Experimental investigation of the brittle-viscous transition in mafic rocks – interplay between fracturing, reaction, and viscous deformation

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Abstract

Rock deformation experiments are performed on fault gouge fabricated from 'Maryland Diabase' rock powder to investigate the transition from dominant brittle to dominant viscous behaviour. At the imposed strain rates of $\dot{\gamma} \sim 3\ 10^{-5} - 3\ 10^{-6}\ s^{-1}$, the transition is observed in the temperature range of (600 °C < T < 800 °C) at confining pressures of (0.5 GPa ≤ Pc ≤ 1.5 GPa). From microstructural observations, the transition is effected by a switch from brittle fracturing and cataclastic flow, to viscous dissolution-precipitation creep and grain boundary sliding. Mineral reactions and resulting grain size refinement by nucleation are observed to be critical processes for the switch to viscous deformation, i.e., grain size sensitive creep. In the transitional regime, the mechanical response of the sample is a mixed-mode between brittle and viscous rheology and microstructures associated with both brittle and viscous deformation are observed. As grain size reduction by reaction and nucleation is a time dependent process, the brittle-viscous transition is not only a function of T but to a large extent also of microstructural evolution.

1. Introduction

Our knowledge about rock strength is largely based on laboratory-derived data. Given the condition that the strength of the lithosphere cannot be greater than that of the constituting rocks and minerals, strength envelopes are constructed, which plot laboratory-derived strength or flow laws for minerals (or rocks) versus depth (as a proxy for pressure and temperature) (e.g Brace and Kohlstedt, 1980; Kohlstedt et al., 1995; Burov, 2011 and references therein). Following the general consensus that the upper crust deforms dominantly by fracturing and the lower crust and upper mantle by viscous creep, the early strength envelopes were constructed using a constant strain rate, two-mechanism model (e.g., Brace and Kohlstedt, 1980). Frictional rock strength (Byerlee, 1978) is plotted against depth to temperature and pressure conditions, where viscous deformation takes place at lower stresses than frictional sliding. Here, the term 'viscous' refers to temperature and rate sensitive deformation mechanisms such as dislocation and diffusion creep (including pressure solution creep).

Rocks deforming in the brittle field change their mode of deformation from frictional sliding along a discrete plane to distributed cracking at elevated pressures (e.g. Karman, 1911; Kirby and Kronenberg, 1984), because frictional sliding at elevated confining pressures requires higher stresses than those needed to form new cracks. This change from discrete to distributed ('ductile') deformation mode is termed "brittle-ductile transition" (BDT) and is also termed the region of semi-brittle deformation (e.g. Kohlstedt et al., 1995). Towards greater depth, the semi-brittle field is terminated by another transition, which is primarily temperature dependent. This transition is termed the "brittle-plastic transition", where the term 'plastic' denotes a permanent, non-brittle deformation without specifying a particular mechanism (Kohlstedt et al. 1995). The term "plastic" may have different meanings in terms of rheology and deformation processes, so that the for a general temperature- and rate sensitive, pressure-insensitive deformation the more general term 'brittle-viscous' transition (BVT) is preferred and will be used in this text. The introduction of a semi-brittle field to the classical strength envelope predicts a broad transitional field from dominant brittle to dominant viscous deformation.

The onset of viscous deformation (BVT, or, alternatively, an increasing α-factor for the effective pore pressure law at the BDT, according to Hirth and Beeler, 2015) within the Earth's crust is generally associated with the disappearance of earthquake rupture. Where the occurrence of the BVT is considered to be a simple function of pressure and temperature (as proxies for depth), the BVT regime may be considered as the lower depth

limit of the seismogenic zone (e.g. Sibson, 1982, 1984). However, at the BVT several processes are competing: fracturing, frictional sliding, crystal plasticity, and diffusive mass transfer. Given the number of parameters controlling the deformation, the BVT, where both, time-dependent viscous mechanisms and relatively more time-independent brittle and frictional processes significantly contribute to the deformation, is a complex system where temperature, pressure, fluid availability, grain size, strain rate, microstructure, rock composition (i.e. mineral assemblage), and chemical environment control the rheology. As a consequence, it is not surprising that there is a lack of laboratory data characterising the BVT in detail, despite pioneering seminal works by, e.g., e.g., Griggs et al. (1960); Heard (1960); Handin (1966); Byerlee (1967, 1968); Tullis and Yund (1977); Brace and Kohlstedt (1980); Carter and Tsenn (1987); and review in Paterson and Wong (2005) and references therein.

In the absence of flow laws for other crustal minerals, the viscous strength of the continental crust was first estimated using a dislocation creep flow law for quartz (Brace and Kohlstedt, 1980). However, large proportions of the lower continental crust, and most of the oceanic crust are of mafic composition and quartz is virtually absent. Strength estimates for these crustal parts could only be assessed since flow law parameters for plagioclase and pyroxene became available (e.g. Mackwell, 1991; Raterron and Jaoul, 1991; Bystricky and Mackwell, 2001; Rybacki and Dresen, 2000; Rybacki et al., 2006; Chen et al., 2006; Dimanov et al., 2003; Dimanov and Dresen, 2005; Dimanov et al., 2007). Most of the data, however, is applicable to high temperature deformation, and experimental studies for mafic rocks at lower temperatures are scarce (e.g. Kronenberg and Shelton, 1980; Shelton et al., 1981; Rutter et al., 1985; Hacker and Christire, 1991; Tullis and Yund, 1987; Getsinger and Hirth, 2014).

One important aspect of the BVT - not represented in the strength envelope plots - is the development of the rheology of sheared rock over time or during a strain history. Fault- and shear zones may show a strain-dependent microstructural evolution, which, in turn, may cause a strain-dependent strength evolution. One of the consequences of such a strain-history-dependent evolution is that fault/shear zones are unlikely to initiate with their final steady state strength. It is frequently seen from natural examples that brittle and viscous deformation can occur

cyclically (e.g. Gratier et al., 2011; Price et al., 2012) or sequentially in time (e.g. Simpson, 1986; Fitz Gerald and Stünitz, 1993; Stünitz and Fitz Gerald, 1993; Trepmann and Stöckert, 2003; Mancktelow and Pennacchioni, 2005; Pennacchioni and Mancktelow, 2007; Fusseis and Handy, 2008; Goncalves et al., 2016; Bukovská et al., 2016).

Relatively little is known about the rheology of fault rocks undergoing the BVT. As fault zones are considered to sustain the highest differential stress values near the BVT, it constitutes a key regime controlling the dynamics of lithospheric fault-zone systems. In this study, we aim to further our understanding of the evolution and the rheology of mafic rocks at conditions where the rocks undergo their BVT. Significant amounts of the global seismic moment occur along faults within the oceanic crust and along subduction zones. A better understanding of the behaviour of mafic rocks at the BVT will potentially aid our understanding of earthquake distribution and seismic hazard in these regions.

Furthermore, in order to address the question of strain dependent rheology, experiments have been performed to various amounts of strain to observe microstructural changes and evolving mechanical properties during fault-/shear-zone formation. One of the aims of this study is to gain insights into the deformation processes active over the BVT in a mafic rock and how initiation and development of fault/shear zones are achieved. Mechanical response is measured and deformation mechanisms are identified in the brittle, the semi-brittle and the viscous field.

2. Methods

2.1 Experimental procedure

2.1.1 Sample material

Experiments were performed on Maryland Diabase (Kronenberg and Shelton, 1980). The Maryland Diabase starting material (Table 1) has a modal composition (by volume) of ~ Plagioclase (Pl): 57%, Clinopyroxene (Cpx): 32%, Orthopyroxene (Opx): 8%, accessories (Qz, Kfs, Ilm, Mag, Bt, Ap): 3% (mineral abbrevations after Whitney and Evans, 2010). The Pl shows a relatively homogeneous composition of ~ An_{65-70} except for a thin rim with a lower Anorthite component of ~ An_{50-55}. The core to rim area ratio is ~ 83 : 17 (± 3). Some of the Cpx grains show a Mg-enriched

core. Pieces of Maryland diabase were crushed with a hand-press and subsequently crushed with an alumina hand-mortar. The resulting powder was dry-sieved to extract the desired grain size fraction ≤ 125 µm.

2.1.2 Experimental setup and sample assembly

Experiments were performed using two modified Griggs-type deformation apparatus at the University of Tromsø, Norway, at confining pressures (Pc) of ~ 0.5, 1.0 and 1.5 GPa, at temperatures (T) of 300, 500, 600, 700 and 800 °C and with constant displacement rates of ~ 10^{-8} to 10^{-9} m s^{-1} (resulting in strain rates of ~ $3{\cdot}10^{-5}$ to $3{\cdot}10^{-6}$ s^{-1} for homogeneous sample deformation). See Table 2 for a list of experiments and conditions.

