Syn-kinematic hydration reactions, grain size reduction, and dissolution-precipitation creep in experimentally deformed plagioclase - pyroxene mixtures

Sina Marti[1], Holger Stünitz[2,3], Renée Heilbronner[1], Oliver Plümper[4], and Rüdiger Kilian[1]

[1]Department of Environmental Sciences, Basel University, Switzerland
[2]Department of Geosciences, University of Tromsø, Norway
[3]Institut des Sciences de la Terre d'Orléans (ISTO), Université d'Orléans, France
[4]Department of Earth Sciences, Utrecht University, The Netherlands

Correspondence: Sina Marti (sina.marti@ed.ac.uk)

Abstract. It is widely observed that mafic rocks are able to accommodate high strains by viscous flow. Yet, a number of questions concerning the exact nature of the involved deformation mechanisms continue to be debated. In this contribution, rock deformation experiments on four different, water-added plagioclase-pyroxene mixtures are presented: (i) plagioclase(An60-70) – clinopyroxene – orthopyroxene, (ii) plagioclase(An60) – diopside, (iii) plagioclase(An60) – enstatite and (iv) plagioclase(An01) – enstatite. Samples were deformed in general shear at strain rates of $3 \cdot 10^{-5}$ to $3 \cdot 10^{-6}$ s^{-1}, 800 °C and confining pressure of 1.0 or 1.5 GPa. Results indicate that dissolution-precipitation creep (DPC) and grain boundary sliding (GBS) are the dominant deformation mechanisms and operate simultaneously. Coinciding with sample deformation, syn-kinematic mineral reactions yield abundant nucleation of new grains; the resulting intense grain size reduction is considered crucial for the activity of DPC and GBS. In high strain zones dominated by plagioclase, a weak, non-random and geometrically consistent crystallographic preferred orientation (CPO) is observed. Usually, a CPO is considered a consequence of dislocation creep, but the experiments presented here demonstrate that a CPO can develop during DPC and GBS. This study provides new evidence for the importance of DPC and GBS in mid-crustal shear zones within mafic rocks, which has important implications on understanding and modelling of mid-crustal rheology and flow.

1 Introduction

Viscous deformation of crustal rocks is usually dominated either by intra-crystalline deformation (dislocation creep), or by a form of diffusion creep together with grain boundary sliding. Two cases of diffusion creep might thereby be differentiated, one where diffusive mass transfer is causing a change in grain shape and grain boundary sliding is a local accommodating mechanism (Lifshitz sliding Langdon, 2006). The other, where grain boundary sliding is the dominat strain accommodating mechanism with diffusive mass transfer ensuring strain compatibility at the grain scale (Rachinger sliding Langdon, 2006). The relative importance of these processes has been discussed by Paterson (1990, 1995), and for the sake of simplicity, we implicitly include the operation of grain boundary sliding when we speak of diffusion creep. Apart from being rate and temperature sensitive, the rheology of viscously deforming rocks is also observed to be material dependent (for a comprehensive list of flow law parameters for different rock types see e.g., Kohlstedt et al. (1995); Shaocheng and Bin (2002); Bürgmann and Dresen (2008); Burov (2011) and references therein). Flow laws for viscous creep exist for different types of rocks, with the majority of these flow laws being determined for monomineralic materials.

In monomineralic aggregates at mid- to lower crustal conditions, grain growth in monomineralic aggregates is assumed to be extensive and the resulting large grain size is expected to render diffusion creep less efficient than dislocation creep (e.g., Brodie and Rutter, 1987; Paterson, 1990). Insights into deformation mechanisms, slip systems, and

flow law parameters have been obtained from experimental studies, e.g., for plagioclase: Tullis and Yund (1985); Shaocheng and Mainprice (1987); Tullis and Yund (1991); Dimanov et al. (1999); Rybacki and Dresen (2000); Stünitz and Tullis (2001); Stünitz et al. (2003); Ji et al. (2004); Barreiro et al. (2007), and for pyroxene: Lallemant (1978); Kolle and Blacic (1982); Raterron and Jaoul (1991); Mauler et al. (2000); Bystricky and Mackwell (2001); Hier-Majumder et al. (2005); Chen et al. (2006); Zhang et al. (2006).

For polyphase mixtures of gabbroic composition, data from high-temperature deformation experiments are published by Dimanov et al. (2003, 2007); Dimanov and Dresen (2005). Depending on the grain size, the differential stress and the volume fraction of pyroxene (as the stronger phase in their pyroxene-plagioclase mixtures), the dominant deformation mechanism identified by these authors is either diffusion creep or dislocation creep. The strain rate of the two-phase aggregates thereby is suggested to be a combination of the strain rates of the individual phases e.g., Dimanov et al. (2003). No mineral reactions were observed in these experiments. In somewhat lower-temperature experiments and mostly under hydrous conditions, Rutter et al. (1985); Getsinger and Hirth (2014), and Stünitz and Tullis (2001) performed deformation experiments with syn-kinematic hydration reactions. Phase mixing was found to be (partly) due to the nucleation of new phases. The authors suggest that the dominant deformation mechanism is grain size sensitive creep by a mix of diffusion creep and grain boundary sliding. Rutter et al. (1985) state explicitly that they interpret diffusion creep in the sense of dissolution-precipitation creep.

In polymineralic mixtures, several processes are known to influence the deformability and the dominating deformation mechanism of the bulk aggregate. The occurrence of mineral reactions and nucleation causes grain size reduction (e.g., Brodie and Rutter, 1987; Fitz Gerald and Stünitz, 1993; Newman et al., 1999; Handy and Stünitz, 2002; de Ronde et al., 2005) and can lead to (further) phase mixing, whereas grain pinning due to secondary phases is likely to impede grain growth (e.g., Olgaard and Evans, 1986; Berger and Herwegh, 2004; Linckens et al., 2011). Furthermore it has been suggested that under certain conditions during diffusion creep, the convergence/divergence rate between two different mineral phases can be faster than between grains of the same mineral phase (e.g. Hickman and Evans, 1991; Wheeler, 1992; Sundberg and Cooper, 2008). The result is more efficient strain accumulation in polymineralic areas compared to monomineralic ones. All these factors enhance diffusion creep rates and thus may lead to a switch of the dominant deformation mechanism from dislocation creep in monomineralic layers, to diffusion creep in polymineralic ones (Etheridge and Wilkie, 1979; Mehl and Hirth, 2008; Linckens et al., 2011; Kilian et al., 2011).

In the absence of fluids, metastable mineral assemblages can be preserved over long time periods (e.g., Jamtveit et al., 2016). When fluids infiltrate, mineral reactions take place.

Under deviatoric stress conditions, deformation is frequently localized along these zones of fluid infiltration and metamorphic reactions (e.g., Austrheim, 1987). A positive feedback between deformation and metamorphic reactions has been recognized for some time but the exact mechanisms of the interaction are still not sufficiently understood. The positive feedback with metamorphic reactions may not be the same for all deformation mechanisms and thus the syn-kinematic occurrence of mineral reactions is a factor that can influence the dominance of a certain deformation mechanism. It has been shown by the experiments of e.g. Linckens et al. (2014); Cross and Skemer (2017) that in the absence of fluids and mineral reactions, phase mixing is seen to be inefficient and necessitate shear strains of > 17 in the case of Cross and Skemer (2017). This is in contrast to fluid assisted deformation in polymineralic rocks, wich are described to have a strong tendency for phase mixing (Kruse and Stünitz, 1999; Kilian et al., 2011; Precigout and Stünitz, 2016). Thus, strain may preferentially localize into wet and/or reactive regions of the lithosphere, promoted by (as mentioned previously) phase mixing and grain size reduction.

In the presence of fluids, mafic rocks are particularly susceptible to reactions during changing temperatures and pressures, representing a suitable material to study the interplay between reaction and deformation. That high strain zones such as ultramylonites usually consist of a phase mixture, indicates their ability to deform at higher strain rates (or lower stresses) than monomineralic aggregates and emphasizes their importance for localizing deformation. In this study, we present results from deformation experiments on water-added plagioclase-pyroxene mixtures. At the imposed pressure-temperature conditions of $\sim 1.0 - 1.5$ GPa and 800 °C, deformation takes place within the lower temperature range of the viscous regime. The metastability of the starting material in the H_2O-present system causes syn-kinematic mineral reactions, thus facilitating the interplay between reaction and deformation in the experiments.

2 Materials and Methods

2.1 Experiments

2.1.1 Starting materials

Experiments are performed on five different starting materials (composition of starting material and chemical composition of minerals are given in Table 1; mineral abbreviations after Whitney and Evans (2010)):

(i) MD: Crushed Maryland Diabase, (Kronenberg and Shelton, 1980; Marti et al., 2017), using a grain size fraction ≤ 125 μm. The Maryland Diabase starting material has a modal composition of plagioclase: ~ 57 vol%, Clinopyroxene: ~ 32 vol%, Orthopyroxene: ~ 8

Table 1. Mineral composition. Representative mineral measurements as normalised oxide wt% and as calculated stoichiometric mineral composition for the different starting materials. All Fe is taken as Fe^{2+} due to the reducing environment in the sample assembly.

wt.-%	Rischuna Albite	Sonora Labradorite	Maryland Diabase Plagioclase core	rim	wt.-%	Cranberry lake Diopside	Damaping Diopside	Damaping Enstatite	Maryland Diabase Cpx	Maryland Diabase Opx
SiO_2	67.87	53.66	51.86	55.67	SiO_2	57.18	54.39	55.98	51.58	52.61
Al_2O_3	20.14	30.37	29.92	27.72	Al_2O_3	0.85	6.37	4.01	1.77	0.75
CaO	0.21	11.09	13.39	10.57	CaO	22.54	17.93	0.80	14.71	1.44
Na_2O	11.71	3.90	3.63	5.11	Na_2O	0.39	1.71	0.27	0.28	0.00
K_2O	0.10	0.39	0.26	0.37	K_2O	0.00	0.00	0.00	0.00	0.00
MgO	0.00	0.00	0.00	0.00	MgO	17.93	16.13	33.28	14.03	19.36
TiO_2	0.00	0.00	0.00	0.00	TiO	0.00	0.34	0.00	0.76	0.28
FeO	0.00	0.00	0.00	0.00	FeO	1.11	2.37	5.13	16.40	25.55
MnO	0.00	0.00	0.00	0.00	MnO	0.00	0.00	0.00	0.48	0.00
Cr_2O_3	0.00	0.00	0.00	0.00	Cr_2O_3	0.00	0.76	0.53	0.00	0.00
Total:	**100.00**	**100.00**	**100.00**	**99.99**	**Total:**	**100.00**	**100.00**	**100.00**	**100.01**	**99.99**
Atoms per 8 oxygen					**Atoms per 6 oxygen**					
Si	2.97	2.42	2.36	2.51	Si	2.04	1.94	1.92	1.95	1.99
Al	1.04	1.61	1.61	1.47	Al	0.04	0.27	0.16	0.08	0.03
Ca	0.01	0.54	0.65	0.51	Ca	0.86	0.68	0.03	0.60	0.06
Na	0.99	0.34	0.32	0.45	Na	0.03	0.12	0.02	0.02	0.00
K	0.01	0.02	0.02	0.02	K	0.00	0.00	0.00	0.00	0.00
Mg	0.00	0.00	0.00	0.00	Mg	0.95	0.86	1.70	0.79	1.09
Ti	0.00	0.00	0.00	0.00	Ti	0.00	0.01	0.00	0.02	0.01
Fe^{2+}	0.00	0.02	0.04	0.02	Fe^{2+}	0.03	0.07	0.15	0.52	0.81
Mn	0.00	0.00	0.00	0.00	Mn	0.00	0.00	0.00	0.02	0.00
Cr	0.00	0.00	0.00	0.00	Cr	0.00	0.02	0.01	0.00	0.00
Total:	**5.01**	**4.96**	**5.00**	**4.97**	**Total:**	**3.95**	**3.97**	**4.00**	**4.00**	**3.99**
An	0.98	0.60	0.66	0.52	En	0.52	0.53	0.91	0.42	0.56
Ab	98.47	0.38	0.32	0.46	Fe	0.02	0.04	0.08	0.27	0.41
Or	0.55	0.02	0.02	0.02	Wo	0.47	0.42	0.02	0.31	0.03

vol%, Accessories: $\sim$ 3 vol% (Qz, Kfs, Ilm, Mag, Bt, Ap).

(ii) An60+En: Synthetic mixture of Sonora labradorite ($\sim$ An60) and Damaping enstatite powder. Grain size fraction of $\sim$ 2 - 125 μm, and 40 - 180 μm.

(iii) An60+Di: Synthetic mixture of Sonora Labradorite and Damaping diopside powder. Grain size fraction of $\sim$ 2 - 125 μm.

(iv) An60+Di: Synthetic mixture of Sonora labradorite and Cranberry Lake diopside powder. Grain size fraction of $\sim$ 2 - 125 μm, and 40 - 125 μm.

