a) shows locations for (b), (c); overview (d) locations of (e) and (f). Note the different BSE contrasts due to different SEM beam settings in areas (a) and (d). (b): Biotite grains with open porosity (red arrow) and patchy ab/K-fsp that cannot be resolved properly in SEM BSE images (blue arrow). (c): Porosity located in between small grains and grain boundaries within/between K-fsp and ab grains (red arrow); small fsp grains (blue arrow) and brighter appearing ab/darker K-fsp between the parent grains. (e): Typical (symplectic) texture of bt and fsp, resulting in fine grains such as pointed at by blue arrow. (f): Ep1 (black arrows) overgrown by ep2/amp (blue arrows). Right blue arrow points at elongated amp forming a tail of previous ep.

215 The locations of TEM foils are marked in Fig. 6a-c, with (vertical) TEM foil in Fig. 6d for overview, and a selection of TEM images in Fig. 6e-l. In TEM sections, extremely fine grains (~50-200 nm) of bt, but also K-fsp, ab, qtz and ep, amp were observed. Most of such grains are dislocation-free. We see evidence for small, defect-poor grains of bt2 overgrowing bt1, while the lattice of bt1 is clearly strained with many dislocations (Fig. 6e). Other bt2 grains have idiomorphic crystal shapes, showing nucleation and growth. They often occur at grain boundaries of other phases (Fig. 6f,h). At the center of the shear zone, a thin nano-crystalline film is found (Fig. 6g). Despite the extremely fine grain size (2-15 nm), the zone is polycrystalline and polycrystalline, as can be seen by the lattice fringes (Fig. 6g) and chemical maps (see Supplementary).

220 In contrast to the relatively defect-free new grains, some old grains still have high defect densities, as for example the >500 nm size K-fsp in Fig. 6g. We assume that these grains are still primary K-fsp that were broken, most likely by the initial brittle crack (Figs. 2, 4a,d). Further, these K-fsp1-relics are also characterized by their chemical inhomogeneity. It shows a depletion

225 in K and enrichment in Na towards its rim.

A few small pores, mostly associated with feldspars, were observed (Fig. 6i), indicating that the fine-grained zones might not be as dense as they appear in SEM. In many areas, the different phases are well mixed, especially in fine-grained zones (Fig. 6j,k), but locally also more monomineralic, fine grained areas exist. In those monomineralic areas, grain sizes are commonly >100 nm, whereas the very well mixed grains show sizes below 50 nm.

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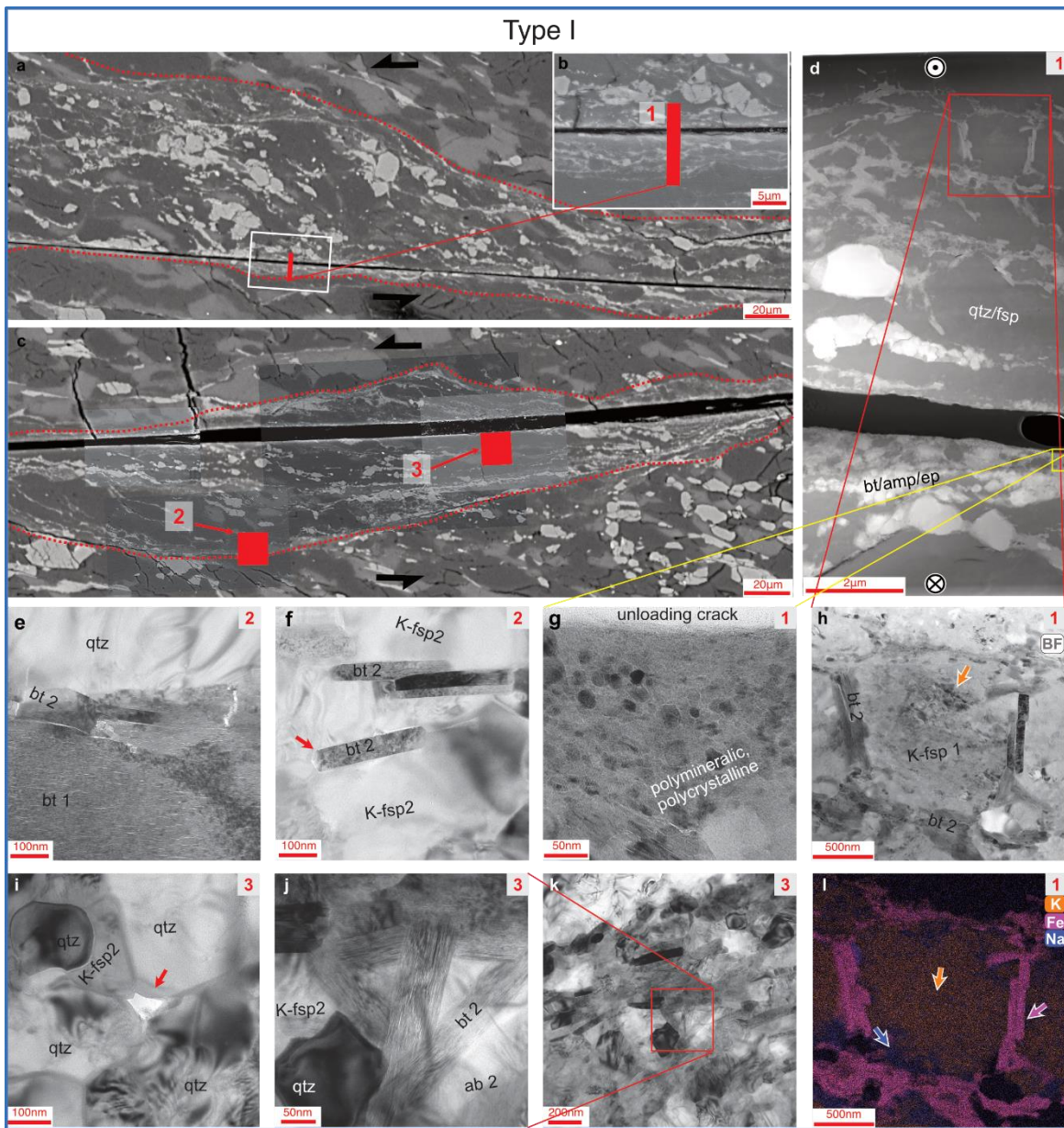


Figure 6: TEM microstructural observations in experiment Type I. (a): SEM overview of locations for TEM sections within high strain experiment 618NW. Rectangular sections are foils cut with a pyramidal technique to allow TEM sections parallel to the thin section orientation. Elongated section 1 is a classical, vertical foil, which was oriented perpendicular to the shear zone and direction (d). Numbers indicate the TEM section appearing in top right corner in (d-l). Images (d-g, i-k) are high resolution TEM images, (h) is a STEM BF image with corresponding chemical element map in (l). Note the similar microstructures on SEM (a-c) and TEM (d) scale. (e) shows a typical overgrowth of primary bt1 by bt2. (f) shows several bt2 grains growing between K-fsp grains. Note the habit of bt2 crystals. (i) shows a pore with concave boundaries at a quadruple grain junction (arrow). The qtz grains are partially dislocation free and partially full of dislocations. (j) shows growth of bt2 grains between fsp and qtz. The qtz is completely dislocation free. (k) is an overview image for (j). (k) also shows phase mixing and a general alignment of elongated bt2/amp minerals.