The sample assembly is shown in Figure 1. Solid salt is used as confining medium. Inner salt pieces are fabricated from potassium iodide (KI) for experiments at T ≤ 600 °C and sodium chlorite (NaCl) for experiments at T ≥ 700 °C. KI is mechanically weaker than NaCl but shows partial melting at T >600 °C at the pressures of the experiments, which is to be avoided. Outer salt pieces are always fabricated from NaCl.

The sample consist of a thin layer of crushed rock, produced by placing 0.11g of MD powder + 0.2 µl H_2O (equals 0.18 wt.%) between the Al_2O_3 forcing blocks along a 45° pre-cut (Figure 1). Forcing blocks are cylindrical with a diameter of 6.33 mm. The sample and forcing blocks are placed in a weld-sealed platinum jacket (0.15mm wall thickness) with a 0.025 mm nickel foil insert. The amount of added water was chosen such that sufficient water is present for solution mass transport processes and mineral reactions, while not inducing mechanical pore pressure effects. No pore pressure effects in the presence of an aqueous fluid were inferred for contents up to 0.5 wt % H_2O by Kronenberg and Tullis (1984) and up to 0.3wt% H_2O by Negrini et al. (2013).

During the experiments, approximately 84% of the inelastic axial displacement is accommodated by a shear displacement along the piston-sample interface and approximately 16% by plane strain thinning of the shear zone. The initial shear zone thickness is calculated from the amount of Maryland diabase powder used, the final thickness is measured on thin sections prepared from the samples after the experiment (Table 2). The shear zone thickness is assumed to decrease linearly

throughout the experiment with increasing piston displacement.

At higher temperatures, the coupling between forcing blocks and rock material in our experiments was found to be poor. Thus, 800 °C experiments were performed using forcing blocks with 6 regularly spaced grooves, ~ 300 µm wide and 150 µm deep, cut into the surface of both, the upper and lower forcing block.

To bring a sample to the desired Pc -T conditions, the σ_1- and σ_3-pistons (Figure 1) are advanced in small increments, alternated with increments of heating, Heating is achieved using a graphite resistance furnace and T is measured with a K-type thermocouple positioned adjacent to the centre of the shear zone (Figure 1). When the desired conditions are reached (after 5 to 8 h, duration is longer for higher Pc experiments), shear zone thickness is at ~ 0.83 mm. During the experiment, only the σ_1-piston is advanced. At the end of the experiment, samples are quenched to 200 °C within 2 minutes while simultaneously retreating the σ_1-piston to remove the load on the sample. After that, samples are brought back to room conditions by slowly removing the remaining load, temperature and Pc, during ~ 3 h.

2.2 Derivation of stresses and strains

2.2.1 Data recording and processing

During the experiment, the confining pressure, axial load, and displacement are recorded using a digital data-logging system (LabView, 7.1) at a sampling frequency of 1 Hz and temperature is monitored using a proportional integral derivative (PID) controller (Eurotherm) attached to the thermocouple. Temperature is held within ± 1 °C by the controller. There is a vertical T gradient in the sample from the center outwards, ranging from ~ 17 °C/mm at 600 °C (Pec, 2014).

The principal stress σ_3 is considered to be equal to Pc (Eq. 1a), and σ_1 is derived from the measured axial force per sample area.

$$\sigma_3 = Pc \quad (1a)$$

$$\Delta\sigma = \sigma_1 - \sigma_3 \quad (1b)$$

$\Delta\sigma$ is corrected for the decreasing overlap of the forcing blocks (i.e. decreasing

sample area) using a cosine-square approximation to the ACF.

$$ac(i) = \cos^2 (90° \cdot sd(i) / L) \quad (2a)$$

$$\Delta\sigma_{AC} = ac \cdot \Delta\sigma \quad (2b)$$

where *ac(i):* relative overlap of the forcing blocks; *sd(i):* shear displacement parallel to the forcing block - sample interface; *L*: maximum possible shear displacement = diameter of forcing blocks (6.33 mm) / cos(45°) = 8.95 mm.

The shear and normal stresses, τ and σ_n, supported by the sample inclined at 45° are obtained by Mohr circle construction from $\Delta\sigma_{AC}$. The effective pore fluid pressures in our experiments is assumed to be negligible, i.e. is taken as zero. Axial displacement is corrected for apparatus stiffness. Advancing the σ_1-piston leads to an increased Pc as a function of the compressibility of the confining medium. The correction is outlined in Richter et al. (2016). Differential stress with the solid salt assemblage in the Griggs rig are usually considered to overestimate rock strength eventually up to 100 MPa (e.g. Green & Borch, 1989).

2.3 Analytical methods

2.3.1 Microscopy

After the experiments, samples are impregnated with epoxy, cut parallel to the shear direction (in some cases also normal to it), and prepared to doubly polished thin sections. Optical light microscope, scanning electron microscope (SEM) and transmission electron microscope (TEM) are used for sample analysis. SEM analyses are performed either with the Zeiss Merlin SEM at Tromsø University, or with a Philips XL30 ESEM at the centre of microscopy (SNI) at Basel University. Chemical analyses are performed using energy dispersive X-ray Spectroscopy (EDS), at 15 kV acceleration voltage and using a ZAF matrix correction.

Transmission electron microscopy (TEM) analyses are carried out at Utrecht University using a FEI Talos 200FX equipped with a high-sensitivity Super-EDX system. TEM images are recorded in bright field (BF), high angular annual dark field (HAADF) and bright field scanning TEM (BF-STEM) modes. BF images are

highly sensitive on crystallographic orientation, whereas contrasts in HAADF images are sensitive to average atomic number (Z-contrast) of the material.

Focussed ion beam (FIB) foils for TEM investigations are prepared in a FEI Helios NanoLab 3G. The FIB foil of the 600 °C experiment is cut perpendicular to both the shear direction and the shear plane. The FIB foils of the 700 and 800 °C experiments are cut parallel to the shear direction and normal to the shear plane.

2.3.2 Image analysis

In this paper, micrographs are always oriented with the shear zone boundaries horizontal and with a dextral sense of shear. The reference coordinate system is shown in Figure 2.

FFT analyses of HR-TEM images: In high-resolution (HR) TEM images where lattice planes of individual crystals are resolved, Fast Fourier Transformations (FFT) can be used to obtain the reciprocal space information akin to a direct diffraction pattern. Lattice fringes are only revealed when the crystal satisfies the diffraction condition. One single HRTEM image of a polycrystalline sample is not likely to reveal lattice fringes in all crystals. Therefore, FFT analysis used to detect diffractions on three HR-TEM images from the same area, with different tilt angles.

EDS profiles: Element concentrations along a line-profile are prepared using the software Fiji (https://fiji.sc/). A line-profile of 10 px width is defined, where the value at each point along the profile is an average over these 10 px. In this manner, the noise is reduced. The values are then normalized to the maximum count value (from the whole EDS map) of each element.

Phase Segmentation: Mineral phase segmentations are performed on BSE contrast SEM images. Phases are differentiated by their different Z-contrast using grey-level slicing. As Pyroxene (Px), Amphibole (Amph) and Zoisite (Zo) have similar Z-contrasts, manual post-processing is necessary to properly segment these phases.
Shape and orientation of shear bands: Shear bands and larger shear fractures are digitized manually on BSE contrast SEM images. The x-y coordinates of the outlines

266 are measured, smoothed (to remove digitizing artefacts) and exported using the
267 program Fiji and analysed with the SURFOR program (Panozzo Heilbronner, 1984;
268 Heilbronner and Barrett, 2014). The SURFOR results yield an orientation
269 distribution function (ODF) of boundary segments of the analysed structures and
270 is presented as a rose diagram. Shear band thicknesses were determined by
271 dividing the digitized shear bands into approximately straight segments, using Fiji
272 to extract the best fit ellipse of each segment and using the short axis of the ellipses
273 as proxy for the shear band thickness.
274 *Grain size and shape:* Grain boundaries are traced manually on SE- and BSE- SEM
275 images or on BF TEM images. The resulting grain maps are analysed with Fiji to
276 derive the grain areas and the x-y coordinates of grain boundaries. The area
277 equivalent diameters are calculated and grain size distributions are presented as
278 histograms of equivalent diameters (d_{equ}). Using a kernel density estimate
279 (MATLAB function 'ksdensity'), the mode of the distribution is determined. Grain
280 shape analyses are performed using the SURFOR program.
281
282 **3. Results**
283 Samples at T ≤ 600 °C develop similar microstructures and we therefore focus in
284 this paper on the evolution from 600 – 800 °C. Reference frames of image
285 orientation and angles are explained in Figure 2a, b; terminology used when
286 describing stress-strain curves is shown in Figure 2c. Shear strain is always given
287 as apparent shear strain γ_a (for calculation, see Appendix).
288
289 *3.1 Mechanical data*
290 *3.1.1 Stress-strain curves*
291 Shear stress vs. apparent shear strain curves are shown for T of 600 °C, 700 °C and
292 800 °C and Pc of 0.5, 1.0 and 1.5 GPa (Figure 3a). Samples at 600 and 700 °C show a
293 positive dependence of τ on Pc between 0.5 and 1.0 GPa.
294 The comparison of the stress-strain data for 700 °C experiments between 1.0 and
295 1.5 GPa Pc shows that the yield point occurs at relatively similar values. The 1.5
296 GPa Pc experiments however, unlike all other experiments, show hardening at 700
297 °C until $\gamma_a \sim 2.3$ where stresses stay at approximately constant levels thereafter.