(v) Ab+En: Synthetic mixture of Alpe Rischuna albite ($\sim$ Ab98) and Damaping enstatite powder. Grain size fraction of $\leq$ 125 μm, and 40 - 180 μm.

Detailed description of the sample preparation can be found in the Appendix. Synthetic plagioclase-pyroxene powders are mixed with phase proportions of $\sim$ 57 vol% plagioclase to 43 vol% pyroxene. Either 0.2 μl (0.18 wt%) or 0.12 μl (0.11 wt%) H_2O is added to the sample.

2.1.2 Experimental conditions and sample assembly

Experiments are performed using the Griggs-type deformation apparatus at the University of Tromsø, Norway. Experiments are run at confining pressures (Pc) of $\sim$ 1.0 and 1.5 GPa, temperatures (T) of 800 °C and (axial) displacement rates of $\sim$ 2·10^{-8} to 2·10^{-9} ms^{-1} resulting in bulk strain rates of $\sim$ 3·10^{-5} to 3·10^{-6} s^{-1}. General shear type of flow is achieved by placing the rock powder (0.11 g) between cylindrical alumina forcing blocks (diameter of 6.33 mm) pre-cut at 45° with respect to the load axis (Appendix Figure A.1). Descriptions of the experimental setup, data recording and data treatment can be found in the Appendices A1 - A3, experimental conditions are listed in Table 2.

2.2 Strain determination

The thickness of the shear zone (measured normal to the shear plane) at the hit-point is $th_0 = 0.75 \pm 0.03$ mm. During the experiment, $\sim$ 86 $\pm$ 3 % of the axial displacement is accommodated as shear displacement within the shear zone, and $\sim$ 14 $\pm$ 3 % is accommodated as plane strain thinning of the shear zone. As in previous experiments, the shear zone thickness decreases linearly with the applied axial displacement (see Marti et al., 2017).

The shear strain is presented as apparent shear strain, γ_a, and calculated as the sum of the incremental shear displacements divided by the instantaneous shear zone thickness and strain rates are given as apparent shear strain rates, $\dot{\gamma}_a$, (see Marti et al. 2017). γ_a is not a direct measure of the simple shear component in the general shear progressive deformation and should not be used to derive the strain ellipsoid or other strain related parameters. Instead, the procedure described by Fossen and Tikoff (1993); Tikoff (1995) is adopted to calculate the true simple shear component (γ) from the general shear

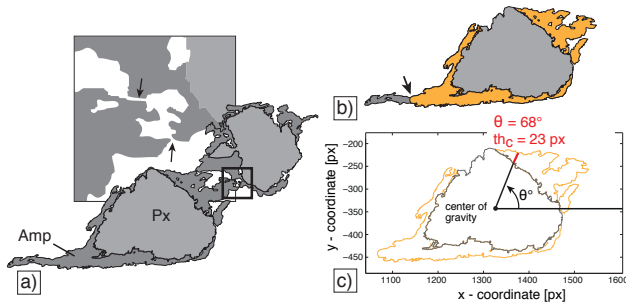



Figure 1. Analysis of amphibole corona thickness. a) Digital phase map of segmented Pyroxene (Px) clasts and associated amphibole (Amp) coronas. Where adjacent coronas are in contact, they are separated manually (close-up, black arrows). b) Long 'tails' of Amp growing in low stress sites around clasts are eventually 'cut' (black arrow) if they are extending too far away from the clast. c) Corona thickness, thc(θ), is determined from the polar coordinates of the aggregate and clast outline as a function of the angle θ ($0° < \theta < 360°$), with θ running counter clockwise from the horizontal.

deformation and used to calculate parameters such as the instantaneous stretching axes (ISA), the orientation of the finite stretching direction, the kinematic vorticity number and the strain ratio given by the ratio of long to short axis of the strain ellipsoid (Table 2). All strains and strain related parameters are calculated for bulk homogeneous sample deformation and do not take into account strain localization.

2.3   Image analysis

After the experiments, samples are immersed in epoxy, cut parallel (in some cases also normal to the shear direction), and prepared to polished thin sections. Polarized light microscope, scanning electron microscope (SEM) and transmission electron microscope (TEM) are used for sample analysis. Grain size and surface fabric are determined as described in Appendix A5.

A special method is developed to study the amphibole coronas that grow on pyroxene porpyhroclasts. Corona thickness is measured as a function of orientation around the clasts (Figure 1). To this end, phase maps of pyroxene and amphibole are created (as described in Appendix A5). Where amphibole coronas of neighboring pyroxene clasts are in contact, individual pyroxene - amphibole pairs have to be separated manually. Manual separation and cleaning was performed by visual inspection and correlation of the original BSE image with the corresponding segmentation. Where necessary, the segmentation was corrected/cleaned with the pencil tool in Photoshop®. Clean phase maps contain segmented pyroxene and amphibole phases, and each pyroxene grain is in contact only with its own amphibole corona. The x-y coordinates of the clast (pyroxene) and the aggregate (pyroxene + corona) outlines are measured and exported using Fiji and a modified version of

the *Jazy XY export macro* (by Rüdiger Kilian, available at *https://github.com/kilir/Jazy_macros*). Using a MATLAB script (available from the author upon request), the x-y coordinates of clast and aggregate outlines are converted to polar coordinates (r-θ), and the corona thickness, thc(θ), is determined at each point along the pyroxene clast as the shortest distance between the clast to the aggregate outline (Figure 1c) as a function of θ. The angle runs counter clock wise from the horizontal. This approach yields good results where coronas follow the clast shape, but tends to underestimate corona thickness where the corona becomes very elongated as e.g. in 'tails' around the clasts. Note that where tails grew extensively long, they were eventually cut, so that the analysis does not include the whole tail length (Figure 1b).

For the interpretation of the amphibole corona evolution it is important to have knowledge of the course and the different stages of an experiment (the details of which are listed in the Appendix). Due to the experimental procedure, the deformation stage is always preceded by an initial hydrostatic part ('lead run-in', Appendix Figure A1c) where the sample is held at $\sim$ hydrostatic conditions for 24 - 30 h. During the initial lead run-in mineral reactions commence prior to sample deformation.

3   Results

3.1   Mechanical data

For all experiments, the mechanical data plotted as shear stress, τ, vs. apparent shear strain, γ_a, show a curve with an initial steep increase of shear stress, reaching a peak value usually after $\sim \gamma_a$ of 0.8 - 1.0 (Figure 2). Peak stress is followed by a slow decrease, often approaching a quasi-steady state shear stress value from $\gamma_a \approx 4$ onwards. The samples with 0.12 μl H_2O added show higher peak stresses and a more rapid shear stress decrease thereafter, compared to samples with 0.2 μl H_2O added. For the Maryland Diabase samples (Figure 2a) at Pc $\approx$ 1.0 GPa, the sample with 0.12 μl H_2O reaches a higher peak stress, but after an additional $\sim$ 0.5 γ_a, it drops to a value similar to the samples with 0.2 μl H_2O. Sample strength of Maryland Diabase at 1.0 and 1.5 GPa reach the same peak stress, but the 1.0 GPa experiments weaken more rapidly within the attained strain range.

The synthetic plagioclase-pyroxene mixtures (Figure 2b) show similar peak stress values (460 - 530 MPa) and all but the An60+En mixture attain similar flow stresses. The synthetic mixtures generally support $\sim$ 60 - 110 MPa higher shear stresses than the Maryland Diabase samples (compare Figure 2a and b). At peak stress, the synthetic mixtures (samples 503, 518 and 519) reach differential stress values near the Goetze criterion. According to Kohlstedt et al. (1995), the Goetze criterion, $\Delta\sigma \leq$ Pc, is an empirically defined stress range where rocks are expected to deform plastically. However, due to the significant weakening subsequent to peak

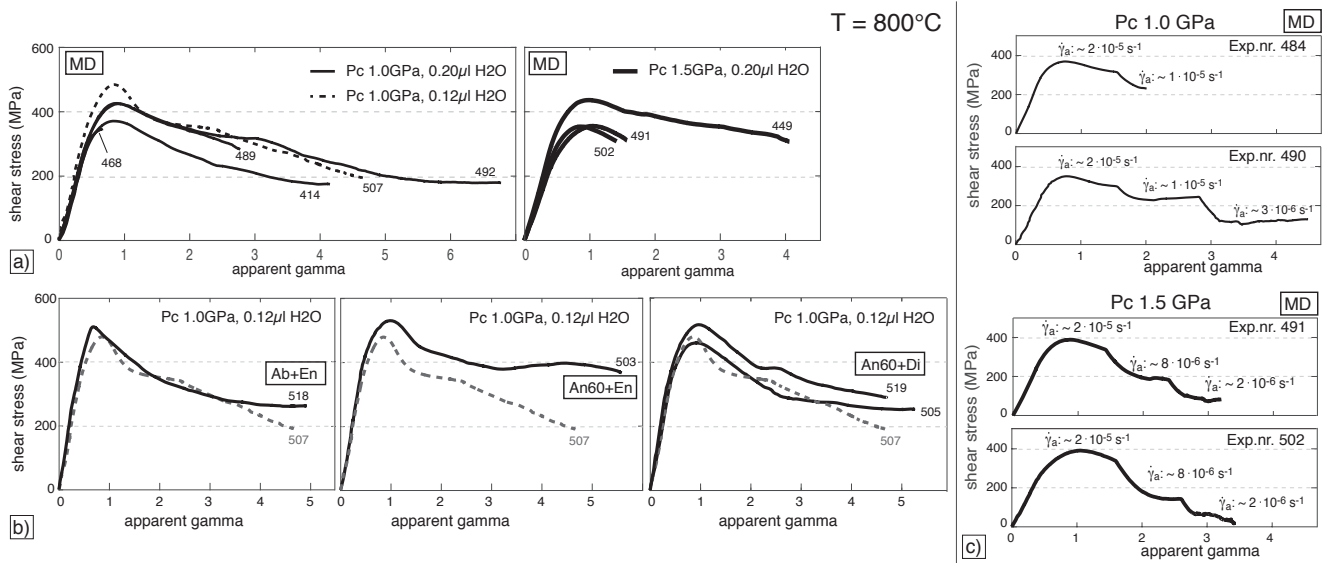



Figure 2. Mechanical data. Shear stress, τ (MPa), versus apparent shear strain, γ_a. Stippled line = Experiment 507 (MD) for reference. a) Maryland Diabase (MD) experiments for different confining pressures, Pc (GPa), and water contents. b) Experiments using different Pl-Px mixtures and constant Pc and water content. c) Displacement-rate stepping tests on MD sample material for experiments performed at different confining pressures.

Table 2. List of experiments.

Exp. nr.	Material	Pc [GPa]	peak τ [MPa]	τ at end [MPa]	mean $\dot{\gamma}_a$ [s^{-1}]	μl H$_2$O added	γ_a	th0 [mm]	thF [mm]	ds [mm]	γ	k	wk	stretching ISA [°]	Ψ [°]	R
414	MD	0.97	407	192	2.1e-5	0.20	5.12	0.75	0.50	2.56	3.32	1.50	0.972	38.1	9.3	14.3
449	MD	1.50	479	337	2.3e-5	0.20	4.51	0.75	0.61	2.75	3.64	1.23	0.994	41.8	11.3	15.6
468*	MD	1.07	348		1.2e-5	0.20	0.70	0.75	0.69	0.48	0.64	1.09	0.968	37.7	28.8	1.9
470*	MD	1.50	446		1.3e-5	0.20	0.86	0.75	0.69	0.65	0.86	1.09	0.982	39.5	27.9	2.3
484**	MD	1.02	371	316 233	1.9e-5 9.5e-6	0.20	 1.97	0.75	0.56	1.33						
489	MD	1.05	428	286	1.9e-5	0.20	3.04	0.75	0.63	1.91	2.54	1.19	0.991	41.1	15.3	8.5
490**	MD	1.00	350	 130	2.4e-5 1.2e-5 2.5e-6	0.20	 5.37	0.75	0.53	2.84	3.72	1.42	0.983	39.7	9.3	16.8
491**	MD	1.52	388	 82	1.7e-5 8.0e-6 2.3e-6	0.20	 4.97	0.75	0.54	2.68	3.52	1.39	0.983	39.7	10.0	15.2
492	MD	1.01	468	197	2.6e-5	0.20	8.95	0.75	0.44	3.94	5.01	1.70	0.978	39.0	5.8	30.8
502**	MD	1.52	391	 33	1.8e-5 8.1e-6 1.7e-6	0.20	 3.90	0.75	0.58	2.26	2.98	1.29	0.985	40.1	12.3	11.3
503	An60+En	1.03	530	367	2.6e-5	0.12	6.55	0.75	0.55	3.60	4.73	1.36	0.992	41.3	8.1	25.4
505	An60+Di (CrLk)	1.01	460	253	2.5e-5	0.12	6.17	0.75	0.55	3.39	4.45	1.36	0.990	41.0	8.5	22.8
507	MD	1.04	479	191	2.4e-5	0.12	5.82	0.75	0.49	2.85	3.69	1.53	0.974	38.5	8.4	17.2
518	Ab+En	1.02	511	263	2.4e-5	0.12	5.83	0.75	0.54	3.15	4.12	1.39	0.988	40.5	8.8	20.0
519	En60+Di (D)	1.02	517	289	2.2e-5	0.12	5.60	0.75	0.54	3.02	3.96	1.39	0.987	40.3	9.1	18.6

Pc=confining pressure, τ=shear stress. $\dot{\gamma}_a$=apparent shear strain rate, th0=shear zone thickness at experiment start, thF=shear zone thickness at experiment end, ds=shear displacement, γ=simple shear component of total strain, k=pure shear component of total strain, wk=vorticity number, Ψ = finite stretching direction, R = ratio strain ellipse major vs minor axis. Angles for stretching ISA and Ψ are given with from east (0°) increasing counter clockwise. MD=Maryland Diabase, Slab=Sonora Labradorite, En=Damaping Enstatite, Di(CrLk)=Cranberry lake Diopside, Di(D)=Damaping Diopside, Ab=Alpe Rischuna Albite. ∗ experiment terminated at peak-stress; ∗∗ displacement-rate stepping test.