Locations of (g) and (h) are marked in (d), a chemical map for (h) is shown in (l). (g) is a highest strain/most localized strain zone (overview in (d)). Light grey on the left is epoxy filling out the opening crack. Note the lattice fringes still visible in grains <15 nm. Just a few μm from (g) (see overview in (d)), a K-fsp grain (h, l) is overgrown by Fe-rich bt2 (right, pink arrow in (l)). The K-fsp has many dislocations (middle, orange arrow in (h)). Note also the chemical zonation of the K-fsp (orange arrow) towards the Na-richer/K-poorer rim (left, blue arrow) (l).

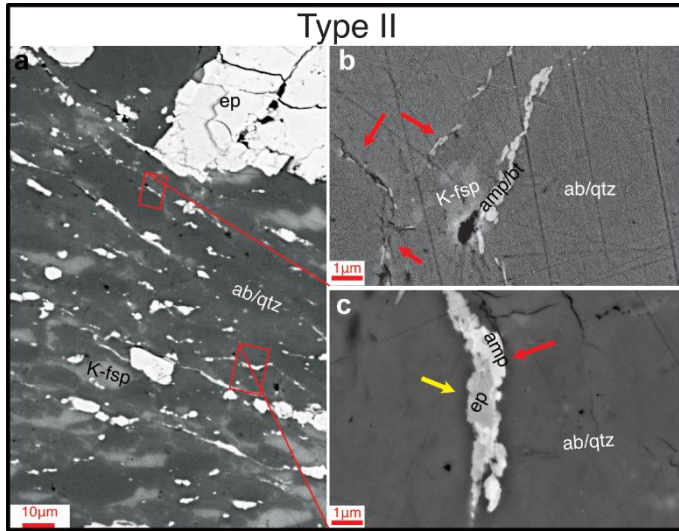


Figure 7: SEM images of Type II experiment 670NN with various DPC-characteristic microstructures. (a): Overview image, located in the center of the sample, shortening vertically. (b) highlights nucleated bt2 at grain boundaries. (c) shows bt1 (yellow arrow) overgrown by bt2 and/or amp (red arrow).

3.3 Microstructures – Type II experiments

In Type II experiments, the first stage of deformation occurs only after the final experimental P,T-conditions have been reached and the sample is at differential stress (exp. 662NN versus 630NN). The first investigated stage is stopped at peak stress (exp. 662NN), with a slight barreling, which indicates that the strain is distributed preferentially in the lower part of the cylinder (Fig. 2d). SEM investigations show that there are conjugate shear bands developing. These are oriented $\sim 30^\circ$ to the core axis (Fig. 4g) and are connected to the corners of the bottom piston. They offset the initial layering or foliation slightly (Fig. 4g), and the step-overs of the initial bands lead to openings, where porosity is induced. However, the orientations of some of the shear bands can be parallel to the preexisting mylonitic bands and do not follow crosscutting orientations in many parts of the sample (Fig. 4i, yellow dashed lines). Hence, these offsets do not define where deformation localizes. Additionally, rounded pores at grain boundaries appear (Fig. 4j).

With increasing strain, the barreling of the sample becomes stronger and deformation is distributed (Fig. 2e). At this stage, there is no geometric evidence for a finer grained or bt/ep-rich band to localize strain preferably with respect to a coarser or qtz/fsp-rich band. Distributed across the whole sample, minerals appear more elongated (image analysis yields approximate grain aspect ratios of feldspar grains of 1:2.3; initial ratios were closer to 1:2). Especially grain boundaries of feldspars show chemical exchange on a small scale, which lead to new composition of the sub-micron scale (Fig. 4h,k). Biotite grain boundaries are also becoming more irregular and new grains (<100 nm) begin to form in their tails and at other mineral phase boundaries (Fig. 4h,k).

250 At high strains (bulk shortening 40%; exp. 670NN), deformation is further continuing to be distributed in the lower part of the sample. The high amount of shortening results also in material that is being “squeezed” out over the edges of the bottom piston (Fig. 2f), and possibly these areas are most affected by external geometric conditions of our experimental set-up. Therefore, we focus our microstructural observations on the central part of the sample only. There, the fsp and qtz grains are even more flattened than in the medium strained sample (Fig. 4i,l, aspect ratio: $\sim 1:2.6$). The shortening leads to rotation of the initial
255 foliation resulting in a shallower dipping angle than the initial 45° . The elongation of host grains marks two different foliations: One flat, $\sim 75^\circ$ to the core axis/principal stress and one steeper foliation at $\sim 55^\circ$ (Fig. 4i). The former, almost horizontal foliation resembles the preexisting foliation of the mylonite bands, while the latter 55° is related to the sample geometry (i.e. bottom pistons) and partially initially developed as conjugate shear zones. It is important to note that at this stage in Type II experiments, there is no evidence for any localization in a narrow band.

260 The boundaries of two feldspars are reacting and it appears that these zones formed a fine-grained, mostly polymineralic, mixture (Fig. 4l). Fine-grained, elongated minerals (bt2 and amp) also occur at grain boundaries of other phases (Fig. 7b). The size, orientation and shape of the small bt2 indicate that these grains must have grown during the experiment. Just as in Type I experiments, primary bt1/ep1 grains are overgrown by small bt2/amp (Fig. 7c). This chemical change and growth also indicate that the grains must have formed during the experiment, i.e. they are synkinematic.

265 On the TEM scale, we chose one area in the maximum strained sample (exp. 670NN) to investigate the tail of an elongated K-feldspar grain and the transition into albite (see above; Fig. 8a). The primary K-fsp visually has an extremely high dislocation density (Fig. 8b), whereas at its rim, new small K-fsp and ab grains occur with partially no dislocations or very low dislocation density (Fig. 8b). The K-fsp and ab are only slightly mixed but, in some areas, very fine-grained ab is found within the initial and new K-fsp grains (Fig. 8b). Pores (~ 50 -60 nm) are observed at grain triple-junctions and grain boundaries (Fig. 8c,d), and
270 the finest observed grains have a similar grain size range (Fig. 8d). No significant changes in the chemical composition of the initial K-fsp towards the ab boundary were observed.