At 800 °C, sample strength is significantly lower than in lower T experiments and within the variability between individual runs, no strength dependence on Pc is detected. 800 °C experiments show a gradual weakening after peak stress and reach a quasi-steady state at a $\gamma_a \sim 4$ onwards (Figure 3a). Note also the lower slope of the initial loading part of the stress-strain curve at 800 °C compared to lower T experiments. Samples at 800 °C deform at stresses below the Goetze criterion (the condition of $\Delta\sigma$ = Pc, which is taken as the upper $\Delta\sigma$-limit of plastic or viscous deformation; Kohlstedt, 1995. In our samples, which are oriented at 45° to σ_1, $\Delta\sigma = 2 \cdot \tau$). At lower temperatures, all sample deform at $\Delta\sigma$ clearly above the Goetze criterion.

3.1.2 Mohr Circle construction

From Mohr circle constructions (Figure 3b), the positive pressure dependence of strength at 600 °C is clearly visible. Apparent friction coefficients (μ') derived from the Mohr circles yield a value of μ' = 0.44. Experiments at 600 °C and 700 °C, 0.5 GPa Pc reach Byerlee's relationship (Byerlee, 1978) at 'yield', whereas at 1.0 GPa, the stresses at both 'yield' and flow stay below it. At Pc ≥ 1.0 GPa, and T ≥ 700 °C, shear stresses are pressure insensitive with an apparent friction coefficient of μ' = 0.05 at 700 °C. No "μ" was determined for 800 °C experiments, due to the variability in the stress values and the sensitivity of the fitted "μ" to low slopes. However, whereas 'yield' stresses do not show any significant pressure dependence, quasi-steady state values reach lower values in the lower Pc experiments at 800 °C.

3.1.3 Stress exponents

Combining constant displacement rate and displacement rate stepping tests, a stress exponent *n* is determined as

$$\tau \propto \dot{\gamma}^{1/n} \qquad (3)$$

where τ: shear stress and $\dot{\gamma}$: shear strain rate. To assess the influence of different data correction routines on calculated stress exponents *n*, some of our data is

calculated with different published correction routines, to show their influence on the determined stress exponents. The corrections compared are:

1) 'R16': Data correction described in Richter et al. (2016). The same procedure for stress calculations is followed in this paper .

2) 'R16 + H&K10': Data correction after Richter et al. (2016) with the stress correction for the solid-salt assembly after Holyoke III and Kronenberg (2010).

3) 'P12': Data correction described in Pec et al. (2012).

4) 'P12 + H&K10': Data correction after (Pec et al., 2012) with the stress correction for the solid-salt assembly after (Holyoke III and Kronenberg, 2010).

n values are determined for experiments performed at T = 300 °C, Pc = 0.5 GPa; T = 500 °C, Pc = 1.0 GPa; T = 700 °C, Pc = 1.0 GPa and T = 800 °C, Pc = 1.0 GPa (Figure 4). For T ≤ 500 °C *n* is large with values > 19, *n* for 700 °C experiments is significantly lower with a value of 5.6 and *n* at 800 °C is as low as 1.9. The *n* values determined from different data correction conventions from the literature vary by 16 – 27% (Figure 4b). For example for the T = 800 °C, Pc = 1.0 GPa experiments, a range of *n* values from 1.6 – 1.9 arises from different data treatment routines.

3.2 Microstructures

3.2.1 Overview

Strain in experiments at all Pc-T conditions localizes into a network of shear fractures and/or shear bands. The term 'shear band' is used to refer to a zone (with a certain thickness) of high strain accumulation, without any implication of a specific deformation mechanism. As opposite to a 'shear fracture', which is accommodating displacement along a plane without any obvious distribution of strain within a volume (at SEM resolution).

The microstructures developed at different temperatures are systematically different (Figure 5). At 600 °C, the microstructure is dominated by brittle deformation. Fracturing is extensive and a foliation (S), defined by elongated aggregate shapes, develops due to cataclastic flow (Figure 5a, b). Larger shear displacements are accommodated along shear fractures and shear bands, usually in Riedel shear (R_1) orientation. Grain size reduction occurs via pervasive micro-fracturing (Figure 5b). No obvious strength difference between Pl and Px is

observed, as interpreted from the similar degree of fracturing and aggregate elongation of the two phases. In rare occasions, delicate pore trails are seen where fractures are partially healed, potentially indicating limited solution mass transfer (Figure 5c).

The microstructures in 700 °C experiments are discussed for the case of Pc = 1.0 and 1.5 GPa. At 700 °C, fracturing of (especially Px-) porphyroclasts is still observed and a weak foliation (defined by elongated aggregate shapes) forms partly by cataclastic flow (Figure 5d). Strain is localized in a network of thin (~ 5 – 15 µm wide) shear bands in C' orientations, cross-cutting the foliation (Figure 5d, e). These shear bands consist of small (<< 1 µm) grains of mainly Pl, Amph and Zo (Figure 5e). Amph and Zo are not part of the starting material and represent syn-kinematic reaction products. The reaction to Amph occurs preferentially along zones of localized deformation such as fractures within Px clasts and along shear bands (Figure 6). The reaction to Zo and more albitic Pl occurs throughout the samples, but small Zo needles predominantly occur in shear bands. Both, the Amph and Zo forming reactions are observed at Pc = 1.0 and 1.5 GPa, but occur more extensively at 1.5 GPa.

At 800 °C broad shear bands form, separating low strain lenses between them (Figure 5f). Shear bands are recognized by small grain sizes and a fine-scale compositional layering defining a foliation (Figure 5g). Fracturing is only minor represented in the microstructure but can still be observed. Mineral reactions occur pervasively throughout the sample (i.e. are not restricted to high strain zones) but are more abundant in shear bands compared to low strain lenses. Amph either grows as coronas around Px grains or as aggregates within shear bands (Figure 5g) and has a composition between Mg-Hornblende to Tschermakite (Amph classification after Hawthorne et al., 2012). Zo grains occur as small needles within Pl clasts or within shear bands. Grain sizes in shear bands are usually < 1 µm. Experiments at 800 °C and 1.5 GPa Pc show very similar microstructures but the Amph and especially Zo reaction are more abundant at the higher Pc conditions.

Some melting is observed at 800 °C (melt vol.-% of total sample vol. ≤ 2), where the melt is mainly seen to form small pockets situated in extensional sites between larger porphyroclasts. No melt bands or shear-parallel melt layers are observed.

The geometry of strain localization changes over the temperature range from 600 – 800 °C (Figure 7). Shear bands and shear fractures at 600 °C tend to be few, with large displacements. At 700 °C, shear bands are more abundant, shorter and more anastomosing compared to the lower T experiments. Fractures with larger displacements are less abundant but still observed. At 800 °C, shear bands are broad and form an anastomosing network. Generally no shear fractures with any significant amount of displacement are observed at 800 °C. The preferred orientations of shear bands and shear fractures with respect to the load axis increases from 30° at 600 °C (or 15° towards the shear zone boundaries) to 42° at 800 °C (or 3° towards the shear zone boundaries; Figure 7).

