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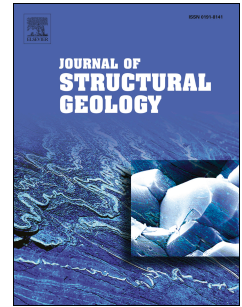


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1 *The role of deformation-reaction interactions to localize strain in polymineralic*
2 *rocks: insights from experimentally deformed plagioclase-pyroxene assemblages*

3

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19 localization.

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25 Abstract

26 In order to study the mutual effect of deformation and mineral reactions, we have conducted
27 shear experiments on fine-grained plagioclase-pyroxene assemblages in a Griggs-type solid-
28 medium deformation apparatus. Experiments were performed at a constant shear strain rate of
29 10^{-5} s^{-1} , a confining pressure of 1 GPa and temperatures of 800, 850 and 900 °C. While the
30 peak stress of plagioclase + orthopyroxene assemblages reaches values between those of the
31 end-member phases, the strength of polymineralic materials strongly decreases after peak
32 stress and reaches flow stresses that stabilize far below those of the weaker phase
33 (plagioclase). This weakening correlates with the coeval development of high-strain shear
34 zones where new phases are preferentially produced, including new pyroxene, plagioclase and
35 amphibole. The reaction products mostly occur as intimately mixed phases within fine-
36 grained and interconnected shear bands, together with different compositions with respect to
37 the starting material. This indicates that deformation significantly enhances the kinetics of
38 mineral reactions, which in turn strongly weaken the deforming sample, here attributed to a
39 switch to grain-size-sensitive diffusion creep through phase nucleation and grain size
40 reduction. Such an interplay between deformation and mineral reactions may have strong
41 implications for the initiation, development, and durability of shear zones in the lower crust.

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53 1. Introduction

54 Strain localization and resulting shear zones are considered to be fundamental features of
55 plate tectonics on Earth (e.g. Tackley, 1998; Bercovici and Ricard, 2012). They accommodate
56 a large amount of strain and have a direct control on rock rheology, so their development is
57 critical to understanding the dynamics of the lithosphere. The formation of viscous shear
58 zones has been considered to result from one or several processes of strain-induced
59 weakening, which expresses as a stress drop at constant strain rate or an increase of strain rate
60 at constant stress (e.g. Paterson, 2013). Possible weakening mechanisms include: (1)
61 Geometric and/or fabric softening, (2) a change in deformation mechanism, commonly due to
62 grain size reduction, (3) fracturing, (4) metamorphic mineral reactions, (5) shear heating (6)
63 water- or (7) melt-induced weakening (e.g. Poirier, 1980; White et al. 1980; Burlini and
64 Bruhn, 2005). While some of these mechanisms apply to monophase rocks, others are more
65 typical of polyphase materials.

66 As a starting point, laboratory-derived flow laws have been determined for the
67 deformation of monophase materials to understand the rheology of important rock-forming
68 minerals in the viscous regime of the lithosphere, including olivine (e.g., Chopra and
69 Paterson, 1981), quartz (Paterson and Luan, 1990), plagioclase (Rybacki and Dresen, 2000;
70 Dimanov et al., 1999) and pyroxene (Bystricky and Mackwell, 2001). For instance, the
71 experiments of, e.g., Rybacki and Dresen, (2000) and Chen et al., (2006), have shown that
72 water may considerably reduce the strength of feldspar and pyroxene aggregates. Plastic flow
73 and grain size reduction by dynamic recrystallization are also best described in deformation
74 experiments of monophase materials, giving rise to state-of-the-art flow laws (e.g. White et al.
75 1980, Schmid 1982, Rutter and Brodie, 1988). These experiments highlighted a potential
76 source of weakening induced by a transition from grain-size-insensitive to grain-size sensitive
77 creep, but this transition is not expected to cause long-term weakening in monophase
78 aggregates, because the efficient grain growth at elevated temperature is expected to
79 counteract the weakening effect of grain size reduction (e.g. Rutter and Brodie, 1988, De
80 Bresser et al., 2001).

81 Except for rare cases, the lithosphere consists of polyphase rocks. Thus, a growing body
82 of literature has also addressed the rheology of polyphase material, given the fact that such a
83 rheology is likely to be different from that of monophase ones (e.g. Bürgmann and Dresen,
84 2008). For instance, the presence of additional phases in polyphase aggregates inhibits grain
85 growth and controls microstructures through pinning (e.g. Olgaard and Evans, 1986; Herwegh

86 et al., 2011). The dominant deformation mechanism can thus be expected to differ as a
87 consequence of phase interactions.

88 In this context, the rheology of polyphase aggregates for gabbroic composition has a
89 fundamental importance for understanding the mechanical behaviour of the oceanic and lower
90 crusts; mafics are the most abundant rock types in these crustal layers (e.g. Weaver and
91 Tarney, 1984; Christensen and Mooney, 1995). However, experimental studies of high-
92 temperature deformation of gabbroic composition are still comparatively rare (e.g. Dimanov
93 et al., 2003, 2007; Dimanov and Dresen, 2005), and these studies have not considered mineral
94 reactions. They focused indeed on the role of secondary phases, grain size, water content,
95 stress and spatial distribution of grains to account for changing flow stress and dominant
96 deformation mechanism in polyphase feldspar-pyroxene aggregates. Yet, other studies
97 suggest that the occurrence of mineral reactions during viscous flow may have the potential to
98 weaken rocks and consequently localize strain (e.g. Stünitz and Tullis, 2001; De Ronde et al.,
99 2004, 2005; Getsinger and Hirth, 2014; Marti et al., 2017, 2018).

100 Strain localization and weakening in viscous shear zones as a result of mineral reactions
101 are mostly achieved through changes in P-T conditions (e.g. Gapais, 1989; Newman et al.,
102 1999) or through fluid-rock interactions (e.g. Austrheim, 1987; Menegon et al., 2015).
103 Changes in P-T conditions commonly occur during oceanic and continental subduction and
104 exhumation along crustal-scale shear zones, where the thermodynamic disequilibrium
105 promotes the growth of new stable minerals (e.g. Gerya et al., 2002; Jamtveit et al., 2016). In
106 addition, shear zones represent permeable pathways for fluids that enhance diffusion and
107 strongly catalyse mineral reactions under both, low (e.g. Fitz Gerald and Stünitz, 1993;
108 Newman and Mitra, 1993; Mansard et al., 2018) and high metamorphic grades (e.g. Brodie,
109 1980; Boundy et al., 1992; Glodny et al., 2003). The importance of mineral reactions lies in
110 the possible grain size reduction and change in deformation mechanism leading to a switch
111 from grain-size-insensitive to grain-size sensitive creep, giving rise to substantial weakening
112 (e.g. Etheridge and Wilkie, 1979; Olgaard, 1990; Stünitz and Fitz Gerald, 1993; Fliervoet et
113 al., 1997; De Bresser et al., 1998; Kruse and Stünitz, 1999; De Bresser et al., 2001;
114 Kenkmann and Dresen, 2002; Precigout et al., 2007; Raimbourg et al., 2008; Linckens et al.,
115 2011; Kilian et al., 2011; Viegas et al., 2016). Mineral reactions also contribute to form
116 mixing zones that play an important role of weakening because the pinning of grain
117 boundaries impedes grain growth and keep the grain size small (e.g. Etheridge and Wilkie,
118 1979; Herwegh et al., 2011).

119 In this contribution, rock deformation experiments on H₂O-added plagioclase-
120 orthopyroxene samples were performed to investigate the rheology and evolution of
121 microstructures with increasing strain. The experiments provide insight into weakening
122 mechanisms and localization of deformation during shear zone development. This study
123 presents a simplified model example of how deformation can facilitate metamorphic
124 reactions, heterogeneous nucleation, formation of fine-grained phase mixtures, and how
125 conversely such an evolution in microstructures eventually results in strain localization and
126 weakening of polyphase aggregates. The study also demonstrates the importance of
127 interaction of individual phases in polymineralic assemblages when dealing with the
128 behaviour of the lithosphere.

129

130 **2. Methods**

131 *2.1 Experimental procedure*

132 The shear deformation experiments were carried out in two Tullis-modified Griggs-
133 type deformation apparatus at the University of Tromsø (Norway) and in a new generation
134 Griggs rig at the University of Orléans (France). The following sections briefly describe the
135 preparation of the sample assembly and experimental procedure.

136

137 *2.1.1 Starting material and sample preparation*

138 Experiments were performed on two-phase plagioclase-orthopyroxene assemblages
139 and, for comparison, pure plagioclase and pure orthopyroxene (Opx) material. The starting
140 material was prepared from gem-quality Sonora labradorite (An₆₀), and Damaping opx from
141 peridotite (Opx: Wo₁-En₈₉-Fs₁₀). The initial mineral compositions are presented in Table 1.
142 The rocks were crushed using a hammer and sieved to sizes grains diameter between 100 to
143 200 µm. Minerals were pre-separated with a Frantz magnetic separator and hand-picked. They
144 were subsequently crushed into an alumina mortar, and then a powder ranging from 10 to 20
145 µm grain size was extracted by settling in a distilled water column. Opx and plagioclase
146 powders were mixed at a ratio of 50 : 50% (by volume) as a slurry in acetone using an
147 ultrasonic stirrer to avoid density/grain size separation (De Ronde et al., 2004, 2005). (*insert*
148 *Table 1*).

149 The mixed powder was placed between 45° pre-cut alumina forcing blocks (Fig. 1).
150 An amount of ~0.12 g powder for 6.33 mm diameter samples (at Tromsø University) or 0.25
151 g for 8 mm diameter samples (at Orléans University) were used with 0.1 wt. % H₂O added to
152 all samples, resulting in a shear zone of ~1 mm thickness. A nickel foil of 0.025 mm thickness
153 was wrapped around the pistons and all inserted into a weld-sealed platinum jacket. NaCl was
154 used as solid confining medium and the temperature was controlled by a S-type (Pt/Pt-Rh)
155 thermocouple. Detailed descriptions of the sample assembly in the Griggs-type deformation
156 apparatus are given in Pec et al., 2012, Marti et al., 2017 and Précigout et al., 2018. (*insert*
157 *Fig. 1*).