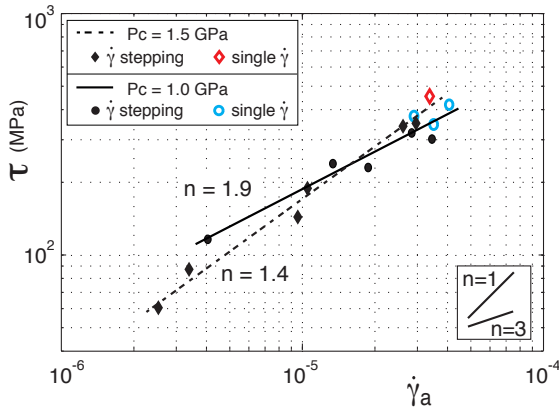



Figure 3. Determination of stress exponents. Shear stress, τ (MPa), versus apparent shear strain rate, $\dot{\gamma}_a$. Two stress exponents, n, are obtained using constant strain rate data and strain rate stepping experiments. For experiments at confining pressures, Pc = 1.0 GPa, n = 1.9, for Pc = 1.5 GPa, n = 1.4. Data for Pc = 1.0 GPa from Marti et al. (2017).

stress, many samples which start above the Goetze criterion then fall substantially below it. The Maryland Diabase samples all stay below the Goetze criterion for all stages of deformation.

5 Strain rate stepping tests on Maryland Diabase sample material at Pc $\approx$ 1.0 and 1.5 GPa have been performed (Figure 2c), to test the sensitivity of shear stress on strain rate. Stress exponents, *n*, of n = 1.9 and n = 1.4 are obtained for experiments at Pc $\approx$ 1.0 and 1.5 GPa respectively (Figure 3).

10 ## 3.2 Microstructures

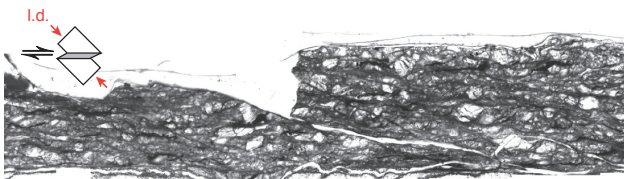
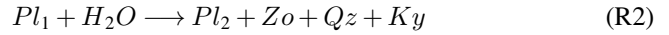
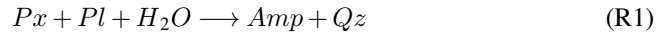



Figure 4. Shear zone overview. Micrograph of sample 492, plane polarized light. Strain localizes into a network of shear bands, anastomosing around low strain lenses identifiable by the large porphyroclasts. Sketch in upper left shows orientation of the micrograph with respect to the loading direction (l.d.) of the sample setup (Appendix Figure A.1).

In all experiments strain is partitioned into a network of shear bands (Figure 4, Figure 5). Their thickness is variable but the main shear band strands usually have a thickness on the order of 40 - 150 μm (e.g. Figure 5d, j) and are char-
15 acterised by strong grain size reduction (Figure 5b, e, k). The following hydration reactions are observed within shear bands and in low strain lenses:

$$Px + Pl + H_2O \longrightarrow Amp + Qz \qquad (R1)$$

$$Pl_1 + H_2O \longrightarrow Pl_2 + Zo + Qz + Ky \qquad (R2)$$

where Pl_2 has a lower anorthite component than Pl_1.

In Maryland Diabase samples, both reaction (R1) and (R2) occur pervasively, with reaction (R1) being the more 25 prominent one. Amphibole grows as reaction coronas on pyroxene clasts and as aggregates, often mixed with quartz, inside shear bands (Figure 5b, Figure 6a, c). Both reactions already initiate during the hydrostatic 'lead run-in' (Appendix Figure A1c) and at the hit point, samples are fully 30 compacted with only few sub-micron sized pores remaining. At peak stress, the volume-% of hydrous reaction products for experiments at 1.0 GPa Pc is $\sim$ 7% (sample 468, duration 38 h = lead run-in and subsequent deformation to peak stress) and $\sim$ 19% for 1.5 GPa Pc experiments (sample 470, 35 duration 46.5 h = lead run-in and subsequent deformation to peak stress). In 1.0 GPa Pc experiments, the volume of hydrous reaction products reaches about $15 - 25\%$ for experiments with durations of $\sim$ 60 - 70 h (lead run-in and subsequent deformation to $\gamma_a \approx 4$ to 6). In 1.5 GPa 40 Pc experiments, the volume of hydrous reaction products reaches up to 31%.

Shear bands in Maryland Diabase experiments are broad and subparallel to the shear zone boundaries (Figure 5c), with an angle $\phi = 3°$ between the preferred orientation of 45 shear bands and shear zone boundaries (see Figure 5 for reference frame). Shear bands are mainly formed by grains with < 1 μm diameter and frequently show a compositional layering between plagioclase dominated and amphibole dominated layers (Figure 5b, Figure 6a, b). Plagioclase 50 layers are either monomineralic or show mixing with zoisite. In amphibole dominated layers, amphibole is frequently occurring together with quartz. Mixing between amphibole and plagioclase is subordinate.

55

In synthetic mixtures of An60+En and An60+Di mixtures (Figure 5d - i), (R2) is the dominant hydration reaction. The volume of hydrous reaction products reaches 1- 9 % for experiments with durations of $\sim$ 66 - 69 h (lead run-in and subsequent deformation). In the An60+En mixture, shear 60 bands are somewhat narrower and more anastomosing. At an angle, $\phi = 9°$, they are also more inclined to the shear zone boundaries compared to the other samples (Figure 5f). Shear bands in both, An60+En and An60+Di mixtures are mainly formed by fine-grained (< 1μm) plagioclase and zoisite. 65
The zoisite reaction predicts the formation of a new plagioclase with a lower anorthite component. The fine grain size within shear bands does not allow for quantitative EDS measurements, but Back-scattered Electron (BSE) images reveal lower Z-contrast (= lower anorthite contents) for plagioclase 70

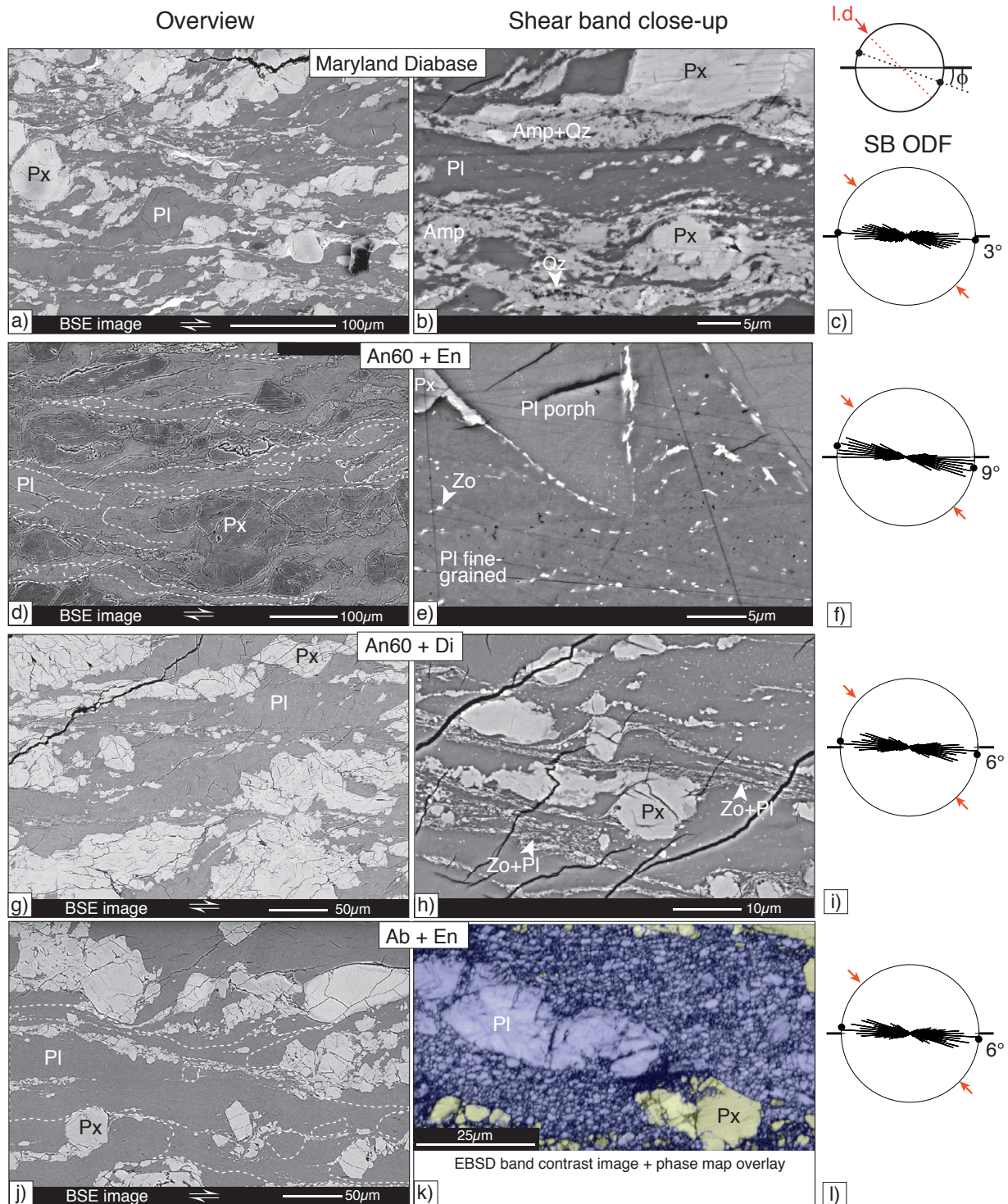



Figure 5. Microstructures of experiments at Pc ≈ 1.0 GPa. a) - c) Maryland Diabase sample material. b) Shear bands are fine-grained and often polymineralic, with the main constituents Pl, Amp and Qz. d) - f) An60+En sample material. Due to the low iron content, pyroxene appears darker than the plagioclase in BSE SEM images. In d) and j), shear bands are traced with white dotted lines. e) Fine-grained Pl + Zo in a shear band next to a Pl porphyroclast. g) - i) An60+Di sample material. j) - l) Ab+En sample material. h) EBSD band contrast image with transparent phase map overlay. Plagioclase appears blueish, pyroxene yellowish. Rose diagrams represent the orientation of the shear bands, black dots indicate preferred trend of shear band segments. Red arrows indicate direction of loading direction. Angle, ϕ, between the shear zone boundary (or forcing block) and the preferred shear band trend is indicated.

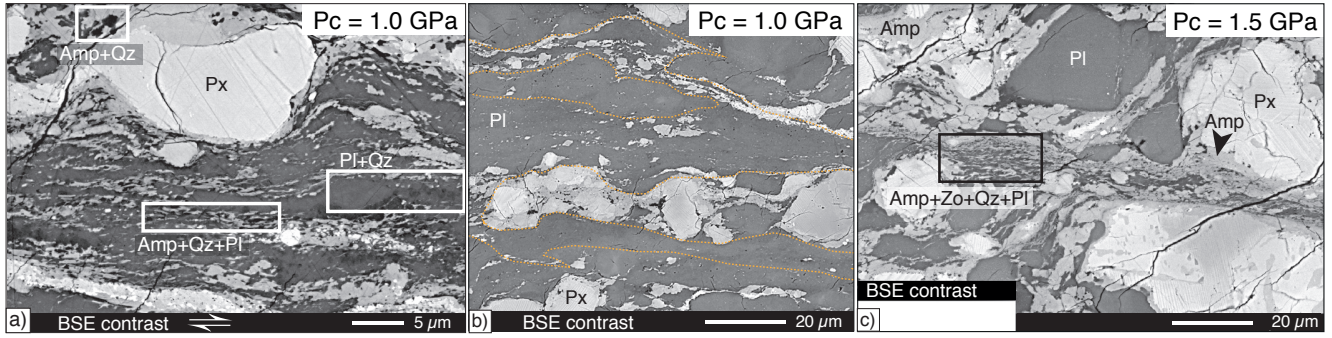



Figure 6. Distribution of phases in Maryland Diabase. a) Area of extensive phase mixing in a shear band: Mixing of Pl+Qz, Pl+Zo, and Amp+Qz. Mixing between Amp and Pl is less frequent. Px clasts show Amp coronas and asymmetric Amp tails. b) Area where shear bands (outlined with dashed orange lines) are predominantly composed of polycrystalline Pl. c) Extensive phase mixing between Amp+Zo+Qz(+Pl) within shear bands. Px clasts show Amp coronas and asymmetric Amp tails.