3.2.2 Shear bands formed in low T experiments

Shear bands at 600 °C usually have a thickness of ~2 to 10 µm (Figure 7b) and occur both, along parts of the sample-forcing block interface and traversing the sample. The shear bands are either formed by ultra-cataclasites (type-I shear bands) or by a material that shows flow structures, seen by perturbation of a micron- to sub-micron scale compositional layering (type II shear bands) (Figure 8a). There is a clear and relatively abrupt grain size gradient over a few microns from the lower strain areas into the type-II shear bands. Whereas the material in the low strain domains is usually pervasively fractured, no fractures or grain fragments are resolved within shear bands (at SEM resolution). A type II shear band as shown in Figure 8a has been studied in more detail by TEM. The material in the lower strain domains that border the shear bands consists of larger (>>100 nm) angular Pl fragments and the boundary to the shear band material is sharp (Figure 8b). The shear band itself is composed of nano-crystalline and amorphous material in lenticular aggregates and layers, both with fairly sharp boundaries. Some crystals, identifiable by their darker appearance due to diffraction, are observed within the amorphous layers (Figure 8b - d). Lattice planes, seen in high-resolution BF HRTEM images and detected in FFT images show that the amorphously appearing layers still contain nano-crystals (Figure 8d).

3.2.3 Shear bands formed in intermediate T experiments

Figure 9 presents microstructures from shear bands developed at 700°C, 1.0GPa Pc. At these 700 °C and Pc of 1.0 and 1.5 GPa conditions, shear bands are fully crystalline and mainly composed of fine-grained Pl and Amph (Figure 9c). Pl grain sizes are on the order of ~100 – 200 nm (Figure 9b; 12). Figure 9e shows the result of TEM EDS analysis over an area containing a small Pl porphyroclast surrounded by fine-grained Pl in the shear band. Plotting the relative amounts of the elements Si, Ca and Al along a profile from the porphyroclast into the fine-grained matrix shows slightly higher Al and Ca contents and lower Si content in the porphyroclast compared to the Pl within the shear band.

3.2.4 Shear bands formed in high T experiments

Shear bands formed at 800 °C (Pc = 1.0 and 1.5 GPa) are fully crystalline. The typical microstructure shows fine-grained Pl interlayered with Amph (+Qz) (Figure 10a, c) and a close-up on the Pl grains shows 'diamond shaped', largely defect-free grains with a weak shape preferred orientation (Figure 10b; 11). The mean axial ratio of grains is b/a = 0.64 and grain sizes are on the order of ~ 0.15 – 0.50 µm (Figure 12). Pore space along grain boundaries is almost absent and grain boundaries are tight (Figure 10b; 11a). Pl grain boundaries show a strong preferred orientation in two maxima ~ 10 – 30° away from the shear plane in both directions (Figure 11). Aligned grain boundaries are frequently observed, where relatively straight grain boundaries can be traced continuous over several neighbouring grains (Figure 11c). The ODF of grain boundary segments is weakly anisotropic with a monoclinic shape, consistent with the global dextral sense of shear.

3.2.5 Grain size distribution of plagioclase in shear bands formed in 700 – 800 °C experiments

Figure 12 presents grain size distributions (GSD) measured from Pl grains within shear bands formed at 700 and 800 °C. Due to the small grain sizes in shear bands at 700 °C, it is difficult to distinguish individual grains. The best results were obtained from SE SEM images of broken surface as shown in Figure 9b. For the 800 °C experiments, grain maps were produced from both TEM and SEM images. Size distributions obtained from TEM and SEM images are similar and the GSD

presented in Figure 12b is measured on Pl grains from TEM images (similar to those shown in Figure 10). The GSD in Figure 12a is measured from grains segmented on SEM images of broken surfaces of a shear band top-view (Figure 9b), thus from a quasi-3D view, whereas the GSD in Figure 12b is from the 2D section of grains measured from a FIB foil in the TEM. Consequently, the two GSD are not fully comparable but yield a semi-quantitative measure of the grain size differences in shear bands between 700 and 800 °C experiments. The size distribution developed at 700 °C is narrow and > 80% of all grains are within 0.11 – 0.25 µm. The mode of the calculated kernel density estimate fit lies at 0.17 µm. The GSD at 800 °C is somewhat broader and > 80% of all grains are within 0.15 – 0.50 µm with a mode of the kernel density estimate calculated fit at 0.30 µm.

3.2.6 Shear band evolution with strain in 800 °C experiments

Figure 13 presents the evolution of shear bands at 800°C, as observed from experiments performed to different amounts of bulk strain, from peak stress ($\gamma_a \sim 0.7$) to a max of $\gamma_a \sim 6.8$. At peak stress, initial shear localization occurs mainly along favourably oriented grain- and phase boundaries. These initial zones are short (~ 100 – 200 µm), distributed (not interconnected) and make an angle of ~ 27° with the load axis. From the microstructure it is apparent that these structures are often dilatant: minor melt seggregations, microfracturing and nucleation of new grains/phases such as Amph and Pl are observed (Figure 14). With increasing strain, the initial shear bands start to connect by the formation of interconnected zones of fine-grained material. These zones make a smaller angle to the shear zone boundaries (or ~ 39° with respect to the load axis) and have a width of ~ 10 – 20 µm. At a shear strain of $\gamma_a \sim 4$, a network of anastomosing shear bands has formed, with ~ 3° with respect to the shear zone boundaries (i.e. 42° to the load axis). The main shear band strand has a thickness of 50 – 150 µm. This microstructure coincides with the attainment of a quasi-steady state in the mechanical data (Figure 13a, b). With increasing strain, the vol.-% of shear bands increases. Irrespective, sample strength varies only slightly between $\gamma_a \sim 4$ to 6.8 despite the significant increase in vol.-% of shear bands (Figure 13b, c).

493 *3.2.7 Shear band orientation from peak stress to higher strains*

494 Figure 15 shows the difference of shear band orientation at peak stress
495 (~ coinciding with initiation of localization) and at higher strains (at quasi-steady
496 state), as a function of temperature. Initial shear bands at all temperatures show
497 the same preferred orientation, with 27° towards the load axis. This orientation
498 stays relatively constant at ~ 30° in 600 °C experiment, but angles increase to 33°
499 at 700 °C and 42° at 800 °C.

500

501 **4. Discussion**

502 The strains attained in our experiments are insufficient to attain full
503 microstructural steady state. However, it is expected that the mechanical data
504 reaches quasi-steady state values. The inferred rheology of the samples (e.g. as
505 approximated by the determined stress exponents) always represents a bulk
506 sample rheology, caused by combined mechanisms of deformation and their
507 different rates in low strain domains and shear bands.

508

509 *4.1 Deformation mechanisms*

510 *4.1.1 low T experiments*

511 At 600 °C, displacement is mainly accommodated by cataclastic flow. The
512 mechanical data shows a clear positive dependence of sample strength on Pc
513 (Figure 3a, b). High *n* values of ~20 (Figure 4) are in accordance with dominant
514 brittle deformation and frictional sliding, as it is observed from the microstructure
515 (Figure 5b). Some viscous component of deformation may be indicated by the fact
516 that the *n* values are not as high as could be expected for purely frictional
517 behaviour.

518 Displacement in 600 °C experiments is localized in a network of shear fractures
519 and fine-grained shear bands (Figure 5a, b; 7), some of which contain amorphous
520 material (Figure 8). The angles of shear bands and shear fractures to the load axis
521 (~27° - 30°, Figure 15) are in accordance with brittle Riedel (R_1) structures.

522 The area-% of shear bands in the thin section is about 1 – 2%. As an upper-bound
523 estimate, if all displacement were to be accommodated by the shear bands, the
524 strain rate $\dot{\gamma}$ within them would be on the order of

525

$$\dot{\gamma} = 10^{-5}\ \text{mm s}^{-1} / (0.64\ \text{mm} \cdot 0.02) \approx 8 \cdot 10^{-4}\ \text{s}^{-1} \quad (3)$$

which is the applied displacement rate divided by 2% of the approximate shear zone thickness. Although the strain rate within the shear bands is likely to be high, it stays well below seismic rates.

Partly amorphous shear bands as we observe them during aseismic brittle faulting have previously been described (e.g. Yund et al., 1990; Goldsby and Tullis, 2002; Janssen et al., 2010; Pec et al., 2012, 2016). Our TEM analyses show that the shear bands consist of amorphous material, with layers and lenses of nano-crystalline material (Figure 8). A clear material difference is seen between Pl and Px, where Pl preferentially becomes amorphous and Px remains largely crystalline, with very small sizes (< 50 nm, Figure 8c). This is similar to the results of Pec et al. (2012, 2016) or Yund et al. (1990): In their granitoid sample material amorphous shear bands form extensively within the feldspatic material.