158

159 2.1.2 Deformation experiments

160 Samples were deformed at constant shear strain rate of $\sim 10^{-5} \text{ s}^{-1}$ to varying amounts of
161 shear strain (see Table 2 for a summary of experimental conditions). Pressures and
162 temperatures were increased alternately over several hours to achieve the target values of
163 800, 850, and 900°C and confining pressures of 1.0 GPa (Précigout et al., 2018). At the
164 desired P-T conditions, a period of hydrostatic hot-pressing was applied. The deformation part
165 of the experiment was started by advancing the deformation piston first through the lead piece
166 and then touching the upper forcing block (hit point), indicating the starting point of sample
167 deformation. The displacement of the deformation piston within the lead piece is
168 characterized by a slow increase of differential stress ('run-in' curve; Précigout et al., 2018),
169 the duration of which may vary depending on the experiment. We thus separated the
170 experiments with a relatively short (< 70h) and long (> 70h) "run-in" period. Following this
171 period, samples were deformed to shear strains of up to $\gamma = 8$ (high shear strain experiments).
172 Several experiments were also carried out without deformation (hot-pressing experiments) or
173 with deformation only up to the peak stress at $\gamma \approx 1$ (peak stress experiments) in order to study
174 the early stages of deformation. The longer run-in periods cause reaction products to nucleate
175 before deformation starts, whereas shorter run-in periods cause reaction products to form only
176 during deformation, allowing to study the relationship between the amount of reaction
177 products over time and the effect of deformation. Experiments reaching similar shear strain
178 with different total durations can be compared (*insert Table 2*).

179 When deformation was stopped, samples were quenched to 200°C within minutes
180 (temperature drop of ~150 to 300 °C/min) to preserve the deformation microstructures.
181 Subsequently, the force and confining pressure were decreased simultaneously to room

182 pressure and room temperature conditions. During initial stages of the decompression, the
183 differential stress is kept above the confining pressure (~ 100 to 200 MPa) to prevent the
184 formation of unloading cracks. After the experiment, the samples were impregnated with
185 epoxy resin and sectioned along the piston axis and parallel to the shear direction to produce
186 thin sections.

187 Experimental data were digitally recorded using the Catman Easy data acquisition
188 system and processed using a MATLAB-based program inspired from the “rig” program of
189 Matej Pec (Pec et al., 2016) and available at
190 <https://sites.google.com/site/jacquesprecigout/telechargements-downloads>. The hit point was
191 defined by curve fitting and the stress-strain curves of the deformed samples were then
192 generated by applying corrections on the displacement and force curves considering the rig
193 stiffness and friction of the apparatus, respectively. Furthermore, the sample compaction and
194 surface change (pistons overlap) were corrected to the displacement over the whole period of
195 sample deformation. The stress correction of Holyoke and Kronenberg (2010) was not
196 applied.

197

198 2.2 Analytical procedure

199 After the experiment, samples were cut along the piston axis in the plane of maximum
200 displacement and vacuum-impregnated with low viscosity epoxy to prepare thin sections for
201 microstructural analysis. The samples were mainly analysed using light and scanning electron
202 microscopy (SEM – TESCAN MIRA 3 XMU) at the ISTO-BRGM (Orléans, France).
203 Starting mineral compositions were determined using electron microprobe CAMECA SX
204 Five (EPMA) at the ISTO-BRGM (Orléans, France) on carbon-coated (20 nm thickness) thin
205 sections at 12 or 15 kV and a beam size of ca. 1 μm . We have analysed the core and rim
206 composition of plagioclase and pyroxene grains in deformed assemblages by EDS. In the
207 mixed zones, the plagioclase-pyroxene compositions were also analysed if the grain size was
208 sufficiently large. The amphibole composition is based on composition of large grains in low-
209 strain zones, because in high-strain zones, individual grains are typically too small to be
210 analysed. Representative analyses of these chemical compositions are given in Table 3. (*insert*
211 *Table 3*)

212

213 2.3 Microstructural analysis

214 Different mineral grains were identified in backscatter electron (BSE) images. Grain
215 shape preferred orientations (SPO) were analysed from manually produced bitmap images
216 using the autocorrelation function (ACF; Panozzo, 1983; Heilbronner, 2002). The ACF was
217 calculated for both, all combined phases (bulk ACF) and each individual phase (local ACF).
218 Because individual grain boundaries are difficult to distinguish (different from phase
219 boundaries which are obvious in BSE images), the ACF is used because it does not require
220 segmentation of the individual grains. This method thus avoids errors and biases caused by
221 the identification of individual grains. For further details about equations related to the ACF,
222 the reader is invited to consult the original study of Heilbronner (1992).

223 Scanning electron microscope/backscattered electron (SEM/BSE) images were used to
224 produce manually digitized phase maps, which allowed the analysis of grain size. The grain
225 size was defined as the equivalent circular area diameter ($d_{\text{equ}} = 2 \times \sqrt{\frac{\text{area}}{\pi}}$) (Heilbronner and
226 Barrett, 2014), and measured using the public domain software ImageJ
227 (<http://rsb.info.nih.gov/ij/>).

228

229 3. Results

230 3.1. Mechanical data

231 The terminology used to describe stress-strain curves is explained in Fig. 2a. While the
232 same colour code is used for different temperatures, a cross on the stress-strain-curve
233 indicates when the forcing blocks started to slip at the sample interface.

234

235 3.1.1 Pure plagioclase and pyroxene samples

236 The pure plagioclase experiments at 800 °C and 900 °C show similar types of stress-
237 strain curves. At 800 °C the sample shows more than 200 MPa higher flow stress than the
238 sample deformed at 900 °C (Fig. 2b), and both samples deform at stresses below the Goetze
239 criterion. The Goetze criterion ($\Delta\sigma \leq P_{\text{conf}}$) is used as an empirical criterion to delineate stress
240 conditions where rocks deform plastically (Kohlstedt et al., 1995). In both experiments, the
241 differential stress slightly decreases after reaching a peak stress at $\gamma < 1$ (Fig. 2b). The pure

242 pyroxene experiment was performed on a larger initial grain size fraction (powder sieved to
243 extract a grain fraction $\leq 40 \mu\text{m}$) than that of the other experiments (10 to 20 μm). The pure
244 orthopyroxene is very strong at 900 °C, reaching a peak stress of $\sim 1600 \text{ MPa}$ at $\gamma \sim 0.6$, well
245 above the Goetze criterion (Fig. 2b). The experiment was stopped during the stress drop as
246 only brittle deformation was expected to occur at these high differential stresses.

247

248

249

250 3.1.2 Plagioclase -orthopyroxene mixtures

251 The experiments on phase mixtures can be divided into two different series depending
252 on the time spent at P-T conditions before the contact between the σ_1 piston and top alumina
253 piston (hit point). After a short “run-in” period ($< 70\text{h}$ at temperature and pressure conditions
254 before hit point), the orthopyroxene + plagioclase (Opx + Plag) mixtures are very strong at
255 800 °C (559NM) and 850 °C (557NM), reaching a peak stress of $\sim 1100 \text{ MPa}$ at $\gamma \sim 0.3$ (Fig.
256 2c). Slip at one of the sample/forcing block interfaces causes $\Delta\sigma$ to drop far below the Goetze
257 criterion. The samples deformed at 900°C show peak stress values close to those at 800 and
258 850 °C, but at higher shear strain ($\gamma \sim 1.5$). At 900 °C, there is a gradual and pronounced
259 weakening after peak stress. In one case, slip occurred at the forcing block interface (Fig. 2c),
260 and the differential stress decreases below the Goetze criterion before stabilizing around 800
261 MPa (OR49NM). In the other case, the sample weakens continuously with a reduction of
262 $\sim 50\%$ in differential stress (Fig. 2c), until reaching a quasi-steady-state shear stress around
263 550 MPa near $\gamma \sim 6.5$ (OR41NM). (*insert Fig. 2*).

264 After a longer “run-in” period at 800 °C (Fig. 2d; $> 70\text{h}$ at temperature and pressure
265 conditions before the hit point), a stress drop occurs at a peak stress value near $\gamma \sim 0.7$ (above
266 the Goetze criterion at $\sim 1250 \text{ MPa}$; Fig. 2d), probably caused by slip at one sample-forcing
267 block interface (OR24NM). In contrast, the other samples at 850 °C (OR38NM) and 900 °C
268 (OR34NM) weaken continuously after peak stress, and then approach a quasi-steady-state
269 shear stress at $\gamma \sim 7.8$ and $\gamma \sim 6$, respectively (Fig. 2d). In these last two experiments, the
270 weakening is very pronounced with $\sim 64\%$ of weakening for the 850 °C sample and $\sim 78\%$
271 for the 900 °C sample compared to peak stress values. The Opx + Plag mixtures show a
272 slightly less steep loading curve compared to shorter run-in period experiments (Fig. 2d),

273 indicating a lower strength of the samples (the loading curve in the solid-medium apparatus at
274 high temperatures is not a purely elastic, but involves a component of permanent sample
275 strain; Richter et al. 2018).

276 These sets of experiments (Fig. 2) demonstrate that pure phase samples are either very
277 strong and deform only in the brittle field (Opx), or relatively weak and deform plastically
278 (Plag) at a moderate to low flow stress without significant weakening after peak stress. The
279 initial strength of mixed phase samples (peak stress) lies between the two extreme values of
280 the pure phase samples, but the mixed phase samples weaken after peak stress and give rise to
281 a final strength far weaker than the weakest of our mono-phase samples (i.e., pure
282 plagioclase).