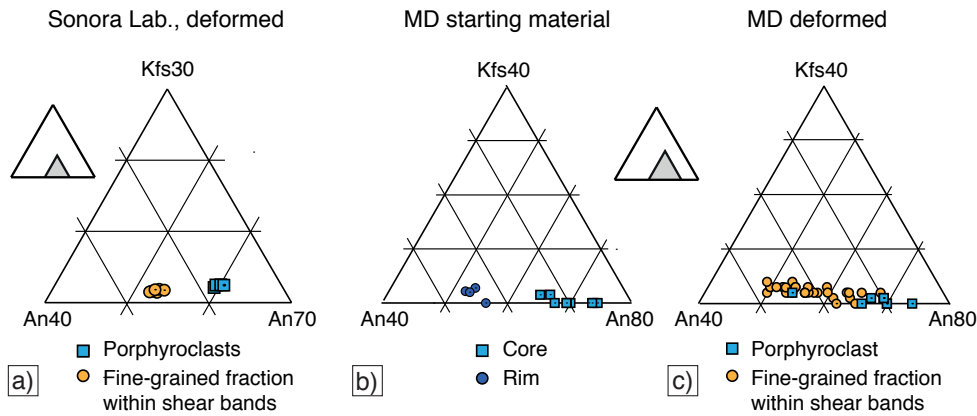



Figure 7. Plagioclase chemical compositions. a) Sonora Labradorite in An60+Di experiment runs. b) Maryland Diabase starting material. c) Maryland Diabase after the experiment. Porphyroclasts vs. small new grain fraction found in shear bands.

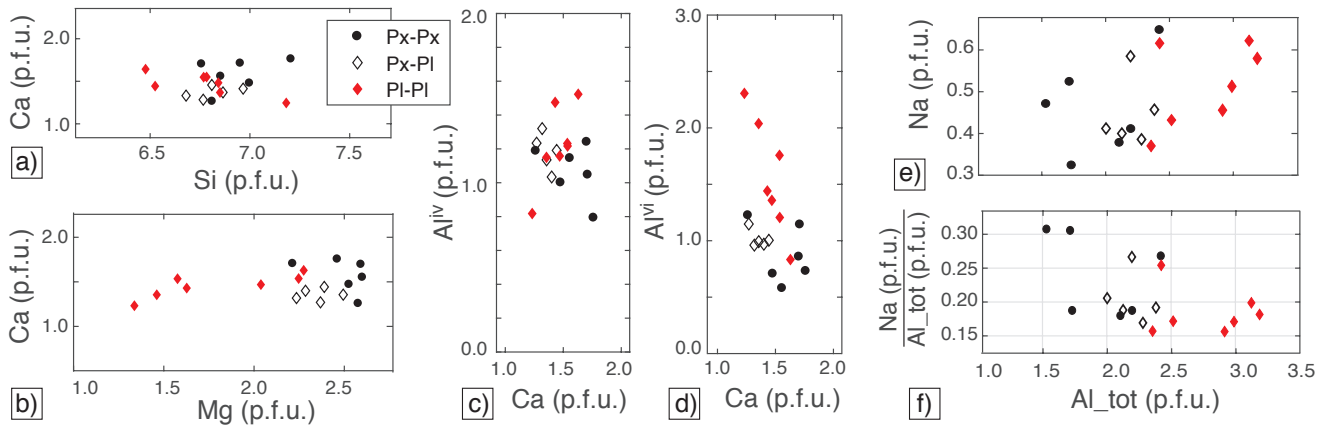



Figure 8. Amphibole chemistry. Amphibole grains of Maryland Diabase experiments performed at Pc ≈ 1.0 GPa. Measurements are grouped acconding to their neighbourhood: Px-Px = pyroxene dominated neighbourhood; Px-Pl = Amphibole grown between pyroxene and plagioclase grains; Pl-Pl = plagioclase dominated neighbourhood. a) Ca vs Si per formula unit (p.f.u.). b) Ca vs. Mg. c) estimated Al^{iv} vs. Ca. d) estimated Al^{vi} vs. Ca. e) Na vs. Al (total). f) Na per Al (total) ratio vs. Al (total).

within shear bands compared to plagioclase porphyroclasts (Figure 5e). Semi-quantitative EDS measurements yield a decrease in anorthite component from $\sim$ An(60) (starting composition) to $\sim$ An(52-55) for grains within shear bands (Figure 7).

In the Ab+En sample, shear bands are broad and sub-parallel to the shear zone boundaries, with $\phi = 6°$ (Figure 5l). Shear bands are pre-dominantly composed of fine-grained plagioclase (Figure 5k) with sizes < 2 μm. No difference in composition between plagioclase porphyroclasts and fine-grained plagioclase within shear band was detected. In high-resolution BSE images, a fine-grained phase with a Z-contrast similar to enstatite is observed. Due to the small grain size EDS measurements are extremely challenging but point to a new type of pyroxene with a somewhat higher Si and Na component.

In all experiments (Maryland Diabase and synthetic mixtures), plagioclase shows extensive grain size refinement. Porphyroclasts are replaced by fine-grained plagioclase, nucleating mainly along porphyroclast rims and along straight internal trails, which are thought to represent former fractures (Figure 5k). The newly nucleated grains generally show a lower anorthite component than the plagioclase in the starting material (Figure 7). In experiments on Maryland Diabase, pyroxene grain size reduction is largely caused by the pyroxene-consuming reaction to Amp (R1, Figure 5b, Figure 6). In the synthetic mixtures however, much of the grain size reduction of pyroxene is caused by fracturing.

The main difference between the microstructures developed at 1.0 and 1.5 GPa Pc (Maryland Diabase experiments) is the increased amount of reaction products at higher Pc (Figure 6). Zoisite and amphibole form more abundantly at 1.5 GPa and amphibole corona surround pyroxene porphyroclasts in early stages of the experiments. Shear bands at Pc $\approx$ 1.0 GPa are mainly composed of a fine-grained mixture of Pl+Amp+Qz+Zo (in order of abundance), as compared to Amp+Pl+Zo+Qz (again in order of abundance) at 1.5 GPa. Additionally, shear bands are somewhat narrower and more inclined to the shear zone boundaries at the higher Pc (compare Marti et al., 2017).

3.3 Amphibole chemistry

For Maryland Diabase experiments at 1.0 GPa, two groups of amphibole are recognized, differing in their Al and Mg per formula unit (p.f.u.), and in their Na to Al ratio (Figure 8, Table 3, Supplementary Table 1). The amphiboles are classified as ranging between Tschermakite and Mg-Hornblende. When labeling the amphibole measurements according to their 2D neighbourhood as observed in the thin section, the Al and Mg contents shows a consistent pattern where high Al - low Mg amphibole grow in plagioclase dominated areas (Figure 8b - d). The Si and Ca contents thereby show no systematic difference between the different grain neighbour-

Table 3. EDS measurements of Amp chemical compositions from samples deformed at Pc $\approx$ 1.0GPa.

normalized to 98 % wt.-%	Amphibole			
	414sm Tschermak.	414sm Mg Hornbl.	490sm Mg Hornbl.	490sm Tschermak.
SiO$_2$	45.18	45.72	47.76	47.31
Al$_2$O$_3$	17.13	14.13	13.06	17.73
CaO	9.24	8.92	9.45	10.02
Na$_2$O	1.63	1.74	2.12	1.84
K$_2$O	1.26	0.86	0.89	0.90
MgO	7.56	9.95	11.24	7.39
TiO$_2$	0.00	1.78	0.00	0.00
FeO	15.99	14.90	13.48	12.81
MnO	0.00	0.00	0.00	0.00
Cr$_2$O$_3$	0.00	0.00	0.00	0.00
Total:	**97.99**	**98.00**	**97.99**	**98.00**
Formula per 23 oxygen				
Si	6.59	6.76	6.89	6.77
Ti	0.00	0.00	0.00	0.00
Al	2.95	2.46	2.22	2.99
Fe^{3+}	0.00	0.00	0.00	0.00
Cr	0.00	0.00	0.00	0.00
Mg	1.65	2.19	2.42	1.58
Ca	1.45	1.41	1.46	1.54
Mn	0.00	0.00	0.00	0.00
Fe^{2+}	1.95	1.84	1.63	1.53
Na	0.46	0.50	0.59	0.51
K	0.23	0.16	0.16	0.16
Total	**15.28**	**15.34**	**15.38**	**15.08**

Amphibole classification after Hawthorne et al. (2012). Tschermak = Tschermakite, Mg Hornbl = Magnesium Hornblende. All Fe is taken as Fe^{2+} due to the reducing conditions in the sample assembly.

hoods. Supplementary Table 1 lists amphibole and plagioclase (starting material and newly nucleated) compositions. Two groups of amphibole compositions are present, which can be distinguished by their Na per Al content ratio (Figure 8e, f). The plagioclase of the Maryland Diabase starting material has an anorthite component of $\sim$ An(65-70), with thin rims of $\sim$ An(52-56) (Figure 7). The core's Na to Al ratio thus is $\sim$ 0.18 - 0.21. Plagioclase is the sole provider for Na and Al in amphibole as pyroxene in the starting material shows only trace amounts of these elements. Most amphibole measurements show an Na:Al-ratio of 0.16 - 0.21 (Figure 8f), consistent with reaction (R1) and the consumption of a plagioclase with a composition of $\sim$ An(65-70). The second type of amphibole, with Na:Al-ratios > 0.25 is comparable to the Na:Al-ratio of the starting plagioclase rim composition of $\sim$ An(52-56) (resulting in Na:Al-ratios of $\sim$ 0.28 - 0.32) and thus again would be compatible with the plagioclase-consuming, amphibole forming reaction (R1).

3.4 Shear bands

3.4.1 Nanostructure of plagioclase within shear bands

TEM images are presented from shear bands formed within the Ab+En sample 518 (Figure 9) and the Maryland Diabase sample 414 (Figure 10). For both samples, micrographs are obtained from foils cut normal to the shear zone boundaries and parallel to the shear direction. Figure 9a shows the interface between an albite porphyroclast and the fine-grained