The boundaries between shear bands and host rock in our samples are sharp, even on the nano-scale (Figure 8b). It appears from the microstructure that crystalline material is comminuted to a certain grain size (< 50 nm) and below that, amorphization (mainly of the Pl) is effective. (Pec et al., 2012, 2016) discuss different possible formation mechanism for their amorphous material and conclude that the most likely mechanism is a type of mechanical amorphization, i.e. high defect densities until crystallinity is lost. Feldspars appear to be particularly susceptible to this process, as also supported by this study. The rheology of these (partly-)amorphous shear bands, however remains yet unclear.

4.1.2 High T experiments

At 800 °C, there is a significant difference in rheology compared to lower T experiments: differential stresses at 800 °C are half as high as at 700 °C and stay always below the Goetze criterion (Table 2; Figure 3a). Initial stress increase during sample loading (before peak stress) shows lower slopes at 800 °C (Figure 3a), indicating a larger component of viscous deformation early in the experiment. The microstructure at 800 °C is dominated by mineral reactions and strong grain size refinement Figure 5f, g; 10). The grain size within shear bands ranges mainly between 0.15 – 0.50 µm for Pl (Figure 12b), with similar sizes for Amph and Zo.

559 Grain size reduction takes place mainly by nucleation of new grains in conjunction
560 with mineral reactions and potentially aided by strain energy reduction by
561 replacing old, defect-rich porphyroclasts by new defect-free grains. The small grain
562 sizes facilitate a grain size sensitive creep mechanism, where the strain rate is
563 proportional to d^{-m} (e.g. Ashby and Verrall, 1973; Coble, 1963; Rutter, 1976), where
564 d is the grain size and m the grain size exponent. Diffusion creep and grain
565 boundary sliding (GBS) are known to only leave few microstructural traces of their
566 activity. However, aligned grain boundaries, equant to weakly anisotropic grain
567 shapes, and low internal defect densities of grains are microstructures
568 characteristic for GBS and diffusion creep (e.g. Elliott, 1973; Boullier and Gueguen,
569 1975; Gifkins, 1976; Drury and Humphreys, 1988; Kilian et al., 2011; Drury et al.,
570 2011). The shear bands consist of small, elongated hexagonal grains, whose grain
571 boundaries can be contiguous over several grain diameters (Figure 11). The
572 contiguous grain boundaries are well orientated for sliding in all orientations. As
573 the experiments are performed with H_2O present, dissolution-precipitation creep
574 (DPC) is interpreted to be the dominant form of diffusion creep.
575 DPC needs to be accommodated by some GBS (Lifshitz sliding of Langdon 2006),
576 but the main strain contribution in very small sized aggregates may take place by
577 GBS, where diffusive mass transport accommodates shape changes (Rachinger
578 sliding of Langdon 2006). As both processes are closely linked and interconnected,
579 the term DPC includes both, diffusion creep and GBS.
580 DPC as a dominant deformation mechanism is in accordance with the low observed
581 stress exponents of $n \sim 1.9$. Usually, stress exponents for diffusion creep are
582 expected to be close or equal to 1 (e.g. Ashby and Verrall, 1973; Coble, 1963;
583 Karato, 2008, Kohlstedt and Hansen, 2015; Paterson, 2013), but higher n values
584 have been suggested for DPC, depending on the driving potentials or chemical
585 potential gradients along the grain contact area (e.g., Gratier et al. 2009, 2013). In
586 low strain domains grain sizes are large and DPC will not be an efficient
587 deformation mechanism in these. Frictional sliding may be active in the low strain
588 domains (but not dominant), increasing the stress exponent of the bulk sample to n
589 = 1.9. Diffusion creep as viscous deformation mechanism has previously been
590 suggested for experimentally deformed basaltic material (under water added
591 conditions) by e.g. Rutter et al. (1985); Getsinger and Hirth (2014), whereas Rutter

et al. (1985) state more in detail, that they interpret DPC together with GBS to be the dominant deformation mechanism.

4.1.3 Intermediate temperature experiments

The microstructure at 700 °C shows characteristics of both DPC (including GBS) and cataclastic flow. Cataclastic flow thereby is mainly observed to contribute to deformation in low strain lenses (Figure 5d; 6). In shear bands, the similar microstructures as in shear bands at 800 °C suggest that DPC is the dominant deformation mechanisms in shear bands at 700 °C too. The main difference between 800 and 700 °C is the somewhat smaller grain sizes in shear bands at 700 °C (Figure 12).

Fracturing at 700 °C is subordinate within shear bands. Fracturing and cataclasis are expected to produce a wide range of grain sizes with angular grain shapes (e.g. Stel, 1981; Storti et al., 2003; Keulen et al., 2007), unlike the observed microstructure. TEM-EDS mapping also reveals a compositional difference between Pl porphyroclast and fine-grained shear band Pl (Figure 9), which is further evidence that the plagioclase grains within the shear bands are not a result of fracturing but rather result from neocrystallization. The chemical differences are small but the coupled Al+Ca decrease with a Si increase from the clast to the shear band is consistent with a change towards lower anorthite content in the matrix Pl of the shear band. The measured change in Pl chemistry between porphyroclasts and fine-grained shear band Pl also excludes subgrain rotation recrystallization and instead points to nucleation as means of grain size reduction.

However, a brittle precursor to the shear bands at 700°C is possible, interpreted from the initiation of shear bands with the same low angle towards the load axis as in the brittle dominated 600°C experiments (Figure 15), as well as by the similar sample strengths for 600 and 700°C experiments. Due to the low strains at shear band initiation (~ at peak stress) it is, however, difficult to identify from the microstructure if and in which proportions viscous or brittle processes contribute at the point of initiating strain localization.

At 700 °C, sample strength as seen from the stress-strain curves is comparable to the 600 °C experiments (Figure 3a) with a significantly lower stress sensitivity on strain rate ($1/n$) (n = 5.6 at 700 °C, n > 19 at T < 600 °C; Figure 4a). The stress

625 exponent of 5.6 is just slightly higher than what would be typical for dislocation
626 creep (n = 3 to 5; Karato, 2008; Paterson, 2013; Kohlstedt and Hansen, 2015), but
627 none of the microstructures indicate evidence for crystal plasticity. Rather, the
628 intermediate n-value is interpreted to result from a combination of predominantly
629 brittle (n-values of 19.5 and higher) and viscous processes (n~1.9), as it is
630 observed in the microstructure.
631
632 *4.1.4 Summary of deformation mechanisms*
633 The dominance of viscous deformation at 800 °C, and of brittle deformation at 600
634 °C is evident, both from the mechanical data and the microstructure. Samples
635 deformed at 700 °C are an intermediate case, where strain is localized into shear
636 bands which are interpreted to deform with a viscous deformation mechanism but
637 with stress-strain curves and strengths more like the brittle-dominated 600 °C
638 samples (Figure 3a).
639 Shear bands formed in 700 °C experiments, like the shear bands at 800 °C, are
640 interpreted to accommodate strain mainly by DPC and GBS. However, low strain
641 lenses at 700 °C show abundant microfracturing and a contribution of cataclastic
642 flow to deformation. The stress exponent is considerably lower at 700 °C than for
643 lower T experiments (Figure 4a), indicating an increased viscous component to the
644 rheology at 700 °C. The stress exponent of n = 5.6 for 700 °C experiments is
645 interpreted as a mixed mechanical response determined by the rheology of
646 viscously deforming shear bands and partly frictional/cataclastic low strain lenses.
647 The transition from dominantly brittle deformation to dominantly viscous flow in
648 our experiments is seen to initiate with the dominance of solution-mass transport.
649 Mineral reactions and nucleation lead to grain size reduction and thus strongly
650 enhance the strain rate of grain size sensitive creep mechanisms. For our imposed
651 experimental displacement rates, the transition occurs around 700 °C (although
652 not fully Pc insensitive). In deformation experiments on whole-rock cores of
653 Maryland Diabase at conditions similar to ours, Kronenberg and Shelton (1980)
654 observed a brittle-viscous transition in their samples around 700 °C for a Pc = 1.0
655 GPa, comparable to our observations. However their strain rate was approximately
656 one order of magnitude lower.

In our experiments, the influence of Pc is less pronounced compared to that of the temperature. Increasing the Pc at, e.g., 600 °C does not lead to a transition to more viscous behaviour. At 700 and 800 °C, the main effect of increasing the Pc is the formation of a higher abundance of reaction products. This indicates a rate-enhancing effect of increasing Pc on solution-mass transport processes and/or reaction kinetics, e.g., by a greater overstepping of reaction boundaries for pressure sensitive reactions.