283 *3.2 Microfabrics and composition*

284 *3.2.1 Pure end-member experiments*

285 The pure Plag samples deformed at 800 °C and 900 °C are microstructurally similar to
286 one another and show homogeneous deformation. Although it is difficult to distinguish all
287 individual plagioclase grains in BSE images, there is a clear plagioclase grain size refinement
288 locally (Fig. 3a). Recrystallized grains do not show different chemical composition with
289 respect to the relict grains. While our experiments are stronger than those carried out by
290 Stünitz and Tullis (2001) (~550 MPa compared to ~360 MPa, but the grain size ranges
291 between 6 and 10 µm in Stünitz and Tullis (2001); it was between 10 and 20µm in this study),
292 the microstructures are similar and no reaction products are formed at the P-T conditions
293 imposed.

294 The pure Opx sample with larger initial grain size (≤ 40 µm compared to 10-20 µm of
295 all other samples) deformed at 900 °C shows extensive fracturing, indicative of brittle
296 deformation (Fig. 3b). The pervasive fractures result in a locally dramatically reduced
297 pyroxene grain size (down to < 1 µm; Fig. 3c). No indication for plastic deformation was
298 detected. (*insert Fig. 3*).

299

300 *3.2.2 Mixed phase samples*

301 *3.2.2.1 High shear strain samples*

302 *3.2.2.1.1 General features*

303 At 850 °C, the high-strain sample is characterized by a single high-strain zone that
304 traverses the sample through the centre from one interface of the forcing block to the other
305 (Fig. 4). The reaction products appear pervasively in the whole sample, but the high-strain
306 zone contains considerably more reaction products (~80%) than the low-strain one (~28%)
307 (determined from BSE images). The grain size of the reaction products is substantially
308 reduced in the high-strain zone with respect to the low-strain regions, where the reaction
309 products develop as coronas at the Opx₁-Plag₁ boundaries or as monophasic aggregates
310 without the development of mixed phase zones (Fig. 5a). The reaction products are identified
311 as newly-formed pyroxene (Opx₂), plagioclase (Plag₂) and amphibole (Amph); all of which
312 were absent from the starting material. The original Opx₁ clasts are locally cut by brittle
313 fractures, reducing the grain size slightly (Fig. 5b), and the fractures are filled with Opx₂
314 reaction products. The transition between low- and high-strain zones can locally be gradual
315 and shows the incipient mixing of phases at the edges of original Opx₁ (Fig. 5c-d). The
316 reaction progressively consumes the pre-existing Opx₁ and induces the development of σ -tails
317 parallel to the shear direction. These tails locally coalesce and form interconnected shear
318 bands of fine-grained reaction products composed of Opx₂, Plag₂ and locally subordinate
319 Amph, which usually have rounded grain shapes (Fig. 5c-d). (*insert Fig. 4*).

320

321 3.2.2.1.2 High-strain zones

322 Reactions in high-strain zones result in intense grain size reduction and in coalescence
323 of foliation-forming aggregates of fine grains into mixed phase shear bands, usually laterally
324 connected (Fig. 5e-f, 6). Some of the shear bands have a synthetic orientation with respect to
325 the bulk shear zone (C'-type orientation in the sense of Berthé et al., 1979), many of them are
326 parallel to the shear direction (C-type orientation). At 850 °C, the majority of shear bands are
327 organized within high-strain zones of ~250-300 μm thick (Fig. 6a-b). Similar features are
328 shared at 900 °C, although deformation is more distributed (Fig. 6c-d-e). (*insert Fig. 5*).

329 The modal proportion of the reaction products reaches locally 80% in the high-strain
330 zones, as these products replace most of the pre-existing large grains, including the original
331 Plag₁ (Fig. 7a). Thus, most of the fine-grained mixed phase shear bands are composed of
332 reaction products (Opx₂, Plag₂ and Amph with minor amount of quartz (Qtz)). The high-strain
333 zones still comprise some original Opx₁ clasts that are embedded in the fine-grained reaction
334 products (Fig. 5e), but these clasts are reduced in size (from reaction) and appear less

335 elongated compared to those of the low-strain zones (Fig. 7b). The Opx_1 appears therefore as
336 a mechanically rigid clast within weaker mixed zones of reaction products, the grain size of
337 which is typically below one micron. (*insert Fig. 6*).

338 The strain is semi-quantitatively estimated from the aspect ratio of the bulk fabric
339 determined by the ACF and angle of the long axis of the bulk ACF with respect to the shear
340 plane (Fig. 7). It should be noted that this method of strain estimate is not an exact strain
341 analysis, given the absence of passive markers or rigid particles with a known orientation
342 before deformation (see also discussion in De Ronde et al. 2004). There is a gradient in the
343 aspect ratio and the orientation of reaction products between low- and high-strain zones. The
344 bulk aspect ratio is higher in the high-strain zones, where the amount of reaction products is
345 the most abundant (Fig. 7). Moreover, the bulk aspect ratio is around one and a half times
346 higher in the high-strain zones compared to the low-strain ones. The reaction products in the
347 high strain zones are strongly oriented subparallel to the shear plane (piston interface) (Fig.
348 7d; α between $\sim 3^\circ$ and 5°). In the low strain zones, the angle between the long axis of the
349 fabric of reaction products and the shear plane is higher (Fig. 7d; $\alpha \sim 9^\circ$). By applying the
350 equation that relates the preferred orientation of passive lines with respect to the shear plane
351 ($\gamma = 2 / \tan 2\theta$; Ramsay, 1980), we can estimate that local shear strain reaches $\gamma \sim 11$ to 16 in
352 the high-strain zones and $\gamma \sim 6$ in the low-strain ones. (*insert Fig. 7*).

353

354 3.2.1.2.3 Mineral chemistry

355 Overall, major compositional changes in plagioclase and opx occur together with grain
356 size reduction in the shear zones (Fig. 8a-b). The chemical composition of new grains of
357 plagioclase and opx distinctly differs from that of relict clasts. While the chemical
358 composition of clasts is mostly An59, the new reaction rim (Plag_2) is more albitic (An52; Fig.
359 8a). The reaction also results in almost complete disappearance of Plag_1 in the high-strain
360 zones (Fig. 7a), and within mixed phase zones and shear bands, the anorthite component of
361 fine-grained Plag_2 is slightly lower than that of new rims (An49).

362 The chemical composition of clasts (Opx_1) varies from En(86) to En(90), while the
363 rim composition (Opx_2) shows a decreasing enstatite content down to En(82) (Fig. 8b). The
364 composition of fine-grained new Opx_2 in the high-strain zone decreases even more to En(79).
365 The reaction products are thus characterized by an enrichment in iron content, together with
366 the formation of new amphibole grains enriched in Mg and depleted in Fe. New amphibole

367 grains appears in both, more and less deformed zones, but they are more abundant in fine-
368 grained shear bands and in the sample deformed at 850 °C (OR38NM – strain is highly
369 localized in this sample). Based on the composition of larger reaction products in the low-
370 strain zones – the grain size is too small in shear bands of the high-strain zones to measure the
371 composition of individual grains – amphiboles are classified as magnesio-hornblende and
372 tschermakite (Fig. 8c). (*insert Fig. 8*).

373

374 3.2.2 Hot-pressing and peak stress experiments

375 For comparisons with deformed samples, some samples have been hot-pressed
376 (without deformation) as long as the experiment duration of the high shear strain samples. At
377 900 °C, hot-pressing samples of Opx + Plag show the development of reaction products (Fig.
378 9a-b) with the same composition of Opx₂, Plag₂ and Amph as the deformed samples. The
379 reaction products occur as thin coronas at the Opx₁-Plag₁ phase boundaries or in cracks. The
380 volume of reaction products reaches about 3% in the sample held for 100h and about 10% in
381 the one held at the same pressure and temperature for 193h. The rims grow concentrically
382 around old grain relicts in hot-pressed samples and therefore do not nucleate in specific
383 locations at plagioclase-pyroxene phase boundaries (Fig. 9a). However, in the sample held at
384 pressure and temperature for 193h, the rims are slightly thicker and locally some aggregates
385 of original Opx₁ are replaced by Opx₂ (Fig. 9c).

386 In samples where the deformation was stopped at peak stress (OR47NM and
387 OR51NM), the microstructures differ slightly in the amount of reaction products and their
388 arrangement with respect to the microstructures of the hot-pressing experiments (Fig. 9d-e).
389 Indeed, the amount of reaction products slightly increase in peak stress experiments compared
390 to the hot-pressing ones. The reaction products are relatively homogeneously distributed in
391 the samples, although they start to coalesce locally to form partially connected aggregates
392 (Fig. 9e). The original Opx₁ and Plag₁ grains are also slightly elongated in the flow direction.
393 When these experiments reach relatively high conditions of differential stress, close to the
394 Goetze criterion, fractures locally affect the pyroxene and cause a slight reduction in grain
395 size. (*insert Fig. 9*).

396

397 3.2.3 Intermediate shear strain experiment

398 One sample was taken to a lower strain and is considered as a sample of intermediate
399 strain between peak stress and high shear strain (Fig. 2c; OR49NM; the distance between the
400 cross and the second cusp in the curve represents slip on the interface and thus a sample strain
401 of $\gamma \sim 5$ is achieved). This sample is characterized by the development of subparallel fine-
402 grained polyphase shear bands (Fig. 10a), both composed of an intimate mixture of fine-
403 grained Opx₂, Plag₂ and Amph (grain size $\ll 1 \mu\text{m}$; Fig. 10b). The shear bands originate from
404 tails that extend from the edges of original Opx₁ (Fig. 10c), and progressively coalesce to
405 form an interconnected network (Fig. 10a). (*insert Fig. 10*).