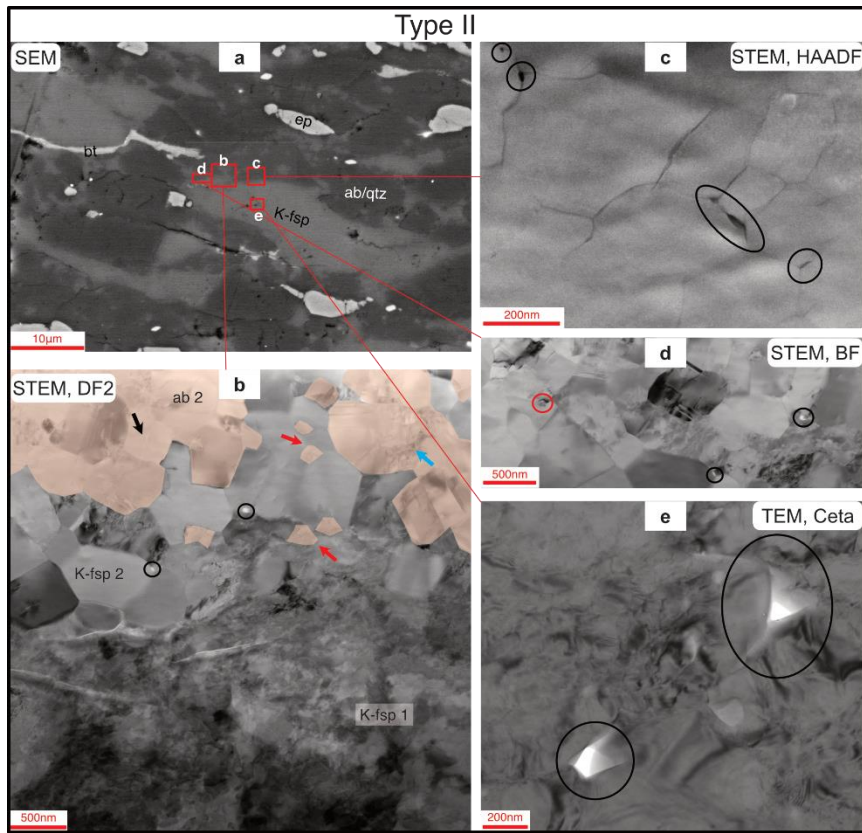


Figure 8: TEM of phase boundary between K-feldspar and albite in high strain Type II experiment 670NN. (a): SEM image shows location of TEM foils at the rims of larger host grains. The K-fsp/ab interface does not appear sharp at this magnification, indicating that the resolution is not sufficient to resolve the grain sizes. (b): TEM foil, DF2 image with ab highlighted based on chemical maps (Supplementary). K-fsp1 shows a high dislocation density, while dislocation-free grains of K-fsp2 and ab2 (like the black arrow) occur at the phase boundary. Blue arrow points at dislocations inside of fine, newly formed ab2. Grains such as indicated by black or blue arrows are counted as large (L), isolated and small grains of one fsp inside of other fsp phase (red arrows) are counted as small (S) in the following grain size analysis Fig. 9. Black circles in (b, c, d, e) highlight open pores at triple-grain junctions and grain boundaries. In (d), red circle shows a nucleated grain with the same size as open pores in the same zone. Pores in (c, d) are in the polymineralic area containing K-fsp2 and ab2, while the pores in E are within the K-fsp1 host grain.

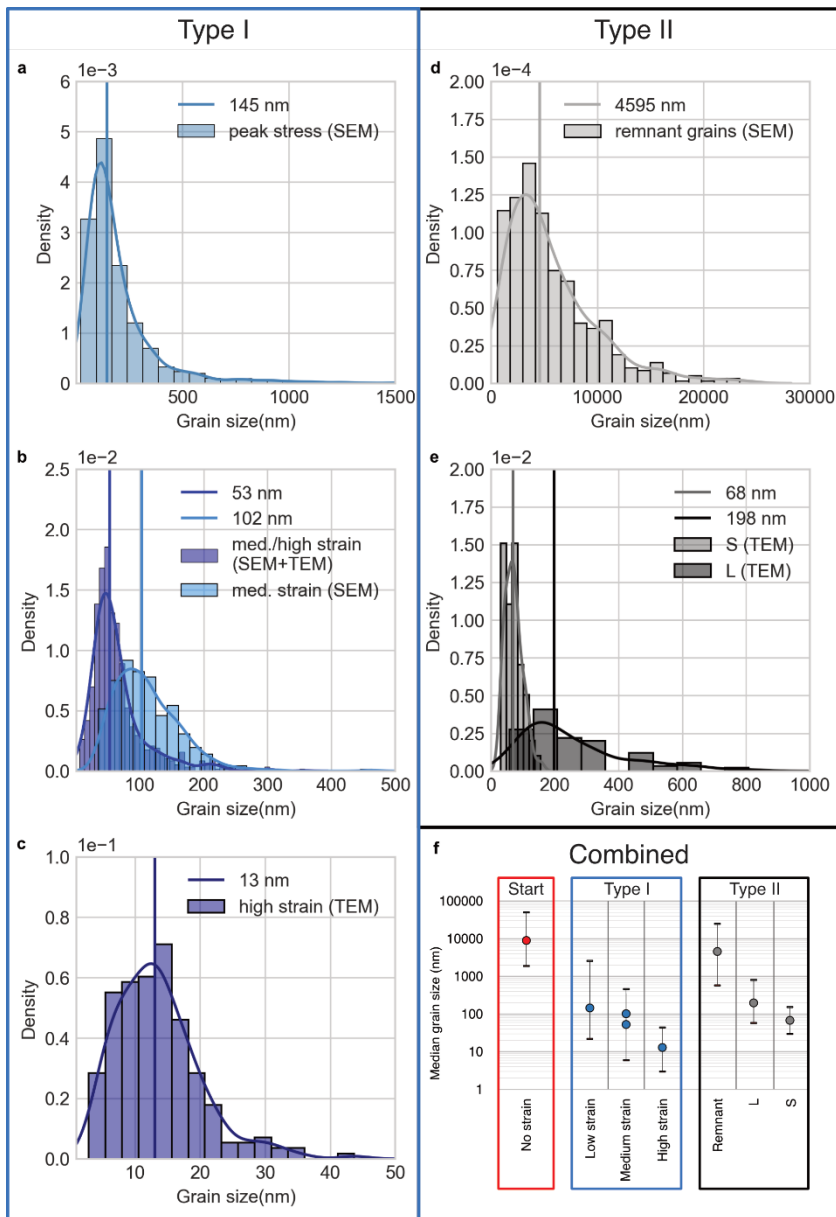


Figure 9: Grain size distributions in pre-fractured (Type I) and not pre-fractured experiments (Type II) and a comparison with the starting material. Grain size reduction to distribution (a) occurs at peak stress, reduction to (b) after shearing to medium strain, and (c) occurs only after shearing to very high strains. All these grain sizes are still present and were measured in the high strain experiment 618NW. (d) shows the grain size distribution of elongated large grains in high strain sample 670NN, while (e) shows the measured grains in fine-grains zones between these grains. “S” refers to isolated small grains, while “L” refers to larger, often clustered grains (see Fig. 8). For a discussion on grains accommodating the strain, see text. Grain size reduction in two types of experiments on a logarithmic scale. The lines in the bar diagrams indicate median values. The dots in F represent the median grain size, while the error bars indicate the maximum and minimum grain sizes measured in the respective samples/zones. Type I grain sizes are measured in sample 618NW, Type II grain sizes in sample 670NN. All grain sizes are formed at different stages but still occur at the same time in the final experimental microstructure. See for text for definitions and further discussion if all grains still accommodate the strain in the final microstructure.

3.4 Grain size evolution in both experiment types

We investigated the grain sizes in both types of experiments and grouped these into different strained zones based on the microstructural observations and spatial distributions (Fig. 9).

280 In Type I experiments, the grain sizes are measured in different zones of the localized shear zone of sample 618NW under the assumption that they represent the different stages of deformation (e.g. grain size gradients on SEM and TEM scale in Fig. 6). In Fig. 9a-c we differentiate between peak stress, medium strain, and high strain grain sizes. We assume that the corresponding grain sizes are formed at the respective strain stages. At peak stress in Type I experiments, the grain size in the deforming zone is reduced to a median grain size of 145 nm (equivalent to microstructures in Fig. 5b). There are still remnant grains from the initial brittle fracture (Fig. 5a) covering a nm to μm range (22 nm to 2.6 μm ; Fig. 9a). Then, with increasing strain, grain sizes
285 are reduced in these areas (as seen in e.g. Fig. 5e,6e,f,h-l) to medians of 102 and 53 nm (measured by SEM and TEM, respectively; Fig. 9b). This grain size is at the lower limit of SEM and upper limit of TEM resolution. Therefore, both together give a more indicative grain size range of 6 to 463 nm. The grain size in the narrow (<200 nm) zone in Fig. 5d,g reduces to a median of 13 nm with a range of 3 to 44 nm (Fig. 9c).