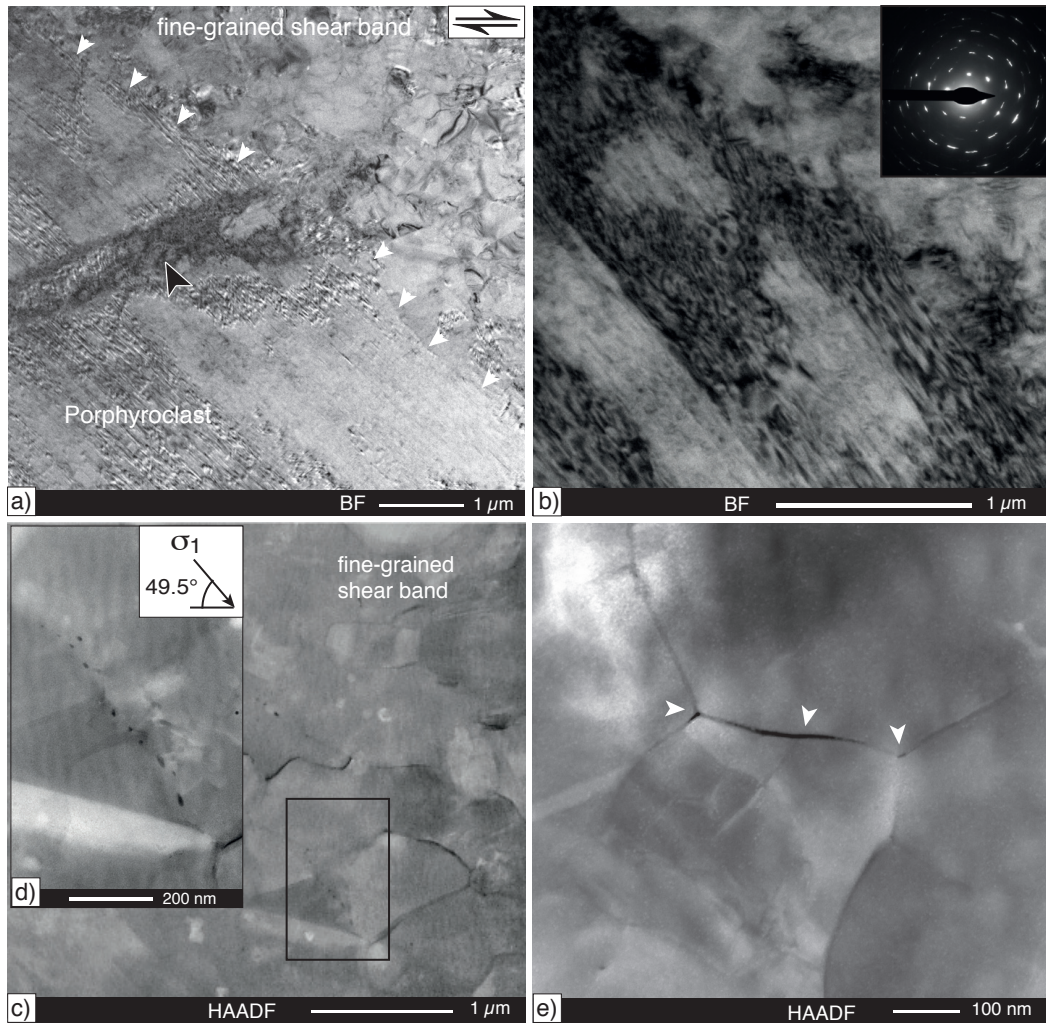



Figure 9. Nano-structures of shear bands in Ab+En sample. Sample (518), shear zone boundaries are horizontal, shear sense is dextral. a) Bright-field (BF) image of a plagioclase porphyroclasts adjacent to a fine grained shear band. White arrows mark the porphyroclast-shear band interface. Black arrow points to a high defect density band within the clast. b) BF image of the internal structure of porphyroclast showing high defect density. Twin lamellae run from upper left to lower right. c) HAADF image of a fine-grained plagioclase in a shear band. Black rectangle marks close-up view in d). d) HAADF image of a pore-trail following several aligned grain boundaries. The local orientation of σ_1 is derived from the orientation of the ISA (Table 2). e) HAADF image. White arrows point to porosity or opening sites developed along two triple-junctions and a grain boundary.

albite matrix of an adjacent shear band. The albite clast has a high defect density, where intragranular domains develop misorientations to each other, as seen in the bright field image or from the rotation of diffraction spots (Figure 9b). However, no recovery to form sub-grain walls is observed. Furthermore, the interface between the clast and the shear band is sharp and no bulges are observed (Figure 9a). Within the shear band, small pores are seen as pore trails along grain boundaries (Figure 9c, d) oriented at a small angle to the expected σ_1.

The shear band formed in the Maryland Diabase sample shows the typical compositional layering between plagioclase dominated layers and amphibole (+Qz) aggregates (Figure 10a). Bright field TEM images reveal largely defect free grains (Figure 10b, d) and grain sizes are similar for amphibole and plagioclase. Grain and phase boundaries are tight and porosity is scarce (Figure 10c, d). Plagioclase grains are weakly anisotropic in shape (not perfectly equant) with a shape preferred orientation sub-parallel to the shear zone boundaries (Figure 10b, d; compare Marti et al., 2017).

3.4.2 Plagioclase grain size distribution within shear bands

2D grain size distributions (GSDs) are determined for plagioclase inside shear bands of the Ab+En experiment 518

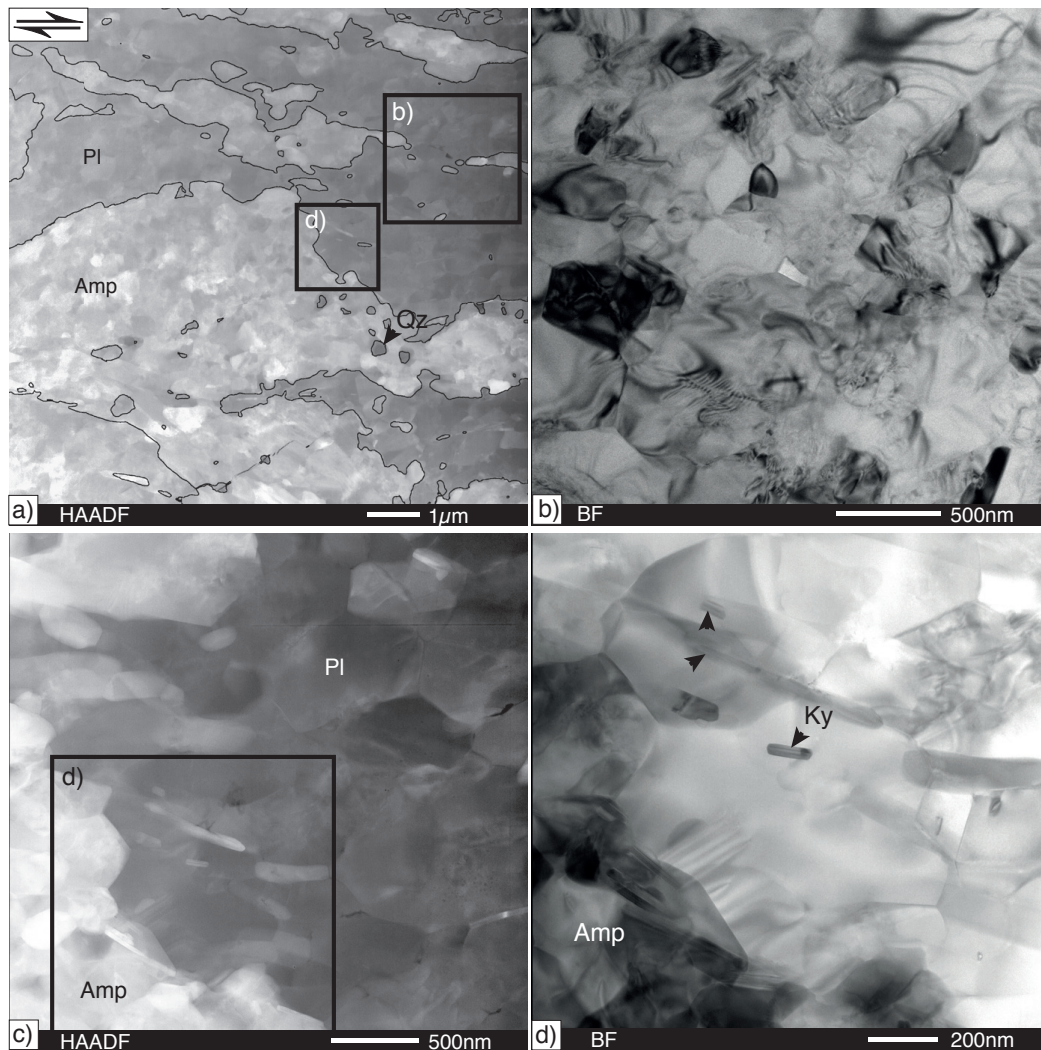



Figure 10. Nano structure of shear bands in Maryland Diabase sample. Sample (414), shear zone boundaries are horizontal, shear sense is dextral. a) HAADF image, overview. Amp aggregates are traced with black lines for better visibility. Rectangle indicate areas shown in b) and d). b) BF-TEM image of small (usually ≤ 600 nm) plagioclase grains with low internal defect densities. Grain boundaries are tight and porosity is scarce. c) HAADF image, overview. Black rectangle indicates area shown in d). d) BF-TEM image of a few Ky and Amp grains growing between Pl grains. Size of all phases is a few 100 nm, grains have a low internal defect density.

and the MD experiment 414 (Figure 11). The distributions in the two samples are similar with somewhat higher frequencies in bins > 1 μm for albite compared to the labradorites of the Maryland Diabase sample. Due to the small grain sizes and extremely narrow grain boundaries, grains are difficult to identify on SEM images and there is some uncertainty concerning the measured GSDs inherent from the grain segmentation. Nonetheless, grains segmented from TEM and SEM images correlate well for the highest frequency bins. Measured on TEM and SEM images, the GSDs for sample 518 have modes at 0.51 and 0.36 μm respectively. In sample 414, the GSDs have modes at 0.23 and 0.30 μm (Figure 11).

3.4.3 Albite crystallographic preferred orientation

Three EBSD maps are collected along one shear band in the Ab+En sample 518. Orientation data of plagioclase from three distinct sites (Figure 12) are used to test for the presence of a crystallographic preferred orientation (CPO). Grains are calculated with a threshold angle of $10°$ and merged across boundaries consistent with the albite twin law. Porphyroclasts and their adjacent fragments as well as grains with < 2 μm equivalent diameter and < 3 points are excluded (Figure 12b).

The pole figures (Figure 12c), which combine the data of all three sites, show a weak, but distinct CPO, where especially [010] and (010) show systematic arrangements of a maxi-

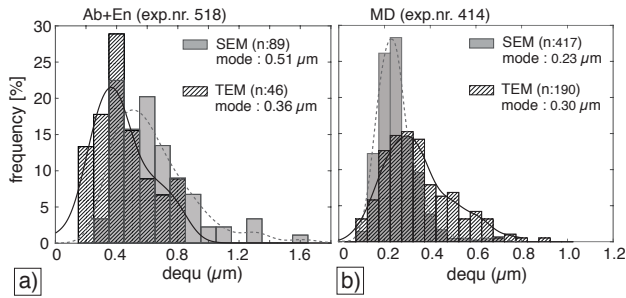



Figure 11. Grain size distributions of plagioclase in shear bands. a) Ab+En experiment 518, and b) the Maryland Diabase experiment 414. 2D grain size distributions (GSDs) in both samples are determined on BSE SEM images and TEM images separately. n = number of grains; solid black (TEM) and dashed grey lines (SEM) are kernel density estimate fits to the GSDs and modes determined from the fit are given in the graph.

mum $\sim 35°$ off the periphery and with a high angle to the trace of the shear band. [100] axes and poles to (110) form local maxima along the trace of the shear band plane. Inverse pole figures (IPF) (Figure 12d) are plotted for each site individually. Reference directions for the IPF are 0° (parallel to the global shear direction) and 90° (normal to the global shear plane). Choosing e.g. the bulk finite stretching direction (+10°) or the shear band trace (-10°) and their normals did not result in significantly different results. Maxima of all distributions have moderate strengths and occupy nearly identical positions for all sites and at each reference direction (showing the close similarity of the CPO developed in all three individual sites). For all distributions, the positions of maxima do not coincide with any common poles to low index planes or directions. Distributions with respect to the shear plane normal are strongest with pole maxima at (-121) (90° reference frame).

3.5 Amphibole coronas

Amphibole grows abundantly in experiments on Maryland Diabase, especially at the higher Pc (e.g. Figure 6c), where pyroxene clasts are surrounded by amphibole coronas already in early stages of the experiment. It has been noted that whereas coronas seem to grow symmetrically during the hydrostatic stage of an experiment, they evolve to become narrower in high stress sites around their host porphyroclast and grow larger in the clasts strain shadow during sample deformation (Figure 13a). To quantify this corona thickness evolution, pyroxene clast - amphibole rim pairs from experiments performed at Pc ≈ 1.5 GPa were analysed, studying the average amphibole corona thickness as a function of orientation around pyroxene porphyroclasts (Figure 13). Coronas are measured at three different stages: at hydrostatic conditions ($\Delta\sigma \approx 0$), at peak stress ($\Delta\sigma > 0$; $\gamma_a \approx 1$) and after considerable deformation ($\Delta\sigma > 0$; $\gamma_a \approx 4$) corresponding to

three evolutionary stages of a typical high strain experiment (Appendix Figure A.1c). Accordingly, three cases are distinguished. The *hydrostatic* case represents the microstructural state at the hit-point after the lead run-in. The *peak stress* case records the microstructural state at the time the sample has reached its maximum strength (including lead run-in and initial sample loading) and the *deformed* case represents the microstructure evolved after the sample underwent high shear strain (including lead run-in, sample yielding and deformation; for an explanation on the nomenclature used see Appendix Figure A.1c).

At hydrostatic conditions (e.g. during the lead run-in), corona growth is symmetrical around the clasts, with an average thickness of 2.4 to 3.1 μm (Figure 13b). In the *deformed* case, the average corona thickness shows an overall monoclinic shape. Assuming that the microstructure after lead run-in (hydrostatic part) is approximately the same as that of the *hydrostatic* case sample, the corona thickness in the *deformed* sample is reduced by $\sim 0.5 - 2$ μm in directions close to the loading direction. Thickness is reduced in most directions except in the range of 346° - 53° and 186° - 232°, where it is increased. That is, on average, the corona thickness is reduced on clast surfaces facing the loading direction, and increased at high angles to the loading direction. At *peak stress* the average corona thickness in direction of loading is the same as in the *hydrostatic* case (~ 2.7 μm) but already increased in almost all other directions. Furthermore, despite the 23 h longer duration of the *hydrostatic* run compared to the *peak stress* run (Figure 13d), coronas did not grow to larger thicknesses in the former.