406

407 3.3 Grain size

408 The overall grain size for both plagioclase and opx grains is strongly reduced with
409 increasing strain. Most of the opx grain size reduction occurs after the peak stress, and hence,
410 during the weakening and development of fine-grained mixed phase zones (Fig. 11). While
411 the original opx grain size remains almost unchanged at peak stress with respect to the starting
412 material (cracking refines the grain size to a mode of $15.6 \mu\text{m}$), the size of opx grains in the
413 reaction products strongly decreases in mixed phase zones at high shear strain (mode of
414 distribution = $0.2 \mu\text{m}$). To ensure accurate grain size determination, we have excluded
415 plagioclase from the measurements because of the difficulty to distinguish individual grains in
416 plagioclase aggregates. Despite this issue, visual inspection suggests that the plagioclase grain
417 size in stable fine-grained mixtures is similar to that of opx. (*insert Fig. 11*).

418

419 3.4 Reaction progress

420 The set of experiments performed at different durations and $900 \text{ }^\circ\text{C}$ is used to illustrate
421 the relation between the volume fraction of reaction products with time and the effect of
422 deformation on reaction (Fig. 12). The volume fraction of reaction products increases from
423 $\sim 3\%$ for 100h to $\sim 10\%$ at 193h if deformation is not applied (OR55NM and OR43NM) (Fig.
424 12a). In contrast, even after a short period of deformation to small strain at peak stress, the
425 volume fraction of reaction products increases to $\sim 11\%$ (OR47NM) and $\sim 18\%$ (OR51NM)
426 (Fig. 12a). This amount of reaction products is higher than the amount documented for the
427 hot-pressing experiment with equivalent duration. With increasing strain, the amount of

428 reaction products also increases and reaches 26% (OR49NM), 35% (OR41NM) and 43%
429 (OR34NM) (Fig. 12a). (*insert Fig. 12*).

430

431 **4. Discussion**

432 *4.1 Mechanical data – strength of single- and two-phased assemblages*

433 The mechanical data of our experiments show a systematic difference in the
434 rheological evolution between monomineralic samples (Opx or Plag) and phase mixtures (Fig.
435 2). The monomineralic samples either deform by brittle mechanisms only (Opx; the
436 experiment was stopped before failing completely), or they deform viscously at steady state
437 stresses following a weakening of less than 150 MPa (Plag). The deformation of pure Opx
438 also gives rise to high differential stress at 900 °C ($\Delta\sigma \sim 1600$ MPa; Fig. 2b). Advances in
439 rock deformation studies provided detailed documentation about the creep behaviour of
440 pyroxene (e.g. Bystricky and Mackwell, 2001; Dimanov et al., 2003, 2005; Chen et al., 2006),
441 but mostly for Cpx; the mechanical data for Opx is indeed limited and restricted to dry Opx
442 only (Bruijn and Skemer, 2014; Bystricky et al., 2016). Nevertheless, based on deformation
443 mechanism maps (Fig. 13), our deformation conditions involve a far higher strain rate – of
444 several orders of magnitude – than the one predicted by the dislocation creep flow law of
445 Bystricky et al. (2016). Even the weaker wet Cpx flow law of Dimanov and Dresen (2005)
446 still indicates strain rates that are one order of magnitude lower than our applied strain rates
447 (Fig. 13b). Thus, dislocation creep deformation of Opx is not expected to accommodate the
448 applied strain rates in our samples, in agreement with the observed brittle behaviour of the
449 pure Opx sample (Fig. 3b). For pure Plag, our flow stresses at the given strain rate for $\gamma > 2$ (=
450 steady state conditions) are in good agreement (Fig. 13c) with the flow law from Rybacki and
451 Dresen (2000) using the conversion between simple shear and coaxial strain rates of $\dot{\epsilon} = \dot{\gamma} / \sqrt{3}$
452 (Gerya, 2010; Tokle et al., 2019). Our results for pure phase Plag and Opx are thus consistent
453 with previous results found in the literature. (*insert Fig. 13*).

454 The mixed Opx + Plag samples deform under continuous weakening after attaining a
455 peak stress, the value of which is intermediate between the end-member strengths of Opx and
456 Plag, in agreement with a Reuss-Voigt- or Taylor-Sachs model of phase mixture (e.g.,
457 Dimanov and Dresen, 2005). However, the Opx in our case is stronger than Cpx and deforms
458 only by brittle processes, so that the peak strength of the 2-phase composite sample is
459 between the two end-members and near the Goetze criterion (Fig. 2). In addition, the

460 pronounced weakening after peak stress produces final flow stresses up to 800 MPa that are
461 far below the Goetze criterion ($\Delta\sigma \leq P_{\text{conf}}$), and below the end-member strength of the weaker
462 phase, i.e., plag. This suggests that the deformation mechanism of the phase mixture differs
463 from those of the end-members, because the strength of the composite should lie between the
464 end-member strengths of a Reuss-Voigt or Taylor-Sachs-model. As discussed below, such a
465 different rheological evolution may be explained by the progressive modification of the phase
466 assemblage and microstructure through chemical interactions, i.e., mineral reactions.

467

468 *4.2. Nucleation and grain size reduction*

469 From figures 4 to 7, the local zones of reaction products accommodate far more strain
470 than the relict phases Opx and Plag, so that the reaction products are responsible for the
471 mechanical weakening of the samples. In addition, the microstructures at peak stress
472 conditions mainly consist of coronas with only very little mixed phase material (Fig. 9d-e).
473 Hence, most of the phase mixing starts after peak stress. Reaction products have a very small
474 grain size of $\sim 0.2 \mu\text{m}$ (Fig. 11). As the intense grain size reduction and phase mixing both
475 start to appear after peak stress (Fig. 14), strain weakening and strain partitioning into the
476 high-strain zones (Fig. 6) likely commence as a consequence of these two processes.

477 There is no flow law for Plag + Opx mixtures, and composite flow laws only exist for
478 Cpx + Plag (Dimanov and Dresen, 2005). We thus plotted the grain size modes and means of
479 the fine-grained reaction products (Opx + Plag) against the bulk stresses of the related
480 experiment into the existing dry Opx, wet Cpx and wet Plag deformation mechanisms maps
481 for a semi-quantitative comparison. The grain sizes and stresses of Opx₂ in the high-strain
482 zones plot in the diffusion creep field (Fig. 13a-b). According to the Bruijn and Skemer
483 (2014) data, pure Opx would still deform at one order of magnitude lower strain rates than our
484 applied strain rates ($\sim 10^{-5} \text{ s}^{-1}$) in the diffusion creep regime (Fig. 13a). The Plag at the same
485 grain size and stresses plots at an order of magnitude higher strain rates than those applied in
486 our experiments (Fig. 13c). Because of strain partitioning, the expected strain rates in the
487 high-strain zones are higher than the bulk ones by a factor of 2 to 3. From the semi-
488 quantitative estimate of the deformation mechanism maps, the observed strain rates in the
489 diffusion creep field for the fine grained Plag - Opx phase mixtures are compatible with those
490 of the existing Cpx data and even slower than those for Plag from existing experimental data
491 (Fig. 13). Thus, accommodation of deformation by diffusion creep in fine grained reaction

492 products is roughly consistent with available rheological data for pyroxene and plagioclase for
493 this deformation mechanism.

494 Several processes are generally invoked to account for phase transformations that may
495 reduce grain size and promote phase mixing, influencing the rock strength. Some workers
496 proposed that melt reactions may strongly influence the mechanical behaviour of rocks of the
497 lower crust by inducing phase mixing and introducing a low viscosity melt in the system (e.g.
498 Rosenberg and Handy, 2005). In our case, the P-T conditions are definitely outside of the
499 melt-forming field, in agreement with the lack of melt in our samples. Another weakening
500 process may involve dynamic recrystallization in the regime 1 of Hirth and Tullis (1992), i.e.,
501 where dynamic recrystallization mostly occurs by bulging. However, dynamic
502 recrystallization does not produce phase mixing, and the pyroxene grain size in zones of
503 mixed phase reaction products falls far below the orthopyroxene piezometer (Fig. 13;
504 Linckens et al., 2014). Furthermore, the new Opx₂-grains have a different chemical
505 composition compared to the starting material (Fig. 8), which excludes fracturing as a source
506 of extensive grain size comminution (e.g. Ree et al., 2005; Park et al., 2006; Pec et al., 2012,
507 2016). In low-strain zones of samples that reach high differential stress conditions, discrete
508 fractures segment Opx₁ clasts, but the fragments have a much larger grain size than the fine
509 grains in high-strain zones. In contrast, phase nucleation as comminution process and a switch
510 to grain size sensitive diffusion creep has been described in previous studies (e.g. Kilian et al.,
511 2011; Herwegh et al., 2011; Wassmann and Stoeckhert, 2013; Hidas et al., 2016; Précigout
512 and Stünitz, 2016; Marti et al., 2018; Prigent et al., 2018). Such a phase nucleation accounts
513 for changing chemistry, phase mixing, and extensive grain size reduction in our experiments.
514 (*insert Fig. 14*).

515

516 *4.3 Mineral reaction and diffusion creep*

517 While the development of reaction coronas induces limited grain size reduction and
518 no phase mixing in low-strain zones (Fig. 5a-b, 14), the grain size reduction is much more
519 intense in high-strain zones, combined with phase mixing (Fig. 5e-f, 6, 14). At the margin of
520 high-strain zones, the early stages of phase mixing are preserved (Fig. 5c-d, 14). Fine-grained
521 shear bands composed of Opx₂, Plag₂ and subordinate Amph extend at the edges of Opx₁
522 clasts and progressively replace the original Plag₁ (Fig. 5c-d). The phase mixing starts at the
523 edges of the original Opx₁ that is gradually consumed by the reaction as evidenced by

524 irregular Opx₁ boundaries, where new grains nucleate along low-stress sites (Fig. 5c). These
525 microstructures suggest that diffusive mass transport takes place as part of the reaction.
526 Nucleation of new grains of strongly reduced size facilitate diffusion creep. Phase mixing
527 may stabilize the grain size by impeding grain growth. Previous studies also showed that
528 phase nucleation forming tails at the expense of clasts may increase the degree of mixing in
529 natural shear zones and experimental samples (e.g. Kruse and Stünitz, 1999; De Ronde et al.,
530 2004; 2005; Holyoke and Tullis, 2006a, 2006b, Kilian et al., 2011, Tasaka et al. 2017,
531 Mansard et al., 2018).