It is to be noted that with each grain size reduction step also the zone in which the strain is accommodated narrows (see
290 companion paper 2). As a result, with a constant vertical displacement, the actual strain rate accelerates, and the grain size reduces gradually. This relationship becomes relevant for the stress-strain evolution and grain size sensitivity of the samples which is discussed in detail in companion paper 2.

In Type II experiments, two types of grain sizes are differentiated. Very fine grains, that are difficult to resolve with the SEM, occur at phase boundaries of large feldspars. The large grain size is manually drawn in SEM images and a median of 4.5 μm ,
295 with a range of 580 nm to 25 μm is obtained (Fig. 9d). These largest grains are only ~50% smaller than the starting grain size of the initial ultramylonite (see also Fig. 10) opposed to the very fine grains. In the fine-grained zone between the remnant grains, two different grain sizes are differentiated: (1) small grains (named group “S”) are isolated phases between the other type of feldspar; (2) larger grains (named group “L”) represent monomineralic clusters of small grains (derived from one fsp). We measure median grain sizes of 198 nm in the “L” feldspars, with a range of 58-808 nm (darker histogram in Fig. 9e). The
300 “S” grain size median is 68 nm with a range of 30 to 155 nm (brighter histogram in Fig. 9e).

4 Discussion

4.1 Deformation mechanisms

4.1.1 Evidence for dissolution-precipitation creep

305 In both experiment types, after deformation, all phases of the starting material (qtz, both fsp, bt/ep) are still observed, even in fine-grained zones, but slight chemical changes occur as well as the formation of new amphibole. We measure chemical changes in the feldspars, too (e.g. Fig. 6h,l). Furthermore, some biotites have an Fe-enrichment with respect to the previous biotites. In addition, the two groups (old and new grains) can be discriminated by high dislocation density versus almost dislocation free grains. The chemical changes and gradients show that the grains must have reacted during the experiment
310 indicating existence of local chemical potentials for ionic exchange at experimental P,T-conditions.

With the low stresses measured for the deformation (far below the Goetze criterion $\sigma_D \geq P_c$), it is very unlikely that the extremely fine grains (observed in the high strain samples in Figs. 6g, 9c) have formed by brittle processes. The elongated minerals are parallel to the shear plane (Fig. 6d,f,k) or filling gaps between grains as in Fig. 6h. Coupled with the densely packed phases, small grain size and narrow size range, and their distinct crystal habitus, this geometry suggests stress-oriented growth (Imon
315 et al., 2004) rather than rigid body rotation of grains after fracturing.

In Type II experiments three types of feldspar grains were identified, where relict grains have a high dislocation density, whereas L (large) and S (small) grains have distinctive low dislocation densities (as in Figs. 8 and 9). The newly formed L-grains have a broad grain size range, while the S-grain size range is narrower, but there is an overlap in their distribution. The newly formed grains have only a very slight change in chemistry compared to their host grain/porphyroclast. (Slight) phase
320 mixing between ab2 and K-fsp 2 (Fig. 8b), dislocation-free grains, and partially well-developed grain triple junctions (Fig. 8b) indicate nucleation and growth rather than dynamic recrystallization via subgrain or bulging recrystallization. The occurrence of pores along grain boundaries and grain triple junctions (while the nucleated grains “S” have a similar size compared to that of the pores) shows that the opening and filling of the pores occurs simultaneously through grain boundary sliding and rapid nucleation within the opened cavity (Ebert et al., 2007; Herwegh et al., 2011; Herwegh and Jenni, 2001; Linckens et al., 2011,
325 2015). The formation of these synkinematic boundary pores has first been described for geomaterials by Etheridge et al. (1984) and rock analogue materials by Ree (1994). More recently, the process has been referred to as creep cavitation (Fusseis et al., 2009; Gilgannon et al., 2017, 2021).

All observations of mechanical data, chemical changes, dissolution and growth of minerals, and grain size reduction given above clearly demonstrate that the predominant deformation mechanism in both types of experiments is dissolution
330 precipitation creep (DPC) rather than dislocation creep or frictional flow. The chemical potential mentioned above promotes diffusive transport, while opening of nanometer-scale voids in dense high strain areas by grain boundary sliding (GBS) serve as nucleation sites for new precipitates. Any DPC mechanism results in a polymineralic mixture, where phases are pinning each other inhibiting/reducing grain growth (e.g. see Herwegh et al., 2011 and references therein), thereby enhancing efficiency of DPC in fine-grained pinned parts of the polymineralic mixture. The pinning leads to a stable microstructure and

335 differentiates the DPC process in our polymineralic rocks from DPC in monomineralic systems. Phase mixing and pinning may be the reason for the onset of DPC in our samples at considerably lower temperatures than, for example, observed in monomineralic quartz (e.g. Ghosh et al., 2022, 2024; Richter et al., 2018).

The DPC in our experimentally deformed polymineralic granitoid mylonites has far reaching consequences. The occurrence of viscous deformation in our samples at 650°C versus mostly frictional deformation in coarser granites in other experimental studies conducted at the same temperature (Pec et al., 2016; Pec and Al Nasser, 2021) demonstrates that the initial grain size is very important for a brittle to viscous transition (Sun and Pec, 2021). In our case, an ultramylonite with a median grain size of ~10 µm is sufficient to activate DPC at relatively fast strain rates of 10^{-6}s^{-1} and locally even faster. At these experimental conditions, onset of DPC cannot be predicted from monomineralic quartz flow laws (e.g. Fukuda et al., 2018; Ghosh et al., 2022; Hirth et al., 2001) and should be investigated experimentally for other polymineralic rocks in the future also.

345 **4.1.2 Rate-limiting steps**

The rate of DPC is controlled by the rate limiting step of one of the major processes (dissolution, transport, precipitation; Gratier et al., 2013; Raj, 1982; Rutter, 1976). In monomineralic systems or in metals, transport (i.e. diffusion) generally is assumed to be rate limiting. In these chemically and crystallographic simple systems, the dissolution and precipitation process is described by well-defined source and sink terms (e.g. Raj, 1982). Chemical potential differences exist in monomineralic systems as the consequence of different orientations of grain boundaries with respect to deviatoric stress (Raaj and Ashby 1982), but the quantification of localized values of the chemical potential are currently impossible to be determined for polymineralic systems. For example, in addition to local stresses, the chemical potentials are also dependent on the (metastable) equilibrium conditions of the mineral phases reacting with each other. It is observed that K-feldspar shows chemical gradients towards the rim (Fig. 6h,l) and new phases with different composition nucleate in our samples – both in Type I and Type II experiments. Hence the chemical potential gradient is a combination of local stresses and local chemical equilibrium of different phases and very difficult to define. Consequently, a quantitative evaluation is not feasible. In the following section, we attempt to assess these parameters qualitatively.