Instantaneous stretching and shortening axes (ISA), finite stretching directions and vorticity numbers (Wk) are calculated for the *peak stress* and the *deformed* case (Figure 13c). The orthorhombic shape of the peak stress corona curve is well described by the ISA, e.g., such that the long side is normal to the shortening ISA. After deformation, the long diameter of the monoclinic shaped corona curve is oriented between the stretching ISA and the finite stretching direction. The short diameter corresponds to the shortening sector around the pyroxene clast. The direction of the maximum corona thickness is at a higher angle with respect to the shear plane than the finite stretching direction (Figure 13b).

4 Discussion

4.1 Physics and chemistry of grain size reduction

There is a drastic grain size decrease (down to diameters < 2 μm) accompanied by the shear band formation (Figure 5, Figures 9-11). Fracturing as an important process of grain size reduction is only observed in pyroxene grains of the synthetic mixtures (e.g. Figure 12b). In these samples, pyroxene only participates in mineral reactions to a minor degree, and the plagioclase hydration reaction (R2) is dominating (ex-

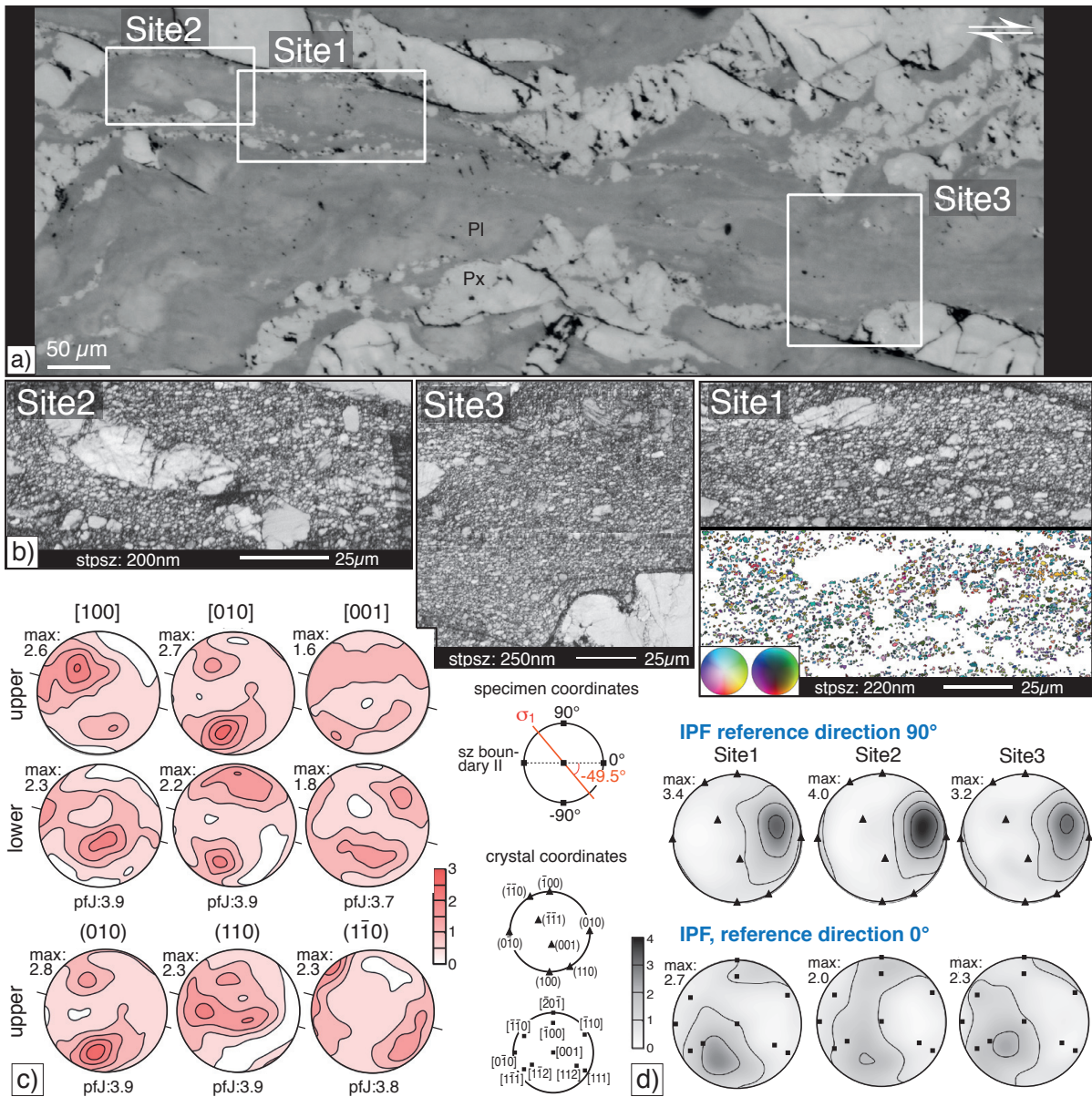



Figure 12. EBSD analysis of Ab+En sample. a) Reflected light image. EBSD map locations are indicated, Pl = plagioclase, Px = pyroxene. b) Band contrast images and EBSD map (for site1, inverse pole figure coloring, x direction). stpsz = step size. c) Pole figures (equal area projections) plotted from combined data of all sites. Orientation data of grains < 2 μm in diameter (and min. > 3 pixels) are plotted. Small black lines at pole figure rims indicate approximate trace of shear band (plunging with 15° to the right). d) Inverse pole figures (IPF) for directions and poles to planes respectively, for reference directions 90° and 0° (normal to the global shear plane, and parallel to shear direction respectively). For the specimen coordinate reference sketch: sz = shear zone. number of grains analyzed: site1: 2'492, site2: 1'647, site3: 3'513

cept for the Ab+En sample). In contrast, pyroxene grains in experiments on Maryland Diabase show grain size reduction by dissolution during the pyroxene-consuming reaction (R1) to amphibole.

5 Fracturing only minorly contributes to the grain size reduction of plagioclase. Instead, grain size reduction is primarily caused by mineral reactions and abundant nucleation of new grains. New plagioclase grains have a different composition from that of the original clasts (e.g. Figure 7). The low defect densities, the narrow grain size range, and the lozenge shaped grains of the very fine-grained pure plagioclase aggregates within shear bands (Figures 9-11) are in accordance with a formation by nucleation and limited growth. Of the two initial mineral phases, plagioclase and pyroxene, plagio-

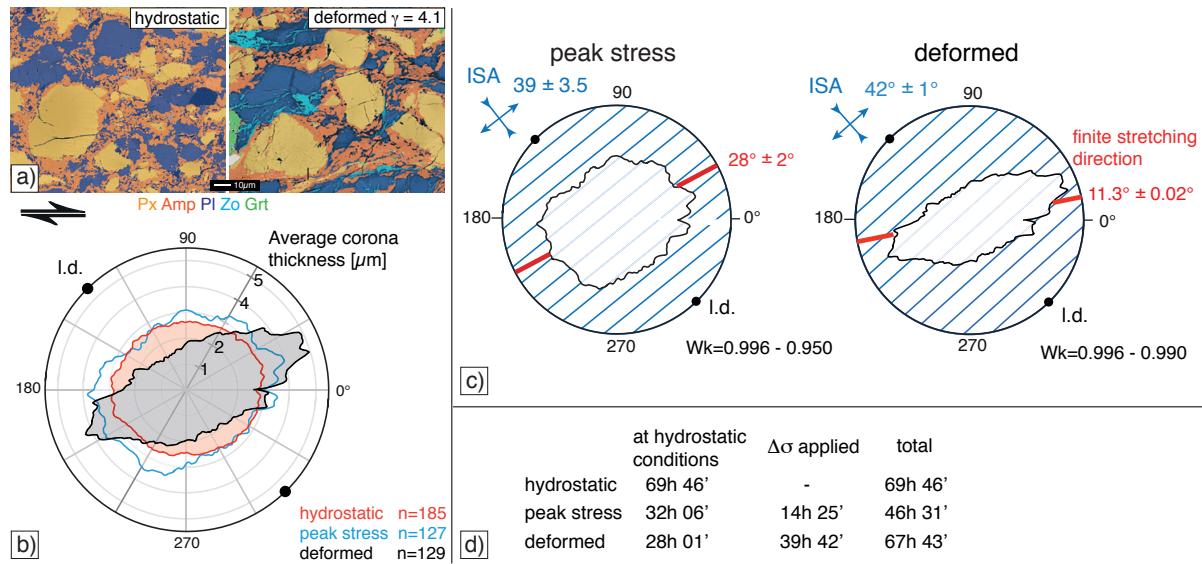



Figure 13. Analysis of amphibole coronas. Thickness of amphibole (Amp) corona on pyroxene (Px) porphyroclasts as a function of orientation, from Maryland Diabase experiments deformed at Pc ≈ 1.5 GPa. a) Phase map of microstructure developed when no $\Delta\sigma$ was applied ('hydrostatic') and in the case of sample deformation ($\Delta\sigma > 0$, 'deformed'). b) Average Amp corona thickness presented as rose diagram. n = number of analysed coronas. Analysis for three different samples are presented, 'hydrostatic', 'peak stress' ($\gamma_a \sim 0.6$) and 'deformed' ($\gamma_a \sim 4$). c) Duration of experiments in hours (h) and minutes ('). d) Kinematic analysis of b), blue lines indicate calculated instantaneous stretching directions. In red, calculated finite stretching direction. l.d. = loading direction. Wk = kinematic vorticity number. The error range in ISA direction, finite stretching direction and Wk is caused by the uncertainty of the starting thickness of the shear zone.

clase is particularly susceptible to grain size reduction via reaction and nucleation of new grains. For all new phases, like zoisite and amphibole, it is clear that reaction and nucleation are the mechanisms leading to a small grain size and to phase mixing.

In the special case of the Ab+En experiment, it is difficult to observe an obvious change in chemical composition of the plagioclase and to connect it to grain size reduction in shear bands. Qualitative EDX measurements reveal possible new pyroxene grains with higher Si and Na contents compared to the starting pyroxene. Due to the very small grain size, however, chemical measurements are challenging. No measurable change in plagioclase composition is detected but in order for the new pyroxene to grow with higher Si and Na contents compared to the starting material, a plagioclase with a higher anorthite component is expected to grow.

Microstructural evidence for grain size reduction by fracturing or dynamic recrystallization (e.g. subgrain rotation or bulging recrystallization) of plagioclase is not observed (see e.g. Figure 9). In addition, had the recrystallization of plagioclase in monomineralic domains taken place by dynamic recrystallization, the resulting grain sizes would imply very high stresses. Using the normalized grain size/stress relationship by Derby (1991), the observed plagioclase grain size mode of ~ 0.4 μm (Figure 11) would require differential stresses of 2 to 2.5 GPa. The observed differential stresses are ~ 500 MPa in the last stages of this experiment (Figure 2) -

they are clearly far too low to produce such a small grain size in equilibrium. As in other samples, grain size reduction of plagioclase in the Ab+En sample is considered to take place by dissolution of original porphyroclasts and nucleation (i.e. "neo-crystallization") of new grains.

4.2   Derivation of the stress exponent

The determined n-values are low, with n = 1.4 and 1.9 (Figure 3) and are thus within the range of expected values for diffusion creep (including grain boundary sliding) with theoretical values between 1 (e.g., Ashby and Verrall, 1973; Coble, 1963; Karato, 2008; Kohlstedt and Hansen, 2015; Paterson, 2013), up to 2 (e.g., Gratier et al., 2009, 2013; Paterson, 2013). The stress exponents determined in this study have to be taken with some caution as deformation of the samples is inhomogeneous. While the shear bands are able to accommodate higher strain rates, the lesser deformed domains in-between seem to still control the overall bulk stress (Marti et al., 2017). Nevertheless, the low stress exponents strongly suggest an absence of frictional deformation and make dislocation creep unlikely.

4.3   Dissolution-precipitation creep and grain boundary sliding

From the mechanical data (including stress exponents), the determined grain sizes and from the nucleation of new

grains, it is concluded that in all samples, the dominant deformation mechanism cannot be frictional or crystal plastic (dislocation creep). Instead, the dominant deformation mechanisms is inferred to be DPC, accompanied and/or accommodated by mineral reactions. Pyroxene is less involved in accommodating strain but plays an important part by being involved in mineral reactions and thereby aiding grain size reduction by nucleation of new grains.

Mineral reactions change the initial phase assemblage of Px+Pl to mostly Pl+Px+Zo in the synthetic mixtures, and Pl+Px+Amp +Qz+Zo in Maryland Diabase samples. Disregarding the differences in amount and type of mineral reactions, strain is always localized into a network of shear bands characterized by intense grain size reduction and phase mixing (to a lesser extent in the Ab+En sample; Figure 5).