4.2 Microstructural evolution and its influence on bulk rheology

4.2.1 Shear band evolution

As seen from 800 °C experiments, shear bands are widening and increase in vol.-% with increasing strain (Figure 13), caused by the on-going process of mineral reactions and nucleation, leading to the replacement of old, coarser-grained porphyroclasts by new, sub-micron sized grains. Shear band vol.-% stays relatively low in 700 °C experiments for the strains achieved, and interconnectivity is much lower than at 800°C (e.g. Figure 7). From the mechanical data it is seen that 700 °C experiments still show a large influence of brittle deformation, which is attributed to the low volume percentage, unfavourable orientation (not parallel to shear zone boundary but inclined against it with ~ 7°), and poor interconnectivity of shear bands. With increasing shear band widening, the viscous rheology of the shear bands at 700 °C is expected to eventually become more dominant.

As reaction and diffusion rates are lower at lower T, more time (or equivalently strain) is needed in the case of the 700 °C experiment to attain a connected network of shear bands as it is observed at 800 °C. However, the positive feedback between fracturing and reaction kinetics (as seen from extensive mineral reactions along microfractures in 700 °C experiments, Figure 6) aids the microstructural change, i.e. grain size reduction by reaction and nucleation, and appears to be an important mechanism in switching from dominant brittle to more viscous rheology.

Comparing the orientations of shear bands formed at different T (Figure 7) it is apparent that the orientations are less favourable for bulk shear displacement in 600 and 700 °C compared to 800 °C experiments, due to the higher inclination of shear bands to the shear zone boundaries. Thus, shear band orientation is an

additional factor determining how the weak phase controls the rheology (cf. Gerbi et al., 2016) .

4.2.2. Shear band influence on bulk rheology in high-T experiments – estimating flow stresses in shear bands from plagioclase diffusion creep flow law

The shear band evolution in 800 °C experiments shows an increasing dominance in shear band orientations (sub-)parallel to the shear zone boundaries (Figure 13) with increasing shear band widening and interconnection. That is, the shear band network evolves into geometrically more favourable orientations. Sample strengths, however, remain relatively high, with shear stress values of $\tau > 190$MPa (Table 2; Figure 3a). Additionally, a quasi-steady state in the stress-strain curves at $\gamma_a \gtrsim 4$ is reached, disregarding a still increasing shear band vol.-%. These observations again suggest that the bulk sample rheology is not simply determined by the rheology of the shear bands.

For our experimental samples, the imposed displacement rate, temperature and the grain size within shear bands are known. At 800°C, where dissolution precipitation creep is interpreted to dominate the deformation, we can attempt to calculate expected stresses within shear bands for the given conditions. At present, however, the lack of, e.g., mineral solubility data and properties of grain boundary fluid films does not allow to calculate strain rates from common dissolution precipitation creep flow laws at the elevated Pc/T conditions of our experiments. We use a simplified approach to estimate the shear band rheology by applying the flow law of Rybacki and Dresen (2000) (shortened as 'RD00') for diffusion creep in feldspars, as has been done previously by e.g. Getsinger and Hirth (2014). The flow law of RD00 has the form:

$$\dot{\varepsilon} = A \cdot \Delta\sigma^{n} \cdot d^{-m} \cdot \exp\left(-\frac{Q}{RT}\right) \qquad (5)$$

where A : constant, $\Delta\sigma$: differential stress, n : stress exponent (usually ~ 1), d : grain size, m : grain size exponent, Q : activation energy, R : universal gas constant, T : temperature.

Under the assumption that the shear bands accommodate the majority of the deformation, a shear strain rate of ~ 1 x 10^{-4} s^{-1} is assumed for them. This is derived from the imposed displacement rate, ~ 1 x 10^{-5} mm s^{-1} divided by the cumulative shear band thickness, ~ 8 x 10^{-2} mm (which is 10 – 20% of the total sample thickness at quasi-steady state in the mechanical data, e.g. Figure 13). Using the RD00 flow law for diffusion creep in a wet plagioclase aggregate, we use a strain rate of 1 x 10^{-4} s^{-1}, a grain size range of 0.15 – 0.50 µm and T = 800 °C. To compare our shear strain rates to the axial shortening strain rates of the flow law, the conversion from axial shortening to simple shear strain rates after Schmid et al. (1987) is used, reformulating Eq. (5) to :

$$\dot{\gamma} = A \cdot \sqrt{3}^{(n+1)} \cdot \tau^n \cdot d^{-m} \cdot \exp\left(-\frac{Q}{RT}\right) \qquad (6)$$

$\dot{\gamma}$: shear strain rate, *A*: constant, *n*: stress exponent = 1, τ : shear stress, *d*: grain size, *m*: grain size exponent = 3, *Q*: activation energy, *R*: universal gas constant, *T*: temperature.

Solving Eq. (6) for shear stresses, they are calculated as:

$$\tau = \exp\left(log\left(\frac{\dot{\gamma}}{A \cdot \sqrt{3}^{(n+1)} \cdot d^{-m}}\right) - \left(\frac{-Q}{R \cdot T}\right)\right) \qquad (7)$$

Resulting shear stresses are between 0.4 – 16 MPa. That is one to almost three orders of magnitude lower than measured in the mechanical data. Conversely, if Eq. (6) were used to calculate the strain rate for the given T and d, with the measured τ = 200 MPa, strain rates of 1 x 10^{-3} to 5 x 10^{-2} s^{-1} would result.
Our experimental samples contain higher wt.-% H_20 compared to the samples of Rybacki and Dresen (2000), which is likely to have a marked effect on the rate of DPC. Nonetheless, despite some uncertainties in the application of the RD00 flow law, the results are expected to yield values within the expected order of magnitude for DPC. The calculated stresses for the shear bands thus suggest that the elevated bulk sample strengths of $\tau \approx 200$ MPa at the given bulk strain rate of 10^{-5} s^{-1} cannot be explained by representing the fine-grained material within shear bands. Rather, an effect by a load-bearing framework of low strain lenses due to

752 insufficient connectivity and unfavourable orientation of shear bands is suggested
753 to explain the observed bulk sample strengths. Bulk sample strength is interpreted
754 to be determined by the combined rate of DPC and GBS in the coarse-grained low
755 strain lenses and the fine-grained shear bands. The onset of DPC does not depend
756 on the occurrence of shear bands but because diffusion lengths are proportional to
757 the grain size, the rate of strain accommodation by DPC will vary strongly between
758 fine grained shear bands compared to low strain lenses which largely preserve the
759 coarse initial grain sizes. Delocalized viscous processes prior to shear band
760 formation are also indicated by the lower initial slope of the loading curve in the
761 mechanical data (Figure 3a), indicating a more viscous component of sample
762 deformation from the start of the experiment . DPC is speculated to cause this
763 viscous relaxation.
764
765 *4.2.3 The influence of strain on reaction rate as seen from the microstructure*
766 At 700 °C, fractures in Px porphyroclasts are extensively decorated by Amph
767 overgrowths (Figure 6), where Amph is forming by the reaction:
768
769 $$Px + Pl + H_2O \rightarrow Amph + Qz \qquad (8)$$
770
771 Amph is seen to grow along Px-internal fractures with no contacting boundary
772 towards Pl, indicating that element transport along the fractures occurred over
773 several µm distance. It is described in the literature that fracturing can lead to high
774 dislocation densities in the host crystal and is frequently associated with porosity
775 (e.g. Fitz Gerald et al., 1991; Fitz Gerald and Stünitz, 1993; de Ronde et al., 2005).
776 Fitz Gerald and Stünitz (1993) interpret from their observations that permeability
777 along the microfractures must have been greatly enhanced, allowing for solution
778 mass transport and mineral reactions along the fracture. Also from our
779 observations, there is a clear positive feedback between deformation and reaction.
780 This seems especially important in the 700 °C experiments, where the positive
781 contribution of deformation on reaction rate is seen more strongly in the
782 microstructure compared to 800 °C experiments. This is not unexpected, as
783 reaction and diffusion rates will increase with increasing temperature and the