In pressure solution or diffusion creep models, minerals dissolve at high pressure sites and precipitate at low pressure sites (Arzt et al., 1983; Elliott, 1973; Gratier et al., 2009; Niemeijer et al., 2002; Weyl, 1959; Wheeler, 1992). Often, the precipitates overgrow an existing grain leading to a change in its shape. In polymineralic systems, as in this study, an extremely fine-grained, partly equigranular, polymineralic mixture of phases indicates that the precipitates preferably nucleate and grow, as this mixture could not have formed through dynamic recrystallization (Ebert et al., 2007; Herwegh et al., 2005, 2011; Linckens et al., 2011, 2015). Another component during dissolution and precipitation is the transport of the material from source to nucleation site. This transport is assumed to be diffusive along grain boundaries. The phase mixing in the fine-grained zones indicates that the diffusion length scale typically exceeds at least one grain size length in the deforming zone (largest grains being ~200 nm). K-fsp1-ab1 phase boundaries in Type II experiments do not show much phase mixing or transport of, e.g. bt2 or amp into the fsp. Similar observations were made in the lower strain areas in Type I. Hence, the diffusive transport length

is at least the grain size and is influenced by porosity/permeability. Therefore, the transport of elements through the system might be more efficient than in classical pressure solution (Wheeler, 1992) and is enhanced by initial porosity from the initial brittle fracture and phase mixing as in Type I experiments. By applying synchrotron-based scanning small-angle X-ray scattering a for the natural starting material of our experiments, Schrank et al. (2025) demonstrated pore sizes from 1 - 280nm and a pore volume in the range of percentages for this dynamic nano-porosity. All these observations imply that the fluid advection through initial porosity plays an important role in driving DPC in both nature and experiment (see section 4.1.4). The growth of grains, as it is typically observed in monomineralic systems, is inhibited/reduced through pinning by other phases. Brodhag et al. (2011) demonstrated that under static conditions, coupled grain coarsening occurs, where the aggregate coarsening depends on growth kinetics, as well as amount, size and spatial distribution of the slow growing phase (Herwegh et al., 2011). Ebert et al. (2008), Herwegh et al. (2005, 2011), and Linckens et al. (2011, 2015) showed that coupled grain coarsening also occurs in naturally deformed polymineralic mylonites, with grain sizes being indicative for specific physical deformation conditions. Based on our new experimental insights we infer that the dissolution rate of minerals is enhanced by a high dislocation density in parent grains, and at the same time assisted by chemical driving potentials. Fast dissolution rates may counteract grain growth if newly nucleated grains are being dissolved as well. In the case of our granitoid mylonites, the combination of the three key parameters leads to more efficient strain accommodation than in coarse or monomineralic samples, accommodating fast strain rates of $>10^{-3}\text{s}^{-1}$ (see companion paper 2).

In high strain samples of Type I experiments, there is no evidence for one of the phases being an inhibiting factor in the overall dissolution and precipitation cycle, since all the phases, including quartz, are observed in the highly localized zone. Hence, instead of a single volumetrically dominating matrix phase with small amounts of a second phase (Herwegh et al., 2011), in our experiments pinning of all phases (see quantities above) by each other leads to a locally stable microstructure in a small-scale micro-chemo-mechanical equilibrium that is specific to the polymineralic assemblage. The dominant operation of DPC because of a stable small grain size results in a weak rheology and viscous behavior for all phases in the system at low temperatures but fast strain rates, which would not be possible in monomineralic systems. This is supported by extrapolation to natural conditions and comparison with various quartz and feldspar flow laws in companion paper 2.

4.1.3 Role of feldspars for dissolution-precipitation creep

With stable and coexisting qtz, K-fsp, ab, ep/bt and amp, our polymineralic system is too complex and our current knowledge too limited to combine the role of the growth/dissolution kinetics and spatial distribution of individual minerals quantitatively. However, we can focus on the feldspar system, given its volumetric dominance in our samples, which is ~54 wt% (albite 37 wt% & K-feldspar 17 wt%). Especially at the interface of K-fsp and ab grains in Type II experiments, the role of reactions between feldspars towards their stable end member composition becomes important (e.g. Fig. 8). As known for the different solvi in feldspars (e.g. Fuhrman and Lindsley, 1988), the K-fsp component in the Na-feldspar and the ab component in the K-feldspar is increasing with temperature. Fig. 10 shows the expected feldspar compositions for starting material and experiment in a calculated binary phase diagram. In our samples, we see this change in compositions qualitatively, for example

in Fig. 6l. Owing to this exchange, we observe in SEM images the backscatter contrast of albite becoming higher in areas of contact between K-fsp and ab, where compositions approach equilibrium compositions. Hence, in our granitic system at 650°C and 1.2 GPa, chemically induced driving potentials for dissolution and precipitation of feldspars become important, especially in fsp-rich areas like band type B (Fig. 1b,d).

405 It has been inferred by various studies from the field and from experiments how the reactivity of feldspars might lead to enhanced solubility and efficient grain size reduction (Fitz Gerald and Stünitz, 1993; Hippertt, 1998; Marti et al., 2018; Stünitz and Fitz Gerald, 1993; Stünitz and Tullis, 2001). This is also supported by the very fine grains nucleating at the phase boundaries between K-fsp and ab in experiment Type II (Fig. 8). The observed microstructural evolution is very similar to feldspar-rich polymineralic experimental samples of e.g. Marti et al. (2018) or natural examples of e.g. Behrmann and
410 Mainprice (1987), Lee et al. (2022), or Menegon et al. (2008), where DPC accommodated or accompanied by mineral reactions is inferred, pointing at an applicability of our observations beyond granitic systems. This focusses the key parameters needed to activate the observed type of DPC to fine grain sizes, phase mixing and chemically induced driving potential.

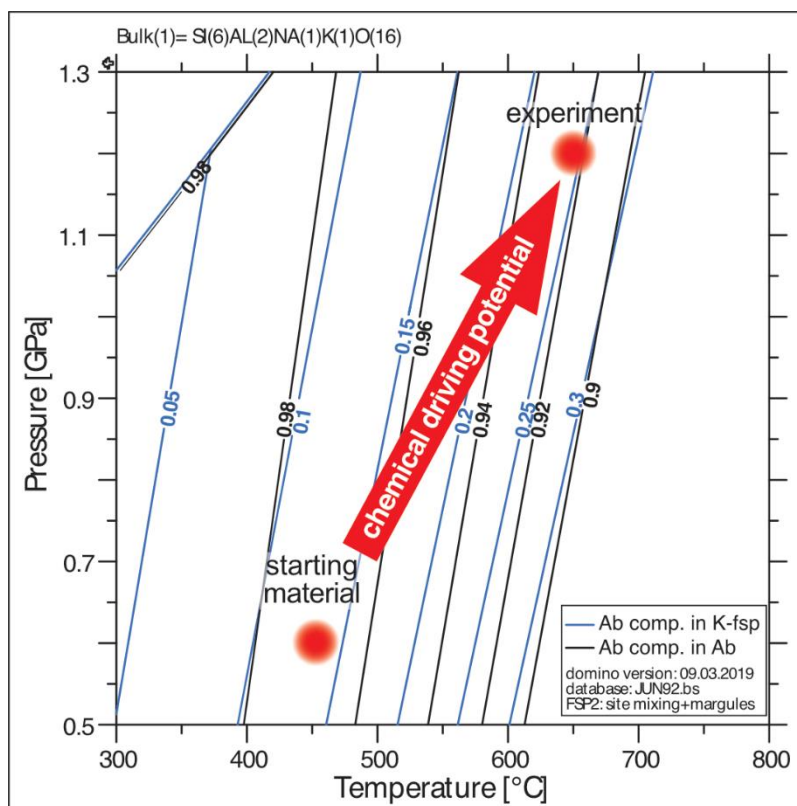


Figure 10: Pressure-Temperature phase diagram calculated for a two-feldspar system K-feldspar (K-fsp) and albite (ab). The difference in feldspar composition between the starting material and that of the experimental condition induces a chemical driving potential towards the stable composition at experimental P,T-conditions, where ab incorporates more K-fsp component and K-fsp more ab.

4.1.4 Role of porosity

The observed DPC differs from classical diffusion creep (Nabarro-Herring creep or Coble creep) by possible advective transport of fluid and/or diffusional transport in a local bulk fluid or distributed fluid film. Porosity and permeability affect the deformation processes by enhancing fluid pathways. Therefore, fluid pathways and porosity are important parameters to evaluate and compare between experiments. In our experiments, two types of porosity are observed: (1) brittle dilatation forming during loading or at peak stress, and (2) shearing induced creep cavities forming during DPC.