The small size of grains in shear bands (Figure 11) clearly favours a grain size sensitive deformation mechanism such as DPC and related, grain boundary sliding. This interpretation is also supported by the strain free interior and grain morphology of the small grains in shear bands (Figure 9 and 10). The activity of solution-mass transport processes is clearly indicated by the vast extent of mineral reactions, which necessitate the movement of chemical components over several 10's of μm at least. The morphology of pores presented in Figure 9 is further supporting evidence for DPC interpretation; the pore trail in Figure 9d is interpreted to have formed by precipitation of plagioclase and entrapment of residual fluid along grain boundaries with a trace sub-parallel to the estimated σ_1 direction (expected opening direction of dilatancy normal to σ_1).

4.4 Evidence for dissolution-precipitation creep of amphibole

Microstructural evidence for DPC is usually scarce but the truncation of grains (or other markers such as chemical zonation) and their overgrowths are sometimes correlated with the deformation by DPC (e.g. Schwarz and Stöckhert, 1996; Wintsch and Yi, 2002; Svahnberg, 2010). The evolution of the amophibole corona thicknesses has been analyzed to investigate if it could be explained in terms of amphibole deformation by DPC.

During the experiments, amphibole grows as a reaction product and pyroxene porphyroclasts are replaced at their rims by amphibole growth coronas. During all stages of an experiment, amphibole is observed to grow stably. Clasts with coronas are predominantly found in low strain lenses, where shear strains are lower than in shear bands. No microstructure can be found to indicate that the reduction in average corona thickness at the compressional sites of the clasts is due to 'shearing-off' of amphibole from compressional to extensional sites by some sort of granular flow. On the contrary, the amphibole coronas, which grow

symmetrically in thickness during the hydrostatic 'lead run-in', become partly dissolved in high stress sites while simultaneously growing thicker in low stress sites during deformation (Figure 13b). These results indicate grain scale DPC of amphibole. It is frequently observed for amphiboles in naturally deformed rocks (e.g., Berger and Stünitz, 1996; Imon et al., 2002; Marsh et al., 2009; Stokes et al., 2012), but has only rarely been reproduced in experimental studies (Rutter et al., 1985; Getsinger and Hirth, 2014).

The direction of the shortening ISA (equal to the direction of the instantaneous maximum principal stress, σ_1) lies within the range of directions of minimum average corona thickness. A correlation between the shortening ISA and the minimum in average corona thickness is consistent with the interpretation of DPC, where material is preferentially dissolved along high stress sites (and re-precipitated along low stress sites). During DPC, the grain shape change is determined by the stress field, where the instantaneous (i.e. for very low strains) shape change is expected to result in an orthorhombic grain shape with the short and long axis at 90° and parallel to σ_1 and σ_3 respectively. With progressive deformation during DPC, grain boundary sliding will take place alongside, causing grain rotation and a deviation from this orthorhombic shape fabric. At the *peak stress*, where shear strain is still small, the rim thickness shows an overall $\sim$ orthorhombic shape (see corona curve in Figure 13c), well aligned with the ISAs. As the ISAs indicate the minimum and maximum principle stress directions, the fact that the corona curve follows the ISAs correlates well with the interpretation that DPC determines the amphibole corona thickness evolution. The monoclinic shape of the corona curve in the *deformed* case can be explained by a superposition of co-axial geometry of deformation by diffusion creep, and rigid body rotation induced by the rotational component of simple shear.

4.5 Albite crystallographic preferred orientation

Dislocation creep and dynamic recrystallization are not considered to occur in our experiments. The large Burgers vectors in plagioclase are unfavourable for intracrystalline deformation, especially at the comparatively low experimental temperatures of 800 °C. Dislocation glide and climb have been suggested to be active in plagioclase at both natural and experimental conditions (e.g., Tullis and Yund, 1985; Shaocheng and Mainprice, 1987; Yund and Tullis, 1991; Rybacki and Dresen, 2000; Shigmeatsu and Tanaka, 2000; Kruse et al., 2001; Lapworth et al., 2002; Stünitz et al., 2003; Ji et al., 2004; Barreiro et al., 2007; Mehl and Hirth, 2008) but usually are not considered to accommodate large amounts of strain. Recrystallization takes place by different mechanisms including neo-crystallization (e.g., Fitz Gerald and Stünitz, 1993; Rosenberg and Stünitz, 2003; Brander et al., 2012; Fukuda and Okudaira, 2013; Mukai et al., 2014)

or by growth of fragments formed by fracturing (e.g., Stünitz et al., 2003; Viegas et al., 2016). In fine-grained aggregates, diffusion creep (in the broadest sense) often dissolution-precipitation creep (DPC), is the main strain accommodating process described for polycrystalline plagioclase aggregates (e.g., Yund and Tullis, 1991; Fitz Gerald and Stünitz, 1993; Jiang et al., 2000; Lapworth et al., 2002; Rosenberg and Stünitz, 2003; Brander et al., 2012; Fukuda and Okudaira, 2013; Mukai et al., 2014; Viegas et al., 2016).

The CPO measured in a shear band of the Ab+En sample 518 is generally weak, but the three independent sites 1 - 3 show very similar CPO patterns (Figure 12c, d). This similarity indicates that these CPOs, although weak, are not random but that there must be a mechanism leading to this weak but systematic CPO.

There are a number of mechanisms that can lead to a CPO within an aggregate. Examples are dislocation glide (e.g., Schmid and Boas, 1950) or directed growth (possibly together with rigid body rotation; e.g. Shelley, 1994; Berger and Stünitz, 1996; Rosenberg and Stünitz, 2003; Getsinger and Hirth, 2014; Viegas et al., 2016). Furthermore, a CPO can form via host-controlled nucleation (e.g., Jiang et al., 2000) or caused by interface-controlled diffusion creep (e.g., Bons and den Brok, 2000; Sundberg and Cooper, 2008). Similar CPOs as the ones shown here have been found in experimentally deformed anorthite and basalt (e.g., Ji et al., 2004; Barreiro et al., 2007), and in naturally deformed basaltic and peridotitic rocks (e.g., Mehl and Hirth, 2008; Viegas et al., 2016; Xie et al., 2003; Drury et al., 2011). Ji et al. (2004) and Mehl and Hirth (2008) interpret the CPOs to be due to dislocation creep in monomineralic plagioclase layers. Viegas et al. (2016) observe no evidence for dislocation creep and suggest that the CPOs may be the result of directed growth and rigid body rotation of grains (with a crystallographically controlled grain shape) during diffusion accommodated grain boundary sliding.

For plagioclase, the most commonly reported slip plane in dislocation glide is (010) (e.g. Kruse et al., 2001). However, although (010) aligns at a high angle to the shear plane (as seen from the pole figures, Figure 12c), the position of the maxima in the IPF is significantly off ($\sim$35°) the pole to (010). Maxima in the IPF are close to [3-14] (for reference direction = shear direction) and (-121) (for reference direction = normal to shear plane). They do not correlate with the [100] or [001] slip directions and the (010) slip plane commonly reported for feldspars in the literature. It is therefore argued that the CPO is not due to dislocation creep but rather caused by some other process(es) during grain size sensitive creep in the predominantly monomineralic albite layers.

The exact CPO forming mechanism could not be determined. It may be speculated that the CPO formation could be due to directed and anisotropic growth during DPC and grain boundary sliding of grains with a crystallographically controlled shape. Studies on plagioclase crystal growth in magmatic environments usually determine [100] and/or [001] as

fast growth directions, and crystals forming with pronounced (010) planes (e.g. Smith, 1974). In hydrothermal growth experiments on albite, Franke and Ghobarkar (1982) report an increasing growth velocity of (110) at higher experimental temperatures (up to $\sim$700°C). The alignemnt of (110) poles within the trace of the shear band (polefigures, Figure 12c) and that of (010) poles at a high angle is conspicuous and may indicate that the CPO is caused by the combined mechanisms of grain rotation during deformation and anisotropic growth.

4.6   Importance of dissolution-precipitation creep in natural rocks

Grain size reduction is energetically unfavourable as it increases the total grain surface area. DPC does not necessarily require the formation of new grains, instead, precipitation could take place as overgrowth rims on existing grains. The intense grain size reduction observed in shear bands within our experiments is most probably caused by high nucleation rates. High nucleation rates are typically attained by a large overstepping of a reaction boundary (e.g., Rubie, 1998; Putnis, 1992), introducing a high driving potential. The start of our experiments represents such an instance of large overstepping of reaction boundaries. The starting materials + H_2O are not in equilibrium at the experimental Pc-T conditions. As the pressurization and heating procedure required to attain the experimental Pc-T conditions takes place within $5-8$ h, the sample material is brought rapidly to a metastable state. Although this rapid change in P-T conditions is unique to experiments, there is widespread evidence from observations of natural rocks that similarly, metastable mineral assemblages can be sustained even at high-grade conditions and to large overstepping of reaction boundaries, when rocks are dry (e.g., Rubie, 1986; Austrheim, 1987; Wayte et al., 1989; Krabbendam et al., 2000; Austrheim, 2013; Jamtveit et al., 2016). Only where fluid infiltrates, mineral reactions are enabled and equilibration can be attained.

TEM analyses revealed high defect densities in the albite porphyroclasts (Figure 9a, b). It has been proposed that a high intragranular defect density results in an increased rate of grain dissolution, speeding up the reaction and/or deformation rate (e.g., Wintsch, 1985; Schott et al., 1989; Stünitz, 1998). However, the observation of, e.g., abundant nucleation along former fractures (Figure 5k) cannot simply be accredited to enhanced reaction rates due to locally increased strain energy (in the sense of high defect densities locally introduced by fracturing according to, e.g., Fitz Gerald et al., 1991; Fitz Gerald and Stünitz, 1993; Stünitz et al., 2003; Trepmann et al., 2007). Fracturing is always accompanied by dilatancy. Fluid infiltrates the fractures, leading to higher solution-mass transport rates (e.g., Fitz Gerald and Stünitz, 1993; Precigout and Stünitz, 2016). Thus higher reaction rates can be expected. The significance of strain energy as a possible rate-enhancing contributor to reaction and nucle-

ation is thus difficult to separate from the effects of enhanced fluid flow.

With the dominant deformation mechanisms, DPC and grain boundary sliding, the samples presented here deform viscously. At the experimentally induced conditions of T = 800 °C and at strain rates of $\sim 3 \cdot 10^{-5}$ s^{-1}, deformation takes place at the lower end of the viscous field, close to the brittle-viscous transition of the studied material (e.g., Marti et al., 2017). However, it is probable that the observed deformation mechanisms are also active at higher temperatures in these types of rocks. The principal constituent phases of mafic rocks (plagioclase + pyroxene + amphibole) all have high strengths in terms of intracrystalline plasticity, even at high temperatures. Where water is absent and DPC suppressed, the build-up of high stresses can be expected in such rocks, at least transiently (e.g., Okudaira et al., 2015). If local stresses are high enough to induce cracking, dilatancy and fluid infiltration may be facilitated. Once reactions start to operate (metastability of mafic rocks is very common because the mineral compositions are very variable and critically dependent on P, T, fluid-composition), a switch to DPC is likely to occur. The resulting deformation takes place with low stress exponents.

4.7 Continued operation of deformation mechanisms at higher strain

In the case of the experimental set-up described here, where the starting mineral assemblages are not in equilibrium at the imposed P-T-fluid conditions, the chemical driving potential for attaining a lower energy assemblage partially controls the reaction rate (although for a stressed system, there is no unique driving force/Gibbs free energy defined, e.g. Kamb, 1961; Wheeler, 2014, 2018). In principle, when the stable assemblage is reached, i.e., when the reaction has gone to completion, the driving potential for dissolution of phases is reduced, and it may be expected that the deformation rate is reduced, too. Interestingly, the measured amphibole compositions show variations, which are most probably influenced by the local mineral composition of the other neighbouring mineral phases (Figure 8). Shear offset and neighbour switching during deformation by diffusion creep and grain boundary sliding will continuously change the local neighbourhood of grains. As the neighbourhood of a given grain changes, so will new surfaces be brought in contact and become involved in reactions, potentially providing a relatively constant chemical driving force for reaction and nucleation, if local disequilibrium prevails. Thus, it may take considerable time until the deforming assemblage has reached complete equilibrium, and some local chemical driving potentials may persist, even when the nominal bulk equilibrium assemblage has formed.

5 Summary and Conclusions

(i) Viscous deformation in experiments with mafic compositions at temperatures of 800 °C and confining pressures of 1.0 and 1.5 GPa at strain rates of $\sim 10^{-5}$ s^{-1} is dominantly achieved by dissolution-precipitation creep (DPC) and grain boundary sliding, accompanied by syn-deformational mineral reactions. No evidence for frictional deformation or significant contributions of dislocation glide or creep to the accommodation of strain can be found for any of the mineral phases. Strain is frequently localised into shear bands, which consist of fine-grained mixtures of neo-crystallized plagioclase and the syn-kinematic reaction products amphibole, quartz and zoisite, none of which are present in the starting material.

(ii) Intense grain size reduction is produced by high nucleation rates, probably caused by a large overstepping of reaction boundaries. Both, deformation and nucleation are localised in shear bands, implying a positive feedback between the two mechanisms.