532 In our experiments, no grain growth is observed in fine-grained shear zones, even for
533 longer experiments. This indicates that the occurrence of phase mixing has probably caused
534 pinning of grain boundaries, impeding grain growth and preserving a fine grain size (e.g.
535 Fliervoet et al., 1997; Herwegh et al., 2011; Kilian et al., 2011, Platt, 2015; Tasaka et al.
536 2017). Together with our predictions based on deformation maps, these features suggest that
537 high-strain shear bands mostly deformed by diffusion creep. One important accommodation
538 process to consider in this regime is grain boundary sliding (GBS; e.g. Langdon, 2006). In a
539 reacting and simultaneously deforming mineral assemblage, GBS has a two-fold effect: (a) it
540 creates new contact surfaces for reaction and (b) it produces potential cavitation sites, where
541 new phases can nucleate (e.g., Kilian et al., 2011, Menegon et al., 2015, Précigout and
542 Stünitz, 2016; Précigout et al., 2017; 2019). In this way, phase mixing is promoted in
543 agreement with 1) the nucleation of fine grains (Fig. 11), 2) an intimate mixing of phases in
544 high-strain zones (Fig. 5e-f, 6), and 3) the fact that the grain size of pyroxene and plagioclase
545 falls within the diffusion creep field (Fig. 13). A switch of deformation mechanism to
546 diffusion creep accounts here for a significant weakening and related strain localization into
547 fine-grained high-strain zones, which hence constitute the weakest parts of the sample (e.g.
548 Schmid et al., 1980, Rutter and Brodie, 1988; Montési, 2007; Précigout et al., 2007;
549 Raimbourg et al., 2008; Gueydan et al., 2014).

550

551 *4.4 Geometrical aspects: connectivity of shear zones and weakening processes*

552 Previous works have shown that the rheological behaviour of rocks and strain
553 localization can be highly dependent on strength contrast between phases, spatial geometry
554 and amount of mineral reactions (e.g., Handy, 1990; Dell'Angelo and Tullis, 1996; Holyoke
555 and Tullis, 2006a; Hansen et al., 2012). Holyoke and Tullis (2006a) found in high-
556 temperature experiments a strong relation between the interconnection of weaker phases and

557 strain localization. It has also been shown by Pec et al., (2016) and Palazzin et al., (2018) that
558 the bulk strength of a given sample remains high if the weak shear bands are not, or poorly
559 connected. Numerical studies also support the idea that phase arrangement can dramatically
560 affect the bulk strength (e.g. Montési, 2007, Montési, 2013; Gerbi et al., 2016). In our study,
561 the fine-grained shear bands are highly connected and oriented sub-parallel to the shear plane,
562 suggesting that the reaction products connect in three dimensions and therefore control the
563 bulk strength of the sample. This arrangement is considered as a type of geometric weakening
564 (e.g. Handy, 1994; Dell'Angelo and Tullis, 1996; Holyoke and Tullis, 2006a; Gerbi et al.,
565 2016). In addition, reaction progress will introduce more of the weak fine-grained material,
566 increasing the probability of reaction products to connect after peak stress, and hence to
567 promote further weakening. Previous shear deformation studies also described the formation
568 of shear bands after peak stress (e.g. Holyoke and Tullis, 2006a; Pec et al., 2016; Marti et al.,
569 2018). However, unlike our study, they do not always show the coalescence of shear bands
570 into a connected high-strain zone that runs throughout the shear zone in high shear strain
571 experiments (Fig. 4).

572 To summarize, our results support the idea that the sample weakening is dependent on
573 two processes: (1) the introduction of fine-grained reaction products, and (2) their
574 connectivity, which increases with an increasing amount of reaction products. The
575 connectivity is nevertheless a complex variable which results from the interrelationship of
576 strain partitioning and reaction progress. In our case, the proportion and arrangement of weak
577 material with increasing strain first evolve into thin and polyphase shear bands, and then into
578 connected broader high-strain zones (Fig. 14). We therefore conclude that the weakening of
579 the samples is caused by the connectivity of the reaction products, giving rise to a bulk
580 weakening that only achieves when the weak shear bands are interconnected.

581

582 *4.5 Influence of deformation on reactions*

583 One effect of deformation to solid-state chemical reactions is that deformation may
584 enhance the kinetics of mineral reactions (e.g. Yund and Tullis, 1991; Wintsch et al., 1995;
585 Baxter and De Paolo, 2004; Imon et al., 2002; Yonkee et al., 2003; Holyoke and Tullis, 2006;
586 De Ronde and Stunitz, 2007, Richter et al., 2016). At the same time, mineral reactions
587 enhance the nucleation rate, giving rise to very small grain sizes that favour the dominance of
588 grain-size-sensitive creep (e.g. Rutter and Brodie, 1988; De Ronde et al., 2005; Marti et al.,

589 2018). In our high-strain samples, we document a strong partitioning of deformation into
590 high-strain zones (Fig. 4). The fact that the amount of reaction products increases with
591 increasing strain at 850 and 900°C (Fig. 7a) strongly suggests that reaction kinetics are
592 enhanced by deformation. Moreover, a higher amount of syn-kinematic amphiboles in high-
593 strain zones indicates that the deformation-reaction feedback is pronounced, particularly in
594 our “850 °C” sample. A similar feedback was found in sheared plagioclase-olivine
595 experiments at 900°C (De Ronde and Stünitz, 2004, 2007), and they showed that the local
596 strain is highly correlated with reaction progress. Recently, Marti et al., (2018) also described
597 the positive feedback between deformation and reaction progress in experimentally deformed
598 plagioclase-pyroxene mixtures, but in these examples, fewer reaction products have been
599 described, probably due to the lower temperatures of their experiments. In addition, the
600 greater reaction progress and larger amount of fine-grained mixed phase zones in our samples
601 may be attributed to the small grain size of our starting material in otherwise very similar
602 experimental procedures to Marti et al., (2018). The partially smaller grain size in our samples
603 provides indeed a greater surface area of phase boundaries as sites of potential reactions.

604 Evidence of mineral reactions enhanced by deformation also arises from comparison
605 between our hot-pressing, peak stress, and high-strain samples (Fig. 12a). The main difference
606 between these samples is the bulk amount of reaction products. While hot-pressing and peak-
607 stress samples both reveal a slight increase in the amount of reaction products with time (Fig.
608 12b), the high-strain samples produced far more reaction products for equivalent duration of
609 experiment (Fig. 12b). Although two data points for hot-pressing samples are probably not
610 enough to provide robust quantitative constraints, this reveals the strong influence of
611 deformation on the amount of reaction products (Fig. 12b).

612 Similar results have been documented in experimentally deformed fine-grained gneiss
613 (Holyoke and Tullis, 2006a; 2006b) and plagioclase + olivine samples (De Ronde and Stunitz,
614 2007). The main reason for the faster reaction progress in deformed samples is probably the
615 introduction of defects into the reactants, i.e. the increase of the activation energy for the
616 reaction at otherwise identical pressure and/or temperature overstepping conditions (De
617 Ronde and Stunitz, 2007). Furthermore, the exposure of new reaction surfaces by grain
618 boundary sliding can be another potentially important factor.

619

620 *4.6 Geological implications – shear localization in nature*

621 Several workers have pointed out that strain localization may control the degree of
622 weakening in polyphase rocks (e.g. White et al., 1980; Handy, 1989), which is of great
623 importance for the rheological behaviour of the lower crust. A large amount of fine-grained
624 weak zones indeed allows to preserve the weak long-term behaviour of deep crustal rocks. It
625 is also well established that the lower crust is lithologically heterogeneous, mainly consisting
626 of mafic rocks dominated by feldspar and pyroxene (e.g. Kirby and Kronenberg, 1987;
627 Christensen and Mooney, 1995; Rudnick and Fountain, 1995). However, current lithospheric
628 models do not take into account the complex interaction between plagioclase and pyroxene on
629 lower crust rheology. They do not either consider mechanical polyphase shear bands to
630 describe the lower crust, yet observed in natural shear zones (e.g. Kanagawa et al., 2008;
631 Kruse and Stünitz, 1999).

632 Mineral reaction and its interplay with deformation is of great importance for strain
633 localization (e.g. Kerrich et al., 1980; Brodie and Rutter, 1985; Handy and Stünitz, 2002;
634 Keller et al., 2004; Mansard et al., 2018). On one hand, cases of preserved metastable mineral
635 assemblages in undeformed rocks are well documented, while deformed equivalents of the
636 same rock body have significantly reacted (e.g. Austrheim and Griffin, 1985, Koons et al.,
637 1987, Früh-Green, 1994). On the other hand, the extent of metamorphic reactions is greater in
638 shear zones, as reaction may cause strain localization by producing a mechanically weak
639 aggregate (Keller et al., 2004). In our experiments, the starting material made of Opx + Plag
640 was deliberately chosen to be out of equilibrium at the experimental pressure and temperature
641 conditions. Although large overstepping of reaction boundary probably occurred with respect
642 to far slower changes of P-T conditions in nature, the experimental deformation of polyphase
643 mixtures is here a good analogue to natural shear zones involving strain-enhanced chemical
644 reactions and reaction-enhanced strain weakening.