The first type of porosity is observed in Type I experiments, where the initial fracture formation results in the creation of a significant quantity of localized porosity (Fig. 4). This porosity and permeability permit the migration of fluid to the comminuted grains. In conjunction with the fine grain size, this porosity is instrumental in achieving near steady-state conditions in an efficient manner. With increasing viscous deformation, the grain size decreases further, the fractured aggregates compact, and the porosity decreases. With increasing strain, local stress differences may allow new phases to grow in between stretched and separated grains, like the K-fsp1 in Fig. 6d,h with bt2 growing around it. Especially at highest strain, the original fracture-induced porosity is almost entirely lost through compaction and growth of minerals (Figs. 4-6).

In Type II experiments, conjugate shear zones form, as seen in low strain experiments, where locally minor porosity is created. The deformation does not localize in these zones, but this local porosity might serve as fluid pathways. At high strains, these openings are closed (Fig. 4). The main shear plane follows the primary mylonitic foliation and is partially influenced by sample geometry (e.g. bottom piston) and does not follow these initial openings. Hence, the role of the initial porosity is negligible, especially compared to pre-fractured samples.

The second type of porosity forms during the deformation at triple grain boundaries and grain boundaries (Fig. 8) and is a result of grain boundary sliding (GBS). New grains nucleate in these cavities (Fig. 8d). Indications for these creep-cavities (Fusseis et al., 2009) are observed in both types of experiments (Figs. 5c, 6i, 8). In both experiment types, only very little of this type of porosity remains, because nucleation and growth rapidly and efficiently fill any cavity formed during DPC and GBS. Hence, this porosity is highly dynamic, where individual pores are created while others disappear resulting in a dynamic permeability. As the pores appear isolated, diffusive mass transport must occur along grain and interphase boundaries. This process is likely to be accommodated by GBS in a manner similar to the granular fluid pump during creep cavitation (Fusseis et al., 2009). By applying synchrotron-based scanning small-angle X-ray scattering a for the natural starting material of our experiments, Schrank et al. (2025) demonstrated pore sizes from 1- 280nm and a pore volume in the range of percentages for this dynamic nano-porosity indicating the importance of nano-porosity for DPC and mass transfer. Note that our experiments indicate that relatively small fluid quantities and their continuous recycling in the deforming polyminerale aggregates are sufficient, not necessarily requiring open systems with large fluid quantities migrating through shear zones.

4.2 Applications to nature: The role of grain size and preexisting fracture for the onset of DPC in polymineralic shear zones

450 4.2.1 (Steady state) grain sizes and flow stresses

An important point for reconstructing the shear zone evolution in natural rocks is whether the grain size is stable at certain strain rates, pressures, and temperatures. One key factor for models will be the surface energy of a system that is increased by grain size reduction or reduced by grain growth. The grain growth and grain size reduction rates may balance each other at steady state. From such an assumption, combined with internal energy contributions, (i.e., dislocation density), grain size
455 piezometers (e.g. Derby, 1991; Twiss, 1977), and P,T & strain rate sensitive paleowattmeters (Austin and Evans, 2007, 2009) are developed. Our experiments show that an additional parameter for the total energy budget of the shear zone in both settings is the chemical driving potential of constituent phases (e.g., feldspars and biotites). The chemical driving potential of metastable phases enhances the dissolution rate in samples during DPC and thus may contribute to enhancing the strain rate. In any case, the metastability needs to be considered in the energy balance for potential steady state conditions. At the same
460 time, pinning inhibits the grain growth leading to a mechanically weak stable microstructure. Many case studies estimate stresses and strain rates from grain sizes calibrated for dislocation creep regimes in monomineralic systems that lack chemical driving and pinning components. Consequently, neither piezometers nor paleowattmeters are thought to be applicable to DPC in polymineralic rocks.

In some recent studies, paleowattmeter have been applied to diffusion creep systems or compared to systems where a diffusion
465 creep component might be contributing to the deformation (Ruh et al., 2024; Tokle and Hirth, 2021) but chemical driving potentials, nucleation rates, or other diffusion parameters and polymineralic chemistry are not included. It is implied that feldspar grain size reduction leads to a very fine grain size, resulting in deformation in the diffusion creep regime, while quartz remains deforming by dislocation creep. Tokle and Hirth (2021) have calibrated the paleowattmeter to match quartz experiments. To evaluate the applicability of paleowattmeters to the granitoid system at our experimental conditions, we first
470 need to assess the representative stable grain size at corresponding stresses and strain rates in our experiments.

Provided the strain is progressively localizing in narrower zones during the whole deformation history, then the strain rate is increasing in the narrowing deforming zone. In experiment Type I we assume that at peak stress the coarse, earlier formed, grains were predominantly accommodating strain in the originally wider zone. During subsequent stages, the near-steady-state-stress is accommodated within the more localized zones consisting of smaller grains. We hence obtain two systems in
475 the same experiment, with two stresses (peak and flow stress), and two different grain sizes and strain rates. This technique gives parameters to evaluate a potential strain rate - grain size sensitivity in these rocks, which we also used to estimate approximate grain size exponents for a constitutive equation in companion paper 2.

Although strain in the not-pre-cracked Type II experiments is more distributed, the observed mechanical weakening and localized grain size reduction at grain/phase boundaries indicates that these experiments have not reached steady state
480 microstructures yet. The strain rate is not homogeneous, and two end member strain rates are active at the same time: A slower

strain rate for the large grains and a fast strain rate for the narrow fine-grained zones in between. If mostly the fine grains are accommodating the strain (interconnected weak layers; e.g., Handy, 1994), the bulk strain rate will underestimate the actual strain rate of these fine-grained zones.

485 The DPC of type II shear zones is distributed over larger regions of the samples and there still is a large volume of primary grains preserved. From observations of the pores, we infer that “S”-grains represent the grain size of newly nucleated grains, followed by grain growth to a grain size of “L” grains. This grain size reduction achieved through nucleation during DPC and GBS is very efficient and leads to grain sizes comparable to “medium strain” stages in Type I experiments (Fig. 9). The continuous deformation of a not pre-fractured rock would either lead to a homogeneous sample with L grain sizes, or to localizing in throughgoing narrower shear zones, where the grain size will decrease after sufficient strain and further grain size
490 reduction by nucleation and phase mixing.

Tokle and Hirth (2021) have shown that the calibrated piezometer would overestimate the grain size of other grain growth laws if they are based on microstructures where pinning with secondary phases occurs (i.e. Wightman et al., 2006). Indeed, inserting approximate values from our experiments into the Tokle and Hirth (2021) calibrated paleowattmeter results in grain sizes of ~ 260 nm for 500 MPa differential stress and 10^{-3}s^{-1} strain rate (as in Type I experiments) or ~ 2 μm for 200 MPa
495 differential stress and 10^{-6}s^{-1} (as in Type II experiments), which is one order of magnitude larger than the observed grain sizes developed through DPC in our experiments. Even if a faster strain rate is assumed in the localized zones, the piezometer overestimates the grain size or would underestimate the actual stresses in natural polyminerale mylonites, where DPC is the dominant deformation mechanism.