784 rate-enhancing effects of deformation on reaction and diffusion may become less
785 important.
786
787 *4.3 Linkage to natural faults*
788 The findings from our experiments suggest that a transition from brittle to viscous
789 deformation in the studied mafic rock type initiates where solution-mass transport
790 processes occur at sufficiently high rates to accommodate deformation at the
791 imposed strain rate. No significant contribution of crystal plastic processes could
792 be observed. DPC and mineral reactions both take place simultaneously. The grain
793 size reduction is caused primarily by mineral reaction and nucleation of new
794 grains. The operation of DPC as a mechanism observed in our experiments
795 depends on the metastability of minerals in the starting material. This situation is
796 typical for most basalts and gabbros at amphibolite facies conditions and lower
797 temperatures.
798 For the continental crust, with its lower geothermal heat gradient compared to the
799 oceanic curst, mafic fault rocks at common strain rates ($< 10^{-9}$ s^{-1}) in the presence
800 of fluids are likely to go through their brittle-viscous transition at much lower
801 temperatures than observed in our experiments. Hydration reactions of feldspars
802 at e.g. greenschist facies conditions can lead to grain size reduction and phase
803 mixing, promoting viscous deformation by grain size sensitive creep mechanisms
804 (e.g. Fitz Gerald and Stünitz, 1993; Stünitz and Fitz Gerald, 1993).
805 In the oceanic crust, where confining pressures (i.e. lithostatic pressures) are much
806 lower for same temperatures compared to the continental crust, brittle-viscous
807 transitional behaviour is likely to occur at higher temperatures, i.e. similar
808 temperatures like in our experiments (e.g., Mehl and Hirth 2008). As pressures
809 generally are lower, fracturing is will be more common and is likely contributing to
810 deformation even to high temperatures.
811 As seen from our experiments, brittle-viscous transitional behaviour is not only a
812 function of the externally applied parameters such as P and T, but especially a
813 function of strain, i.e. microstructural evolution. This is an important factor to
814 consider, as typically rheology of rocks is modelled as a material property without
815 considering an evolution. We consider that this strain dependent rheology, as
816 observed in our experiments, can be extrapolated to natural fault systems. Where

817 the strain dependency of rheology bears the potential to lead to a 'time-dependent'
818 brittle-viscous transition as microstructure evolves towards favouring viscous
819 deformation (as seen for the 700 °C experiments in our study).
820
821 **5. Summary and conclusions**
822 At the imposed experimental displacement rates, a transition from dominantly
823 brittle to brittle-viscous to dominantly viscous is observed between the
824 temperatures 600, 700 and 800 °C. The brittle-viscous transition in our study is
825 observed to occur via a switch from fracturing to diffusion creep (in the sense of
826 DPC) and grain boundary sliding as dominant viscous deformation mechanisms.
827 Viscous deformation in our experiments starts with the onset of diffusive mass
828 transport and starts to dominate the rheology when fine-grained, interconnected
829 zones have formed.
830 The important processes enabling viscous rheology are 1) efficient solution-mass
831 transport, 2) grain size refinement, which in our case as a result of reaction and
832 nucleation, and 3) shear band interconnection.
833 We observe a brittle-viscous transition not only as a result of increasing
834 temperatures but also via a microstructural evolution. Especially as the brittle-
835 viscous transition is approached in terms of temperature (in our case at 700 °C),
836 more evolved microstructure in shear bands allows for viscous deformation,
837 whereas low strain lenses still show largely brittle deformation. This leads to a
838 likely transient, mixed mechanical response of brittle and viscous rheology. For
839 constant syn-kinematic conditions (i.e. strain rate, P and T, fluid availability, etc.)
840 the syn-kinematic microstructural evolution with strain (and time) is expected to
841 change the relative importance of brittle and viscous deformation in favour of the
842 viscous processes.
843
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Figure Captions

Figure 1 : Sample assembly. a) Schematic cross-secion of sample assembly. Inset shows details on sample. b) Thin section of sample 449 after deformation. Black arrow indicates unloading crack. FB = forcing block, SZ = shear zone.

Figure 2 : Reference frame and definitions. a) Micrographs are oriented with the shear zone boundaries parallel to the x-direction with a dextral sense of shear. b) In rose diagrams, preferred orientations are marked with black dots; θ = angle between orientation and applied load (σ_1 direction); Φ = angle between orientation and shear plane (shear zone boundaries). c) Stresses are plotted as shear stress τ versus apparent shear strain γ_a, see Appendix. Stages of the experiment are indicated.

Figure 3 : Mechanical data. a) Shear stress vs. apparent shear strain for experiments performed at T = 600, 700 and 800 °C, using confining pressures (Pc) of ~ 0.5, 1.0 and 1.5 GPa. Shear strain rate for all experiments is ~ 3 x 10^{-5} s^{-1}. b) Mohr diagrams for same experiments. σ_3 = confining pressure; σ_1 = yield stress. (black lines) or quasi-steady state (grey dotted lines). In red, apparent friction coefficient μ' and angle of internal friction. μ' values decrease with increasing T and increasing Pc. Stress values usually stay below Byerlees relationship except for 600 and 700 °C experiments at Pc = 0.5 GPa.

Figure 4 : Derivation of stress exponents. a) Shear stress vs. apparent shear strain rate, with calculated stress exponent n; slope of linear fit is 1/n. b) Different stress exponents calculated for different data-correction routines shown for two experiments performed at T = 800 °C / Pc = 1.0 GPa and T = 300 °C / Pc = 0.5GPa. R16 = after Richter et al., 2016; H&K10 = after Holyoke & Kronenberg, 2010; P12= after Pec et al, 2012 (see Methods).

Figure 5 : Microstructure development across the brittle-viscous transition. Experimental conditions are indicated, dextral shear sense applies to all. a) and b) At T = 600 °C, fracturing is extensive; a foliation (S) is developed by cataclastic

flow; shear displacement is accommodated along shear bands and shear fractures in Riedel shear (R) orientations. c) Pore trails along fractures indicate partial healing. d) and e) At T=700 °C, fracturing is extensive in Px porphyroclasts; a foliation (S) is developed partly by cataclastic flow. e) Shear bands are recognized by a fine-scale compositional layering and intense grain size reduction; hydrous reaction products Amph and Zo are beginning to form. f) and g) At T = 800 °C, broad shear bands (white stippled lines) anastomose around low strain lenses; they are characterized by grain size reduction and the formation of a foliation parallel to the shear band boundaries; hydrous reactions products Amph and (to a lower extent) Zo are formed.

Figure 6 : Hydrous reactions at 700 °C. Left: digitally produced phase maps, right, BSE image of the same area. Dark grey = Pl; orange = Amph; bright grey = Px ; sample deformed at T = 700 °C, Pc = 1.0 GPa. Amph follows zones of high strain such as shear bands, or fractures within Px clasts.

Figure 7 : Shear band morphology as a function of temperature. Shear bands and shear fractures are traced in red. Light background layer are BSE contrast images of the shear zones. Rose diagrams (surface ODFs) show orientation of boundary segments of the traced structures. Horizontal shear bands developed at the forcing block-shear zone interface are omitted from the analysis. Rose diagrams show the dominance of more shear zone parallel shear bands at higher experimental T. b) Shear band width distributions. Shear bands stay largely below 10 µm at 600 °C, and get broader at higher T, reaching up to 70 µm for the presented 800 °C experiment.

Figure 8 : Micro- to nanostructures of shear bands developed at 600 °C. a) SEM BSE image of a shear band formed in the sample near interface with the forcing block; shear sense is dextral. b) - d) TEM images of a shear band similar to the one shown in a). Kinematic reference frame for images b) - d) is given in upper right corner in b). b) BF TEM image. Increasing grain size refinement from top to lower half of the image; cryst = crystalline; amor = amorphous. Note few remaining crystals (darker) within amorphous layers. c) HAADF (left) and BF

TEM image (right) of nano-crystals mainly formed by Px; amorphous layers correlate with the typical darker grey-value of Pl. d) High-resolution BF image from a central part of the shear band; nano-crystalline layer enclosed between amorphous domains. White squares denote areas where diffraction spots are detected in FFT analyses (see Methods); locally, amorphous layers contain nano-crystals, identified both from diffraction spots in FFT images and from their dark appearance in the BF image.

Figure 9 : Micro- to nanostructures of shear bands developed at 700 °C. a) SEM BSE image of a shear band. b) SE SEM image of the surface of a shear band; mostly Pl grains are visible; white arrow indicates shear direction, with top to the upper right. c) HAADF image showing a Px porphyroclast adjacent to a fine grained shear band formed by Pl + Amph; the Px porphyroclast shows a thin reaction corona of Amph; black arrow points to porosity within the Px clast. d) HAADF image of a Pl porphyroclast with surrounding fine-grained Pl in a shear band; the trace of an EDS profile is marked. Kinematic reference frame is the same as in c). e) Element counts of Si, Ca and Al (normalized to max. count value of the respective element) versus distance (pixel) along the profile marked in d); mean values (blue) are indicated for the porphyroclast and the Pl of the shear band.