645 In agreement with previous experiments (e.g. Rybacki and Dresen, 2000; Bystricky
646 and Mackwell, 2001; Dimanov et al., 2003), our findings confirms that Opx + Plag
647 assemblages are very strong, but as soon as these assemblages react, the material is viscously
648 deformed and substantially weakened. The deformation of Opx + Plag assemblages also
649 promoted diffusion-controlled chemical reactions, giving rise to the nucleation of new
650 intrinsically strong phases, i.e., plagioclase and pyroxene (Fig. 2a). However, these strong
651 phases nucleated as very fine grains within weak mixed phase zones (Fig. 6, 11), causing a
652 switch in deformation mechanism, and thereby extending the range of conditions where fine-
653 grained mixed zones are weaker than the unreacted assemblages. In addition, a critical

654 parameter for the weakening to be effective is the connectivity of small weak zones at large
655 scale. Our experiments demonstrate that the connectivity of weak material arises from
656 feedbacks between deformation and phase reactions, causing a significant drop of the bulk
657 sample strength, i.e., strain-induced weakening. The same effect may be therefore expected on
658 a larger scale of the lower crust if small shear zones highly connect each other.

659

660 **5. Conclusions**

661 In this study, we have performed rock deformation experiments on plagioclase +
662 pyroxene and pure end-member assemblages using the solid-medium Griggs-type apparatus at
663 a pressure of 1 GPa and temperatures of 800, 850 and 900°C. The analysis of pure end-
664 member assemblages reveals that 1) pure Opx deforms only by brittle mechanisms and 2)
665 pure Plag deforms plastically at steady-state stress following a limited weakening after peak
666 stress. In contrast, plagioclase + pyroxene assemblages show extensive strain-related
667 weakening caused by mineral reactions. At the onset of deformation, new phases nucleate in
668 aggregates as mixed phase tails and shear bands at the expense of original opx and
669 plagioclase. The change of phase composition together with phase mixing indicates that grain
670 size reduction originates from the consumption of reactants and nucleation of new phases. As
671 deformation and reaction progress, the thin shear bands then coalesce to form highly
672 connected material in the high-strain zones. Together with the occurrence of a significant
673 weakening coeval with grain size reduction and phase mixing, our predictions suggest that
674 shear bands deformed by grain-size-sensitive creep. The degree of connectivity, i.e. the
675 formation of connected reacted material to a large extent, also played a major role in
676 controlling the bulk sample strength. This highlights the importance of strain partitioning and
677 shear bands connection to weaken shear zones. Providing analogue documentation of natural
678 shear zones, our findings therefore emphasize the interactions between deformation and
679 reaction to localize strain in ductile shear zones, particularly in mafic rocks.

680

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690

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1047

1048

1049 **Figure Captions**

1050

1051 **Fig. 1.** Sample assembly of the shear experiments using a Griggs-type apparatus. **a)** Schematic cross-
1052 section of the assembly in a new generation Griggs rig at the University of Orléans (France). **b)**
1053 Schematic drawing of sample geometry in a 3D view. Sample powder is inserted between 45° pre-cut
1054 alumina forcing blocks. Modified after Tarantola et al., 2010. **c)** Sample geometry at the end of the
1055 experiment: th_0 = initial thickness, th_f = final thickness.

1056

1057 **Fig. 2.** Differential stress (MPa) versus shear strain (γ) plots. **a)** Set of terms used to describe the
1058 mechanical data. **b)** Stress-strain curves showing the mechanical behaviour of two pure plagioclase
1059 samples deformed at 800 and 900 °C. The mechanical curve of a pure pyroxene sample deformed at
1060 900 °C with an initial coarser grain size ($< 40 \mu\text{m}$) is also plotted. **c-d)** Set of experiments deforming
1061 an Opx + Plag mixture at different temperatures (800, 850 and 900°C), and at constant confining
1062 pressure, strain rate and water content. These experiments are separated in two graphs according to the
1063 different duration of the “run-in” section. The colour coding refers to the temperature of the
1064 experiments.

1065

1066 **Fig. 3.** SEM-BSE images representative of microstructures documented in pure plagioclase (**a**) and
1067 pyroxene (**b-c**) experiments. **a)** Plagioclase grain size refinement (blue arrows) by dynamic
1068 recrystallization. **b)** Fractures in pyroxene grains. **c)** Close-up of the pyroxene grains showing local
1069 extensive grain size refinement in cracks (orange arrows). pl = plagioclase.

1070

1071 **Fig. 4.** SEM-BSE images across of the high-shear-strain sample (**a**) and related phase map digitized
1072 manually (**b**), showing the overall strain gradient in the shear zone from top to bottom. This transect is
1073 characterized by a heterogeneity in the amount of reaction and deformation. rp = reaction products,
1074 opx = orthopyroxene, cpx = clinopyroxene, pl = plagioclase, amph = amphibole, alumina FB =
1075 alumina forcing block.

1076

1077 **Fig. 5.** SEM-BSE images representative of microstructures observed in the high-shear-strain
1078 experiments. **a-b**) The reaction products appear as coronas around the original Opx₁ and as aggregates
1079 in low-strain zones. Pyroxene is locally fractured and filled with reaction products (white arrows). **c-d**)
1080 The transition zone shows the development of fine-grained tails and shear bands extending at the
1081 edges of Opx₁. These tails form fine-grained mixed zones rich in Opx₂, Pl₂ and Amph. **e-f**) High-strain
1082 zones textures are made of intercalated fine-grained polyphase shear bands. In these zones, an
1083 extensive phase mixing between Opx₂, Pl₂ and Amph is observed. The original Pl₁ almost completely
1084 disappears, while some Opx₁ clasts, which are reduced in size, remains within these fine-grained
1085 mixed zones. opx = orthopyroxene, cpx = clinopyroxene, pl = plagioclase, amph = amphibole, qtz =
1086 quartz.

1087

1088 **Fig. 6.** SEM-BSE images representative of microstructures observed in the high-strain zones. Three
1089 samples deformed to high shear strain are documented: one deformed at 850°C (**a-b**) and two others
1090 deformed at 900°C (**c-d-e**). In each case, the high-strain zones are made of fine-grained polyphase
1091 shear bands, mainly composed of Opx₂, Pl₂ and Amph. However, the volume fraction of reaction
1092 products is larger at 850°C and the deformation is more localized compared to samples deformed at
1093 900°C, which show more distributed deformation and lower amounts of amphiboles at the scale of the
1094 shear zone. Opx = orthopyroxene, cpx = clinopyroxene, pl = plagioclase, amph = amphibole, qtz =
1095 quartz.

1096

1097 **Fig. 7.** Analysis of the phase content (a), aspect ratio (b-c) and shape preferred orientation (d) across
1098 the high-shear-strain sample shown in Figure 4. For reference, panel (a) shows the evolution of phases
1099 as volume fraction through the transect. Opx₂ and Amph as reaction products are grouped together.
1100 Panel (b) displays the bulk aspect ratio R*, while the panel (c) displays the aspect ratio R* for
1101 individual phases. Panel (d) shows the preferred orientation of angle α^* of the bulk fabric with respect
1102 to the piston interfaces. While the amount of reaction products is higher in the high-strain zone
1103 compared to low-strain ones, the ACF analysis reveals a higher aspect ratio of the fabric in the high-
1104 strain zones, essentially of the reaction products, which are preferentially oriented subparallel to the
1105 piston interfaces. Opx = orthopyroxene, cpx = clinopyroxene, pl = plagioclase, amph = amphibole.

1106

1107 **Fig. 8.** Chemical composition of plagioclase (a), pyroxene (b), and amphibole (c). The chemical
1108 compositions of plagioclase and pyroxene are divided into three subsets: clast-core, clast-rim and fine
1109 grains. The frequency corresponds to the number of grains analysed considering one point per grain.

1110

1111 **Fig. 9.** SEM-BSE images representative of microstructures observed in the hot-pressing experiments
1112 (a-b-c) and peak stress experiments (d-e). a-b) Thin reaction coronas that start forming at the Opx₁-Pl₁
1113 interphase boundaries. This consists essentially of Opx₂ and Plag₂. c) Aggregate of original Opx₁
1114 replaced by Opx₂. d) Representative microstructures observed at peak stress where the reaction
1115 products start to form aggregates that are partially connected in the direction of extension. e) Irregular
1116 grain boundaries composed of small grains of newly formed reaction products. Opx = orthopyroxene,
1117 cpx = clinopyroxene, pl = plagioclase, amph = amphibole.

1118

1119 **Fig. 10.** SEM-BSE images showing characteristic microstructures of the nascent mixing of phases. a)
1120 Incipient interconnection of thin shear bands filled with fine-grained polyphase mixture (yellow dotted
1121 outline) subparallel to the shear plane (horizontal direction). b) Close-up of shear bands showing fine-

1122 grained mixture of Opx₂, Pl₂ and Amph. c) Close-up of incipient mixing forming tails at the edges of
1123 original Opx₁. Opx = orthopyroxene, cpx = clinopyroxene, pl = plagioclase, amph = amphibole.

1124

1125 **Fig. 11.** Grain size evolution of pyroxene between peak-stress experiment (mode: 15.6 μm ; mean: 15.3
1126 μm) and the newly formed Opx₂ in fine-grained mixed zones in high-shear-strain experiments (mode:
1127 0.2 μm ; mean: 0.4 μm). The black curve represents a best-fit to the log-normal distribution. The
1128 average value for the aspect ratio of new Opx grains is $a/b = 1.23$.

1129

1130 **Fig. 12.** Evolution of the amount of reaction products as a function of time in plagioclase – pyroxene
1131 experiments at 900°C. Symbols denote types of experiments performed, which include hot-pressing,
1132 peak-stress, intermediate and high-shear-strain experiments. The graph in (a) shows the different types
1133 of experiments, strain rate and associated mechanical data in stress-strain graph. b) Evolution of the
1134 volume fraction of reaction products (RP) with time, and effect of deformation on reaction and sample
1135 strength. Avrami curve fitting for hot-pressed or deformed samples are colour coded. One curve for
1136 deformed samples is fitted for peak-stress samples, while the other is fitted for higher-shear-strain
1137 samples. The duration of the deformed experiments is taken from the hit-point.