Despite the lack of a grain growth inhibiting term and a term for chemical driving potential or nucleation rates, the original
500 paleowattmeter (Austin and Evans, 2007), predicts grain sizes of ~ 20 nm in Type I experiments and ~ 150 nm in Type II as flow stresses and strain rates of monomineralic quartz aggregates. Surprisingly, these values are in agreement with the range of grain sizes observed during the experiments: 150-10 nm in Type I, 200-50 nm in Type II. Therefore, our study opens the question whether in some cases the paleowattmeter could predict the stresses from grain sizes in polyminerale shear zones that formed through DPC. In this case, pinning in polyminerale rocks must be counterbalanced by very efficient/fast nucleation
505 and growth rates leading to the self-sustaining and balanced system with a stable grain size. It should be mentioned though that creep data of quartz for these grain sizes and strain rates are not available from experiments, so that the extrapolation goes way out of the existing data range. More experiments are needed to confirm this observation and to improve corresponding flow laws used to build the paleowattmeters.

For a quartzo-calcitic polyminerale system (calcareous shales), Gratier et al. (2023) recently published a flow law including
510 the diffusive and grain boundary sliding parameters. It is to be noted that for granitoid rocks still a flow law is required, which considers reaction kinetics, chemical driving forces, diffusion pathway lengths and other rate limiting steps to quantitatively describe the stress/grain size relationships.

4.2.2 Strain localization mechanisms

Under natural greenschist facies metamorphic conditions, ample evidence exists that in the middle to upper granitoid crust deformation is strongly localized in fine-grained polymineralic ultramylonites being surrounded by coarse-grained low to moderate strain domains (Choukroune and Gapais, 1983; Fitz Gerald and Stünitz, 1993; Marquer et al., 1985; Stünitz and Fitz Gerald, 1993; Wehrens et al., 2016). Two general types of strain localization have been observed: (i) classical gradual strain localization across strain gradients and (ii) quasi abrupt changes along sharp contacts from non/weak- to highly localized deformation. (ii) Has been interpreted as strain-localization via brittle precursor microfractures (e.g. Fitz Gerald and Stünitz, 1993), followed by a viscous deformation that may or may not follow a mesoscopic brittle fracture (e.g. Fitz Gerald and Stünitz, 1993; Stünitz and Fitz Gerald, 1993; Mancktelow and Pennacchioni, 2005; Pennacchioni and Mancktelow, 2007; Wehrens et al., 2016). The mesoscopic fracture may be absent, indicating that the important features for the initiation of viscous deformation are the microfractures (which may accompany the mesoscopic fracture as a damage zone). In contrast, (i) evolved as a progressive ductile strain localization (e.g. Choukroune and Gapais, 1983; Marquer et al., 1985; Wehrens et al., 2016). In the case of the Aar Massif (Basement outcrops in central Switzerland), Wehrens et al. (2016) observed a tendency for (i) and (ii) localization in plutonites with granitic and granodioritic compositions, respectively. A ~5-10 vol% increased content of interconnected sheet silicates in the latter case, is thought to promote a viscous strain localization behavior.

Although having been performed at amphibolite metamorphic conditions, our new Type I and II experiments can now contribute substantially to an improved knowledge on the difference between these two end-member strain localization behaviors observed in nature under greenschist to amphibolite facies metamorphic conditions. Besides extrapolation to classical long-term viscous crustal strain rates, our experiments might also give insights into more episodic deformation styles as is inferred for example for slow slip events or the viscous precursors of seismic runaway cycles. Note that for some such cases, our chosen experimental conditions (e.g. temperature, strain rate) may be identical or at least close to the natural ones not requiring extrapolations over orders of magnitudes between experiment and nature. Our experiments demonstrate that a definite relationship between bulk and localized strain rates, grain size evolution and the role of grain size, porosity, and local phase composition exists for DPC in polymineralic rocks: Owing to its grain size sensitivity, diffusion /dissolution precipitation creep (DPC) is known to dominate as deformation mechanism in fine-grained rocks at elevated temperature conditions resulting in a weak rheology of the deforming rock. In the case of granitoid rocks with grain sizes in the range of millimeters to centimeters, grain sizes are too large to allow for an efficient deformation by DPC. Hence, to activate DPC in an initially coarse-grained natural rock, some precursor mechanism reducing the grain size is always required (leading to phase mixing and subsequent activation of DPC): e.g. (1) a brittle grain size comminution (Bürgmann and Dresen, 2008; Fitz Gerald and Stünitz, 1993; Menegon et al., 2013) or (2) the initiation of dynamic recrystallization (dislocation creep) until a fine grain size is reached (Bercovici and Skemer, 2017; Ricard and Bercovici, 2009; Rutter and Brodie, 1988; Schmid, 1982) or (3) reaction with nucleation of small sized new phases (Ceccato et al., 2018; Fitz Gerald and Stünitz, 1993; Mansard et al., 2020a, b; Marti et al., 2017, 2018; Menegon et al., 2008; Stünitz and Tullis, 2001).

Similar to the observed end member localization behaviors in nature, our experiments show two types of grain size reducing mechanisms in granitoid polymineralic rocks: (1) Brittle fracturing, which reduces the grain size significantly (Fig. 4d); (2) Reaction of different phases due to chemical driving potentials when being exposed to a differential stress (Figs. 5, 6, 7, 11). Accompanying grain boundary sliding, DPC is directly activated at phase boundaries in the latter case. This relationship of processes demonstrates that initial brittle deformation at the microscale, combined with other processes, is one end-member precursor to localize strain. Similar microstructures have been observed in many natural settings by, e.g. Bürgmann and Dresen, 2008 (and references therein), Fitz Gerald and Stünitz (1993). Mesoscopic fractures may or may not be associated with such brittle microscale grain comminution (Goncalves et al., 2016; Mancktelow and Pennacchioni, 2005; Pennacchioni and Mancktelow, 2007), but also reaction weakening can be very effective instead, as also observed on natural faults (by e.g. Ceccato et al., 2018; Menegon et al., 2008). Hence, both, strain localization and more distributed strain, may form in granitoids in nature. In both cases the stresses are decreasing or strain rates are increasing due to an increased activity of DPC. In terms of the rheological behavior of the two end member localization mechanisms in nature, our results indicate that fracture-induced strain localization reaches weaker DPC rheologies after lower acquired strains compared to the purely viscous strain localization. Therefore, the lower the deformation temperature in granitoid rocks, the more efficient fracture-induced initiation of DPC-assisted strain localization is. At high finite strains, the mechanical strength of both localization types might be rather similar once polymineralic DPC steady-state microstructures evolved, as can also be inferred from the grain size evolution (Fig. 9f).

An important parameter which is still poorly understood in natural shear zones is the level of inhomogeneity needed to efficiently support localization of strain (Ceccato et al., 2022). At our experimental conditions brittle grain size reduction initiates DPC in a very narrow zone and is always leading to strain localization. The less localized deformation in Type II experiments, i.e., no localized deformation of more fine-grained or more mixed mylonitic bands compared to initial fsp-qtz rich bands indicates that the rheological contrast between the mylonitic bands was not sufficient for strain localization. Thus, not every inhomogeneity in a granitoid rock may serve as an agent for strain localization, so that the rheological evolution of the granitoid system may vary (as in section 4.1.2). The key differences between the deforming systems are the initial grain sizes and porosity. More experiments are necessary to further estimate the influence of phase mixing, porosity and grain size, by e.g. gradually modifying grain sizes and compositions.