(iii) Amphibole is seen to accommodate displacement via dissolution-precipitation creep, as interpreted from the evolution and distribution of amphibole coronas on pyroxene porphyroclasts.

(iv) A weak but systematic crystallographic preferred orientation (CPO) of albite - unrelated to any known slips system operating in dislocation creep - is formed in the shear bands. The CPO thereby is interpreted to have developed as a result of the deformation via DPC and grain boundary sliding.

(v) The deformation in the samples takes place under conditions of pronounced weakening in all cases. The weakening is induced by strain localization into shear bands that show strong grain size reduction. Grain size reduction, in turn, is due to nucleation of new phases, demonstrating the direct relationship between mineral reaction, grain size refinement, as well as the operation of DPC and grain boundary sliding (as grain size sensitive mechanisms), resulting in viscous deformation with low stress exponents.

Code and data availability. The MATLAB code for analysing amphibole reaction corona thicknesses is available from the author upon request (sina.marti@ed.ac.uk). Mechanical data and chemical analyses are available from the author upon request.

Appendix A: Methods

A1 Sample preparation

Maryland Diabase rock powder is fabricated by crushing Maryland Diabase pieces with a hand-press and subsequently with an alumina hand-mortar. The resulting powder is dry-sieved to extract a grain size fraction $\leq 125~\mu$m. The plagioclase in Maryland Diabase shows a relatively homogeneous composition ($\sim$ An65-70) except for a thin rim with lower anorthite component ($\sim$ An50-55). The core to rim area ratio is 83 : 17 ($\pm$ 3). Some of the clinopyroxene grains show a Mg-enriched core and clinopyroxene grains generally show orthopyroxene exsolution lamellae.

The diopside and enstatite material is a mineral powder provided by Jacques Precigout (University d'Orléans) and Holger Stünitz (University of Tromsø, University d'Orléans) with grain sizes of 40 - 125 μm for Cranberry Lake diopside, 40 - 180 μm for Damaping enstatite. Damaping enstatite and diopside are derived from a peridotite xenolith, the Cranberry Lake diopside from a calc-silicate rock. The albite material is extracted from an albite-quartz vein formed along a joint from the Alpe Rischuna area, Switzerland. Sonora labradorite are labradorite megacrysts formed in basaltic deposits from the Pinacate volcanic field, Sonora, Mexico. From Sonora labradorite, Alpe Rischuna albite and Damaping diopside a powder (grain size fraction $\leq 125~\mu$m) is produced in the same manner as described for the Maryland Diabase powder. As the Sonora labradorite material shows some accessory calcite, the powder is cleaned with HCl_{aq} (10%). Subsequently, the powder is placed in a funnel with a grade 602 h qualitative filter paper with a pore size of 2 μm and rinsed thoroughly with distilled water. The powder retained by the filter is then dried in an oven at $\sim 110~°$C. After this treatment, no calcite is detected in the material.

Synthetic plagioclase-pyroxene powders are mixed with a phase distribution of $\sim$ 57 vol% plagioclase to 43 vol% pyroxene. To produce the synthetic mixtures, the powders are placed in a 5 ml glass beaker with acetone and mixed using an ultrasonic stirrer (procedure of de Ronde et al., 2004). When most of the acetone is evaporated, the slurry is dried in an oven at 110 °C. This procedure prevents grain size and density sorting of the minerals.

The sample is then placed in a platinum jacket (0.15 mm wall thickness) with a 0.025 mm nickel foil insert. The Pt-jacket is weld-sealed with a Lampert welding apparatus while the sample is encased in a cooled brass piece (T $\sim$ 4 °C) to minimize sample heating and resulting potential water loss.

Solid sodium chloride (NaCl) is used as confining medium (Appendix Figure A.1a). K-Type thermocouples (with metal tubing) positioned next to the sample are used for most experiments. Only for long-duration experiments of more than 6 days, S-type thermocouples (with mullite tubing) are used, as the mullite tubing is more durable in the corrosive environment of the heated salt.

A1 Experimental procedure

Confining pressure, axial load, and displacement are constantly recorded. The force on the load-piston is measured using an external load cell, whereas the displacement of the load-piston is measured with either a direct current displacement transducer (resolution $\sim$ 1 μm) (Rig 1) or a noise-less digital linear-transformation measurement system (resolution = 0.1 μm) (Rig 2). Pc is measured via the oil pressure in the hydraulic pumping system and T is monitored and regulated to within $\pm$ 1 °C via an Eurotherm controller.

To bring the sample to the desired Pc-T conditions during pressurisation, the independently movable Pc- and load-pistons are alternately advanced (thereby raising the pressure), accompanied by stepwise increases of temperature until the desired Pc-T conditions are reached (Figure 2). The pressurization procedure usually takes 5 – 8 h. During the actual deformation experiment, only the load-piston is advanced. The experimental setup necessitates that each deformation experiment starts with a so-called 'lead run-in', where the load-piston is advanced through a thin ($\sim$ 1.5 mm) top lead layer. During this stage, the sample is expected to experience $\sim$ isotropic pressure. During the initial lead run-in, the sample is under approximately hydrostatic conditions for several hours (usually between 24 - 30 h). Once the hit point is reached, sample loading initiates. At the end of an experiment, the sample is quenched to 200 °C within 2 min while simultaneously removing the differential load. Subsequently Pc, load and T are slowly and simultaneously reduced to room conditions within about 5 h.

A2 Evaluation of mechanical data

Axial displacement is corrected for apparatus stiffness. σ_3 is assumed to be equal to the confining pressure, Pc (Eq. (A1)). With increasing advancement of the load-piston, the pressure inside the vessel increases and Pc is corrected for this (see e.g., Richter et al., 2016). The differential stress, $\Delta\sigma$, acting on the sample is derived from the difference between the axial load (F) with reference to the load at the hit point (F0) (Eq. (A2)), and the cross-sectional area of the forcing block (31.47 mm^2)

$$\sigma_3 = Pc \tag{A1}$$

$$\Delta\sigma = (F - F_0)/31.47 mm^2 \tag{A2}$$

$\Delta\sigma$ in the sample is corrected for the decreasing overlap of the forcing blocks (see procedure described in Marti et al.,

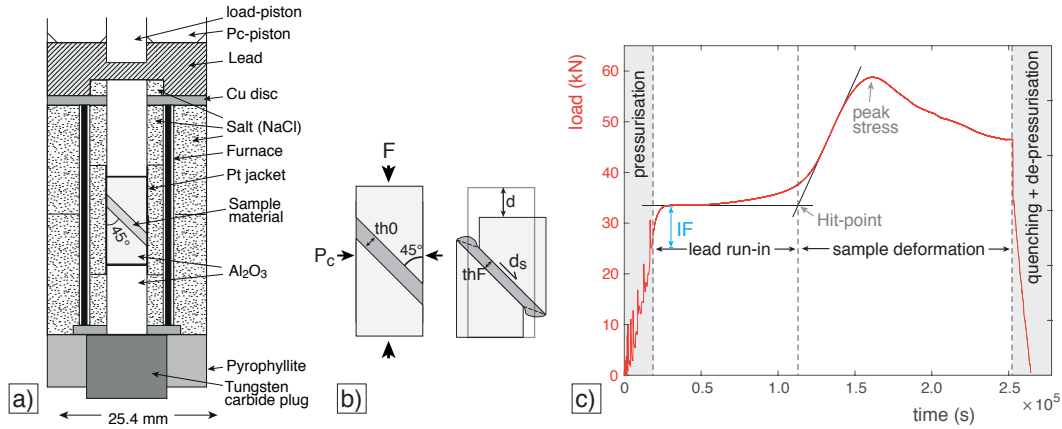



Figure A.1. General shear experiments. a) Sample assembly in cross-section. b) Schematic sample cross section at start of experiment (left) and after sample deformation (right). F = load induced by load-piston, Pc = confining pressure, d = axial displacement, ds = shear displacement, th0 = shear zone thickness at start, thF = shear zone thickness at end of experiment. c) Phases of an experiment, red line = load F(t). During 'pressurization', the sample is brought to the desired Pc-T conditions. Black dot denotes start of experiment. IF = initial load increase caused by machine friction. Phase 1: 'lead run-in', the sample not loaded. Phase 2: sample supports load and deforms. 'Hit-point' denotes onset of sample loading. During 'quenching + de-pressurization', the load is released and the temperature brought to ambient conditions.

2017). The shear and normal stresses, τ and σ_n supported by the sample inclined at 45° are obtained by Mohr circle construction from $\Delta\sigma$. The effective pore fluid pressures in our experiments is assumed to be negligible, i.e. is taken as zero and the deviation of σ_1 by 3-6° from 45° is neglected as well.

Stress exponent, *n*, is calculated for a relationship

$$\tau = \dot{\gamma}_a^{1/n} \tag{A3}$$

between shear stress, τ, and strain rate, $\dot{\gamma}_a$, (Eq. (A3)) using data of displacement-rate stepping and single-displacement rate experiments.

Over the years and between the different laboratories, different data treatment routines have been developed and are in use. The variations in calculated stresses using the different methods can influence determined stress exponents (*n*) from the data, causing variations on the order of 16 – 27 % on determined n values (see Marti et al., 2017)

A3   Strain and flow descriptors

The ISA and the finite stretching direction can be derived because the initial thickness of the shear zone is known (with an error of ± 0.03 mm) and the end-thickness can be measured after the experiment from the thin section. This uncertainty associated with the initial thickness leads to some uncertainty concerning the amount of thinning, and therefore also to a range of values for the ISA, the finite stretching direction and Wk.

A4   Microstructure analysis

- Data acquisition

SEM analyses are performed either with the Zeiss Merlin SEM at Tromsø University, or with a Philips XL30 ESEM at the Nano Imaging Lab of the Swiss Nanoscience Institute (SNI) at Basel University. Chemical analyses are performed using standardless energy dispersive X-ray Spectrometry (EDS), at 15 keV acceleration voltage with a ZAF matrix correction for spectra quantification.

(Scanning) transmission electron microscopy ((S)TEM) analyses are performed at Utrecht University using a FEI Talos 200FX equipped with a high-sensitivity Super-EDX system. TEM images are recorded in bright field (BF) mode, whereas STEM images in high angular annular dark field (HAADF) mode. BF-TEM images are highly sensitive on crystallographic orientation, whereas contrasts in HAADF-STEM images are sensitive to average atomic number (Z-contrast) of the material. Focussed ion beam (FIB) foils for TEM investigations are prepared using an FEI Helios NanoLab G3. The FIB foils are cut parallel to the shear direction and normal to the shear plane. Both, the FEI Talos 200FX and the Nanolab 3G are located at the Electron microscopy square at Utrecht University.

- EBSD analysis

For Electron backscatter diffraction (EBSD) measurements, thin sections were polished with colloidal silica suspension and subsequently coated with a thin layer of carbon. Samples were analysed in the Zeiss Merlin SEM at the University of Tromsø, with a Nordlys nano camera in high vacuum at 15 keV acceleration voltage and probe currents of $\sim$ 15

nA. Data is acquired with the Oxford AZtec software and processed with Channel 5 and the MATLAB toolbox MTEX (available at https://mtex-toolbox.github.io; Bachmann et al., 2010). Orientation data is analyzed and plotted using MTEX. The de la Vallée Poussin kernel with a half-width of $10°$ was used for pole figure contouring, whereas a kernel half-width of $12°$ was used for contouring of inverse pole figures (IPF). All pole figures and IPFs are calculated with Laue symmetry. Pole figure J-index (PFJ; e.g., Bunge, 1982; Mainprice and Silver, 1993) are given as a measure of texture strength. The index has a value of 1 for a random distribution and is infinite for a single orientation.

- Image analysis

Phase segmentation: Phase segmentation is performed on BSE SEM images, where individual minerals are identified by their Z-contrast. Using the software Fiji (link at: http://fiji.sc/Fiji) and the plugin Statistical Region Merger, automatic pre-segmentation was achieved. The automatic segmentation was then manually inspected and corrected where necessary.

Grain maps: Grain maps were produced by manually tracing (supervised segmentation) grains from SEM or TEM images. Grains were analyzed for their 2D area using Fiji, and for each grain, the diameter of the area equivalent circle, d_{equ}, is calculated.

$$d_{equ} = 2 \cdot \sqrt{(A/\pi)} \qquad (A4)$$

where A is the area of the digitized shape. Grain size distributions are presented as histograms of the 2D number weighted distribution of equivalent diameters.

Shear band orientation: Shear bands are traced and digitized on BSE SEM images. The x-y-coordinates of the outlines of the digitized structures are smoothed to remove pixel artefacts, and exported using Fiji. The smoothed x-y-coordinates are used as input for the SURFOR program (Panozzo Heilbronner, 1984; Heilbronner and Barrett, 2014), which determines the orientation distribution function (ODF) of the boundary segments. The ODFs are presented as rose diagrams.

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