1138

1139 **Fig. 13.** Deformation mechanism map for dry orthopyroxene (a), wet clinopyroxene (b) and wet
1140 plagioclase (c) at 900°C and 1 GPa. On these maps, we plot the differential stress and grain size of
1141 Opx at peak-stress conditions and in mixed zones of high-shear-strain experiments in both cases, as we
1142 consider that the grain size of plagioclase and pyroxene is similar. The deformation conditions of the
1143 pure Plag sample is also plotted. The grain size is represented in box-and-whisker diagram that
1144 includes the median (white lines), mean (green lines) and mode (blue lines). The flow laws are from
1145 Bruijn and Skemer, 2014 and Bystricky et al., 2016 for dry orthopyroxene, from Dimanov and Dresen

1146 (2005) for wet clinopyroxene and from Rybacki and Dresen (2000) for wet plagioclase. The grain size
1147 piezometer for Opx (yellow line) is taken from the study of Linckens et al. (2014).

1148

1149 **Fig. 14.** Schematic textural and microstructural evolution of plagioclase-pyroxene mixture from hot-
1150 pressing conditions (**A**) through peak-stress conditions (**B**) to strongly deformed shear zones (**C**). This
1151 figure illustrates the different stages of strain localization and weakening during deformation.

1152

1153 **Tables**

1154

1155 **Table. 1.** Composition of plagioclase (Plag) and orthopyroxene (Opx) as starting material.

1156

1157 **Table. 2.** Summary of experimental conditions. Type: HP - hot-pressing, PS: peak stress, D: deformed
1158 samples to varying amounts of shear strain. A cross marks is added to the type of deformation when
1159 the forcing blocks started to slip at the sample interface; τ_{peak} : differential stress at peak, τ_{flow} : steady-
1160 state differential stress, τ_{end} : differential stress at end of experiment, γ : shear strain, th_0 : thickness
1161 initial of the shear zone, th_f : final shear zone thickness.

1162

1163

1164 **Table. 3.** Representative chemical composition of plagioclase, orthopyroxene and amphibole.

1165

Table. 1.

	Sonora labradorite		Damaping Mg-Opx	
	Wt. % oxides	Atoms per 8 oxygen	Wt. % oxides	Atoms per 6 oxygen
SiO ₂	53,87	2,434	55,71	1,929
Al ₂ O ₃	29,41	1,566	3,81	0,155
CaO	11,68	0,565	0,42	0,016
Na ₂ O	4,06	0,356	0,07	0,005
K ₂ O	0,46	0,027	0,02	0,001
MgO	0,09	0,006	32,51	1,678
TiO ₂	0,08	0,003	0,07	0,002
FeO	0,38	0,014	7,21	0,209
MnO	0,05	0,002	0,18	0,005
Total	100,08	4,972	100,01	4,000
	An60		Wo1	
	Ab38		En88	
	Or2		Fs11	

Table. 2.

Exp. Nr	Material	Type	T	P	H ₂ O	τ_{peak}	τ_{flow}	τ_{end}	γ	th0	thf
			[°C]	[GPa]	μL	[MPa]	[MPa]	[MPa]		[mm]	[mm]
535NM	Plag	D	900	1	0,12	620	524	524	2,9	0,75	0,57
537NM	Opx	PS	900	1	0,12	1600	-	1546	0,9	0,75	0,69
557NM	Opx + Plag	PS ^x	850	1	0,12	1067	-	577	0,4	0,75	0,59
559NM	Opx + Plag	PS ^x	800	1	0,12	1111	-	350	0,5	0,75	0,67
OR24NM	Opx + Plag	D ^x	800	1	0,25	1280	-	866	3,1	1,1	0,87
OR34NM	Opx + Plag	D	900	1	0,25	781	114	126	7,6	1,1	0,68
OR38NM	Opx + Plag	D	850	1	0,25	1038	339	339	8,0	1,1	0,63
OR41NM	Opx + Plag	D	900	1	0,25	1094	542	542	7,0	1,1	0,72
OR43NM	Opx + Plag	HP	900	1	0,25	-	-	-	-	1,1	1,1
OR47NM	Opx + Plag	PS	900	1	0,25	989	-	989	0,6	1,1	0,91
OR49NM	Opx + Plag	D ^x	900	1	0,25	1111	800	800	6,0	1,1	0,73
OR51NM	Opx + Plag	PS	900	1	0,25	901	-	901	0,8	1,1	1
OR53NM	Plag	D	800	1	0,25	904	746	746	4,7	1,1	1
OR55NM	Opx + Plag	HP	900	1	0,25	-	-	-	-	1,1	1,1

Table 3.

	Plagioclase			Orthopyroxene			Amphibole
	Core	Rim	Fine-grains	Core	Rim	Fine-grains	
SiO ₂	54,70	55,35	55,10	55,48	55,57	55,77	49,82
Al ₂ O ₃	30,89	28,99	29,50	3,91	4,62	5,63	10,62
CaO	10,24	9,90	9,65	1,03	1,14	1,13	12,50
Na ₂ O	3,87	4,97	5,51	0,24	0,01	0,10	2,10
K ₂ O	0,14	0,28	0,25	0,03	0,02	0,03	0,00
MgO	0,07	0,22	0,04	32,04	30,29	27,90	19,22
TiO ₂	0,13	0,04	0,00	0,06	0,07	0,08	0,21
FeO	0,13	0,16	0,04	7,17	8,40	9,75	3,61
MnO	0,00	0,02	0,05	0,07	0,03	0,05	0,00
Total	100,17	99,93	100,14	100,03	100,14	100,43	98,08
	An59	An52	An49	Wo2	Wo2	Wo2	Magnesio-hornblende
	Ab40	Ab47	Ab50	En87	En84	En81	
	Or1	Or1	Or1	Fs11	Fs13	Fs16	

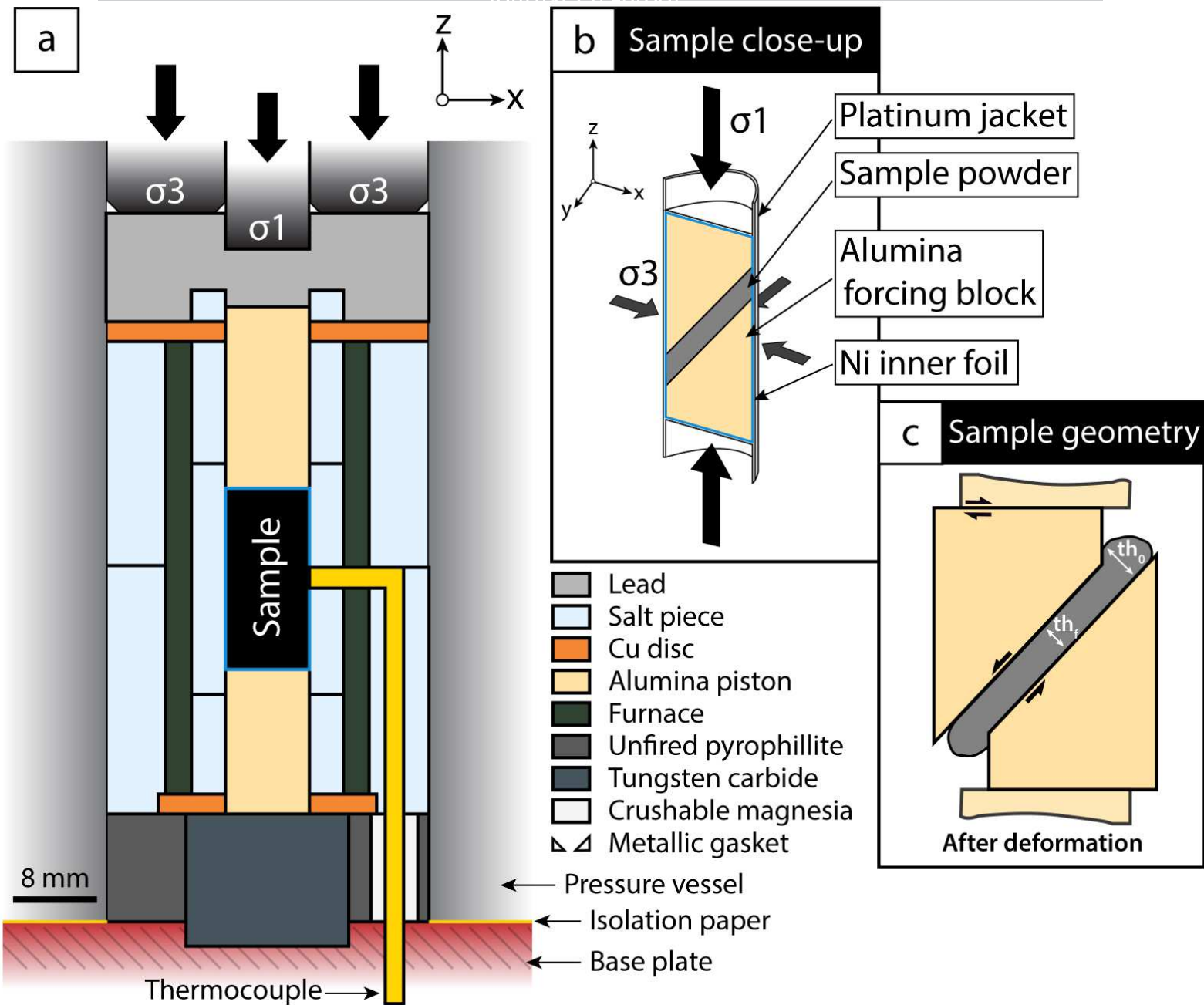


Fig. 1.