As outlined above, pre- fractured shear zones are narrow while deformation in the non-pre-fractured sample is widely distributed. The high strain zones in Type I experiments do not widen during sample deformation. Instead, they stay highly localized although being subjected to the same piston displacement rates as Type II experiments. The reason for this variation in strain partitioning must be related to how deformation energy is distributed within the deforming sample cylinders. The rapid grain size reduction processes in Type I experiments allows for initiating of DPC. Given the small shear zone width in Type I experiments (10-20 μm), deformation energy is distributed in a small volume. At the very fine grain sizes the grain size sensitivity of DPC results in fast strain rates. The opposite is the case for the non-pre-fractured Type II experiments.

Commonly, a strain energy for natural samples can be calculated from the stress and strain of a sample, following piezoelectric relationships (e.g. Austin and Evans, 2007, 2009; Giraud and Giraud-Audine, 2019; Regenauer-Lieb and Yuen, 2003). The defined sample size and strain rate allows us to estimate an energy flux for both types of experiments, which is the total energy dissipated in the sample per time E_{flux} (in J s⁻¹). is a function of (shear/differential) stress σ (in Pa), (shear/coaxial) strain rate ($\dot{\gamma}$) $\dot{\epsilon}$ (in s⁻¹) and unit volume V (in m³) (Handy, 1994b):

$$E_{flux} = \sigma_{shear} \cdot \dot{\gamma} \cdot V \text{ (shear stress),} \quad E_{flux} = \sigma_{diff} \cdot \dot{\epsilon} \cdot V \text{ (differential stress)} \quad (1)$$

In fact, when comparing the energy flux values for experiments 618NW – Type I and 670NW-Type II, the total energy flux in those would be in the same order of magnitude: $\approx 2 \cdot 10^{-7}$ J/s (618NW, Type I) and $\approx 1 \cdot 10^{-7}$ J/s (670NW, Type II) (using values from Table 1, calculations with Eq. 1 and in Table in supplementary). Moreover, Type I experiments show nearly steady state stresses while Type II experiments show continuous strain weakening (Fig. 3). Classically, shear zone widening is observed when strain hardening occurs while shear zone narrowing is the consequence of strain softening processes (e.g. Hull, 1988; Mitra, 1992; Means, 1995). In this context, Type I experiments are interpreted to be microstructurally energetically optimized resulting in more or less constant shear stress without need for large changes in shear zone width. Our experiments therefore suggest that two energetically stable microstructures exist (Type I and Type II experiments), and it is the grain size evolution path that defines which of the two microstructures determines the strain rate and shear zone width. Here we can again link to nature, with the occurrence of both (see summary Fig. 14 in Wehrens et al., 2016), the fracture induced highly localized granitoid shear zones with abrupt grain size refinements (e.g. FitzGerald and Stünitz 1993) and the classical ductile strain gradients with progressive grain size reduction and the classical transition from proto-mylonites to ultramylonites (e.g. Fig. 9 in Wehrens et al. 2016).

5 Summary and Conclusions

Two types of experiments on a natural granitoid ultramylonite were performed at 650°C and 1.2 GPa confining pressure. In Type I experiments, an early induced brittle fracture served as precursor for the initiation of localized deformation through dissolution and precipitation creep (DPC). The final deformation required only low differential stresses of ~ 500 MPa at fast strain rates of $\sim 10^{-3}$ s⁻¹. In coaxial Type II experiments, deformation initiated through reactions at phase boundaries and led to distributed DPC. The distributed strain resulted in continuous strain weakening to < 200 MPa at strain rates of $\sim 10^{-6}$ s⁻¹.

Evaluation of the strain evolution in the experiments highlights that grain sizes, porosity, and chemical driving potential are important driving forces for the activation of DPC and localization of strain. During deformation, grain growth is inhibited through phase mixing and pinning, which results in continuous deformation in the DPC field. However, the initial grain size and porosity define whether the deformation localizes directly or if distributed small grain size domains are created and are continuously widened. Chemical gradients caused by mineral metastability in combination with the distribution of the minerals/phases at given physico-chemical deformation conditions, may play an important role for both, the localization

processes as well as the preservation of microstructural steady state. Comparisons with other polymineraltic studies indicate
610 that these processes are applicable to a broad range of mineral assemblages.

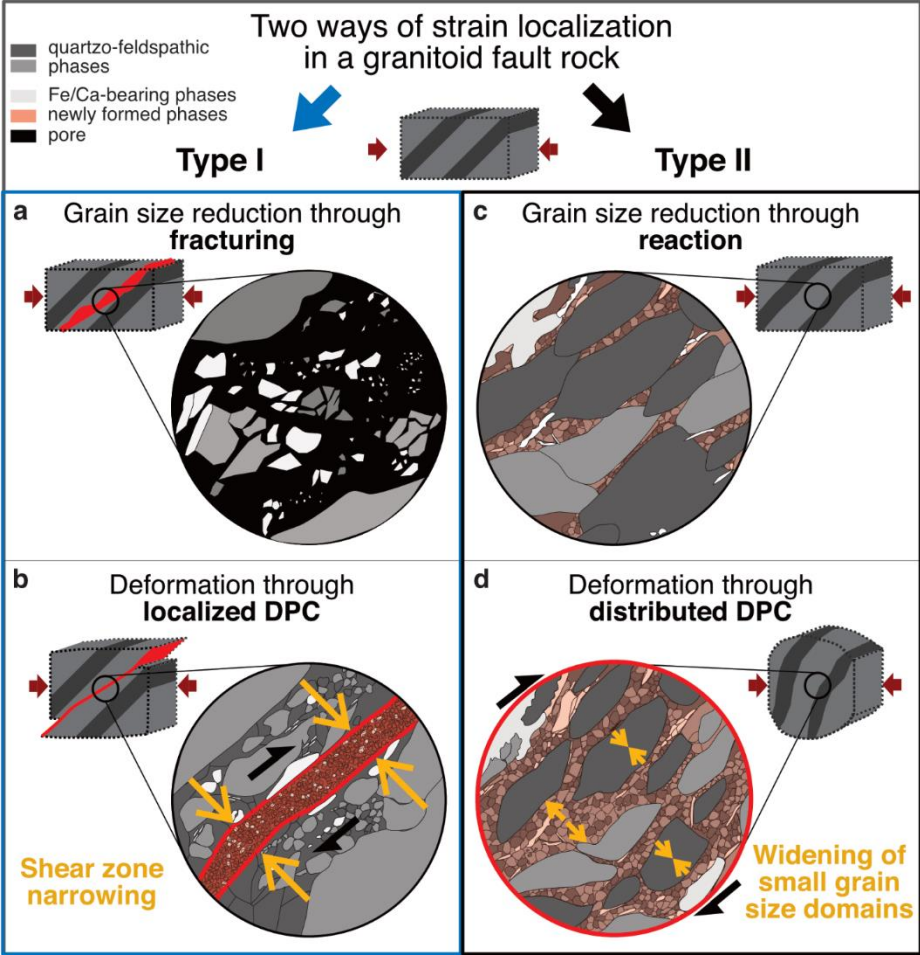


Figure 11: Summary of the different grain size reduction and strain localization mechanisms. The schematic microstructures are drawn as seen in SEM images. Independent of the initial grain size reducing mechanism, grain boundary sliding accommodated by dissolution precipitation creep (DPC) is accommodating the strain in granitoid fine-grained rocks. Pinning of grain growth leads to a stable microstructure. If a fracture is created (a), that reduces the grain size, then the deformation will localize within the fracture and deform in a very narrow, very fine-grained zone deforming by DPC (bound by red lines in b). If no fracture exists, reactions between minerals will lead to grain size reduction (c) and subsequent activation of DPC. In this case, the deformation is distributed (red circle in d).

Code availability/Data availability: All images and data are included in the article and supplementary material or referenced in the text.

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