Figure 10 : Micro- to nanostructures of shear bands developed at 800 °C. a) SE BSE image of a shear band with typical compositional layering of Pl dominated layers alternating with Amph+Qz(+Pl) mixed layers. b) BF TEM image of Pl grains within a shear band; grains show a low defect density; porosity is low and grain boundaries are tight. c) HAADF image showing the typical compositional layering of Pl dominated layers alternating with Amph+Qz aggregates. d) BF-STEM image of the same area as in c).

Figure 11 : Shape of plagioclase grains in shear bands at 800 ° C. a) BF TEM image of diamond-shaped Pl grains with a weak shape preferred orientation. b) Rose diagram (surface ODF) of Pl grain boundaries preferentially oriented at Φ = 10-30° . The ODF shows a weak anisotropy consistent with the dextral sense

of shear. c) Grain boundary segments are visualized separately for horizontal (-30° < Φ < 30°) and vertical (30° < Φ < 150°) orientations (marked black in rose diagrams).

Figure 12 : Grain size distribution of plagioclase in shear bands. a) Sample 416 deformed at T = 700 °C, Pc = 1.0 GPa; area equivalent grain diameters, d_{equ}, were determined from SE images as shown in Fig. 9b. b) Sample 414 deformed at T = 800°C, Pc = 1.0 GPa; area equivalent grain diameters, dequ, were determined from TEM images of the FIB-foil shown in Fig. 10. Dark grey bars represent >80% of all grains; black line = ernel density estimate fit, number of grains, n, and mode of curve fit are indicated.

Figure 13 : Evolution of shear bands with increasing strain. a) Central parts of shear zones deformed at T = 800 °C and Pc = 1.0 GPa to increasing apparent shear strains; sample 449 (Pc = 1.5 GPa) is included for comparison. Shear bands are shown in orange; rose diagrams with surface ODF of shear band boundary segments on right. With increasing strain, dominant shear band orientation becomes more shear zone parallel and shear bands become wider and better interconnected. b) Stress - strain curves for samples shown in a). c) Increase of area fraction of shear bands with increasing shear strain. Although geometry of shear bands at 1.0 and 1.5 GPa differ, their area-% is identical..

Figure 14 : Zones of initial shear localization. BSE SEM image of sample 460 deformed at T = 800 °C, Pc = 1.0 GPa. Black arrows point to minute segregation of melt; nucleation of new Amph (white arrow) and Pl grains is observed.

Figure 15 : Comparison of shear band orientation formed at peak stress (left) and at higher strains at quasi steady state. Rose diagrams are surface ODFs of shear zone boundary segments. Initiation of shear band shows comparable preferred angles for all temperatures. At higher temperatures however, shear band orinetations are more shear zone parallel with increasing strain, i.e. microstructural evolution.

Table 1 : Composition of Maryland Diabase starting material**.** EDS measurements as oxyide wt.-% and calculated to stoichiometric mineral formula.

Table 2 : List of experiments and experimental conditions.

T = Temperature

Pc = Confining pressure, averaged between γ_a = 1.5 to end of experiment; (*) denotes peak stress experiments where average Pc is calculated between beginning and end of experiment.

Peak τ = Maximum shear stress.

Flow τ = Shear stress during quasi-steady state or at end of experiment.

γ_a = Total apparent shear strain.

$\dot{\gamma}_a$ = Apparent shear strain rate: (1) constant displacement rate experiments. $\dot{\gamma}_a$ calculated as the average value between γ_a = 1.5 and end of experiment; (2) displacement rate stepping tests. $\dot{\gamma}_a$ as average value for each setting.

Apendix Figure 1 : General shear setup within initial state and shear zone thickness, th0, and state at end of experiment and final shear zone thickness, thF. For representation purposes, the displacement is displayed to occur asymmetric by shift of only the upper forcing block.

Appendix Table 1 : Different measures for shear strain.

th0 = estimated shear zone thickness at start of experiment (Appendix Figure 2).

thF = measured shear zone thickness at end of experiment (Appendix Figure 1).

d = axial displacement of σ 1-piston.

sdF = displacement of σ 1-piston parallel to shear zone boundary (Appendix Figure 1).

k = th0/thF = pure shear component.

thF/th0 (%) = relative thickness of sample after deformation.

Γ_{eff} = effective shear strain (Appendix Eq. (A4)).

γ_{TH} = shear strain determined from relative displacement of forcing blocks as measured on thin sections.

γ_{comp} = simple shear component (Appendix Eq. (A5)).

γ_a = apparent shear strain (Appendix Eq. (A2)).

R_f = aspect ratio of finite strain ellipse (after Fossen & Tikoff, 1973).

ϕ = orientation of finite stretching direction (after Fossen and Tikoff, 1973).

ε_m = strain magnitude (Appendix Eq. (A6)).

Appendix

A1. Derivation of shear strains

General shear experiments like the ones described in this paper often experience sample thinning, even if the samples are pre-compacted or annealed before the actual start of the shear deformation. Thinning continues throughout the experiments, as inferred from samples deformed under identical conditions to different total strains. It is usually observed that the sample material does not escape sideways. The strain is therefore taken to be plane strain and calculations are made in 2 dimensions by assuming a combination of pure shear (thinning of shear zone) and simple shear (displacement parallel to the piston-sample interface).

Measurements indicate that the samples thin linearly from the beginning of the experiment to the end. Correlating the shear zone thickness, *thF*, with the axial displacement, *d*, a linear trend of shear zone thinning is evident (Appendix Figure 1). The initial thickness, i.e., the thickness of the compacted samples at the start of the experiment, depends on temperature. Two initial thicknesses for T = 800°C and T ≤ 700°C are derived (Appendix Table 1).

For strain calculations, the axial displacement, *d*, of the σ_1-piston is partitioned into a shear component parallel to the 45° sample piston interface, and a thinning component normal to the shear zone boundary. Dividing the total shear displacement *sdF*, by the final thickness *thF*, γ_{TH} is derived (Appendix Figure 2).

$$\gamma_{TH} = sdF / thF \quad \text{(A1a)}$$

$$sdF = (d - (th0-thF)\cdot\cos(45°)) / \cos(45°) \quad \text{(A1b)}$$

where: *sdF*: total displacement parallel to the piston-sample interface; *th0 / thF* : initial / final thickness of sample.

In this study, the shear strain is calculated from the experimental record as the sum of individual increments of shear displacement divided by the instantaneous shear zone thickness. The shear strain derived in this manner is referred to as apparent shear strain γ_a :

$$\gamma_a(\mathrm{k}) = \sum_{i=2}^{k} \frac{\mathrm{sd(i)} - \mathrm{sd(i-1)}}{\mathrm{th(i)}} \qquad (A2)$$

sd(i) : displacement along piston-sample interface; *th(i) :* shear zone thickness at time *i*. The shear strain values calculated in this fashion depend on the thinning of the sample. γ_a is termed 'apparent', as it is not a real measure of simple shear. The pure shear component of the sample strain is calculated as

$$\mathrm{k} = \mathrm{th0} / \mathrm{thF} \qquad (A3)$$

Following Fossen and Tikoff (1993), the effective shear strain, Γ_{eff}, and the simple shear strain component, γ_{comp}, are derived as

$$\Gamma_{\mathrm{eff}} = \mathrm{k}^{-1} \cdot \tan(\psi) = \mathrm{sdF} / \mathrm{th0} \qquad (A4)$$

$$\gamma_{\mathrm{comp}} = \Gamma_{\mathrm{eff}} \cdot \frac{2 \ln(k)}{k - k^{-1}} \qquad (A5)$$

The difference between the strain measures is small as shown in Appendix Figure 3. Note that, if thF = th0, i.e., if k = 1, all strain measures coincide: $\gamma_{\mathrm{TH}} = \gamma_{\mathrm{a}} = \Gamma = \gamma_{\mathrm{comp}}$. In order to be able to convert from axial to shearing experiments, the strain magnitude is derived after Schmid et al. (1987):

$$\varepsilon_m = \frac{1}{\sqrt{3}}[(\varepsilon_1 - \varepsilon_2)^2 + (\varepsilon_2 - \varepsilon_3)^2 + (\varepsilon_3 - \varepsilon_1)^2]^{1/2} \qquad (A6)$$

where ε_m = strain magnitude; ε_1, ε_2, ε_3 = axes of the strain ellipsoid calculated after Fossen and Tikoff (1993). Plane strain conditions are assumed with $\varepsilon_2 = 1$.