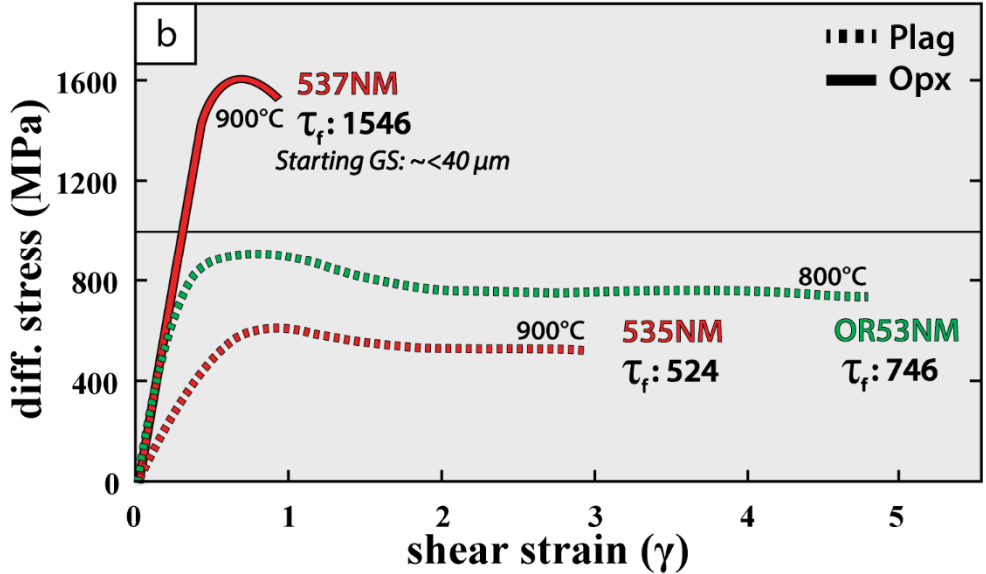
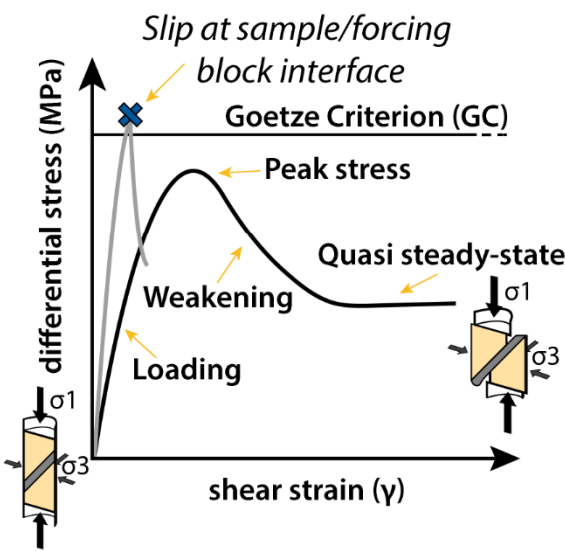
a

Conditions

Temperature: 800-850-900°C
 Pressure: 1 GPa
 Strain rate: 10^{-5} s^{-1}

Starting GS: 10-20 μm
 except for 537NM $\sim < 40 \mu\text{m}$

Pure Opx - Plag experiments



Mixture Opx + Plag experiments

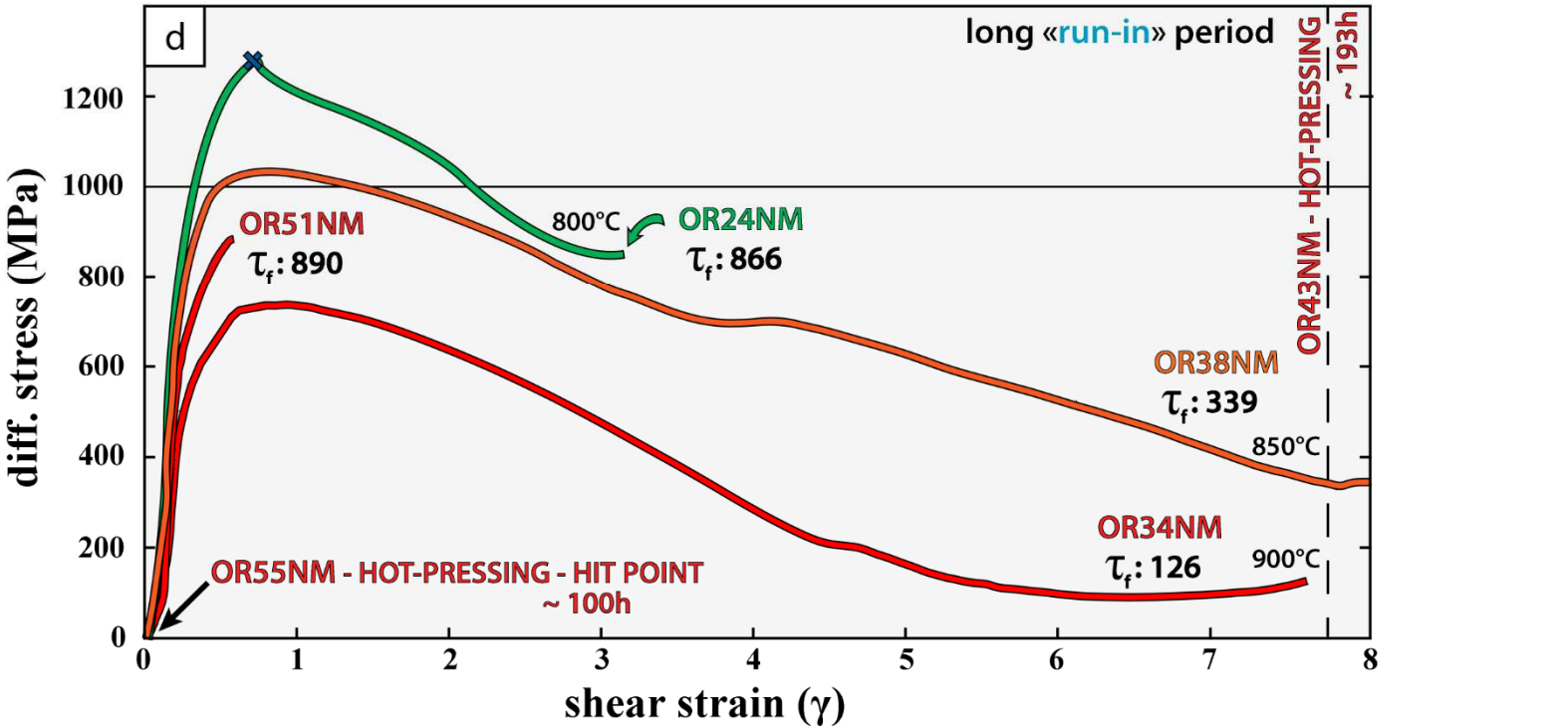
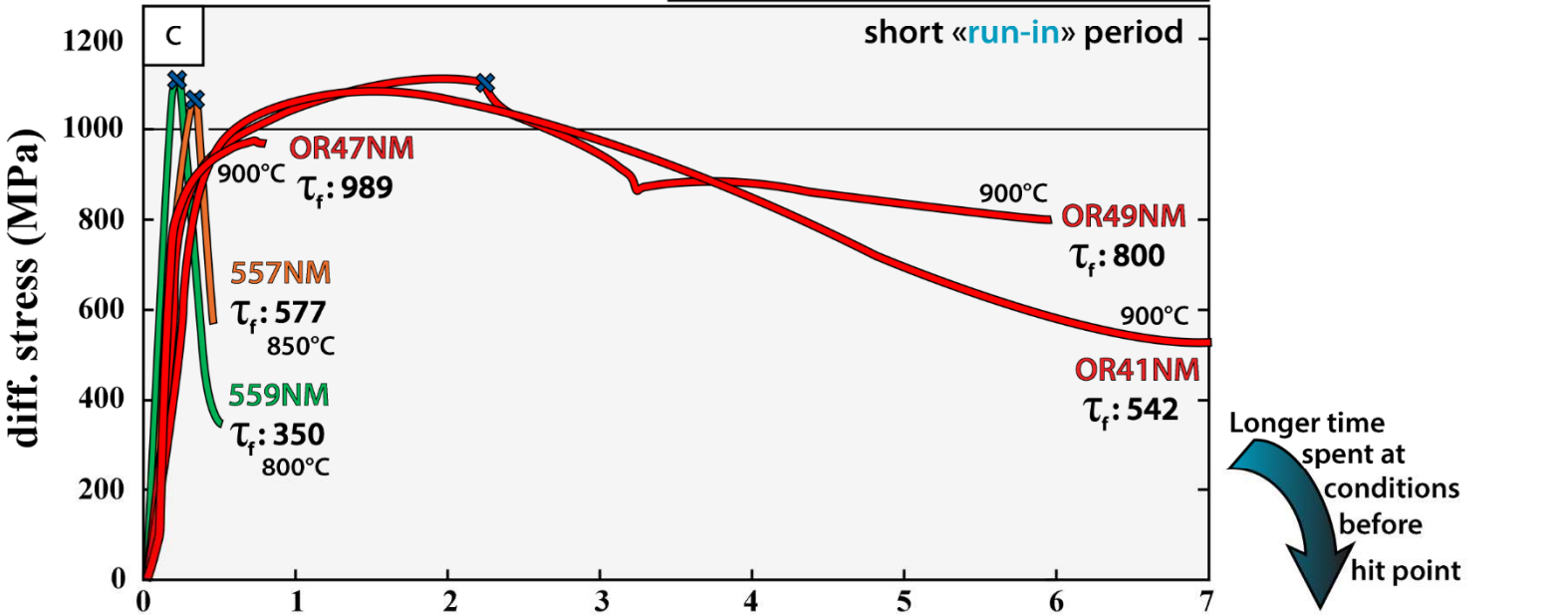


Fig. 2.

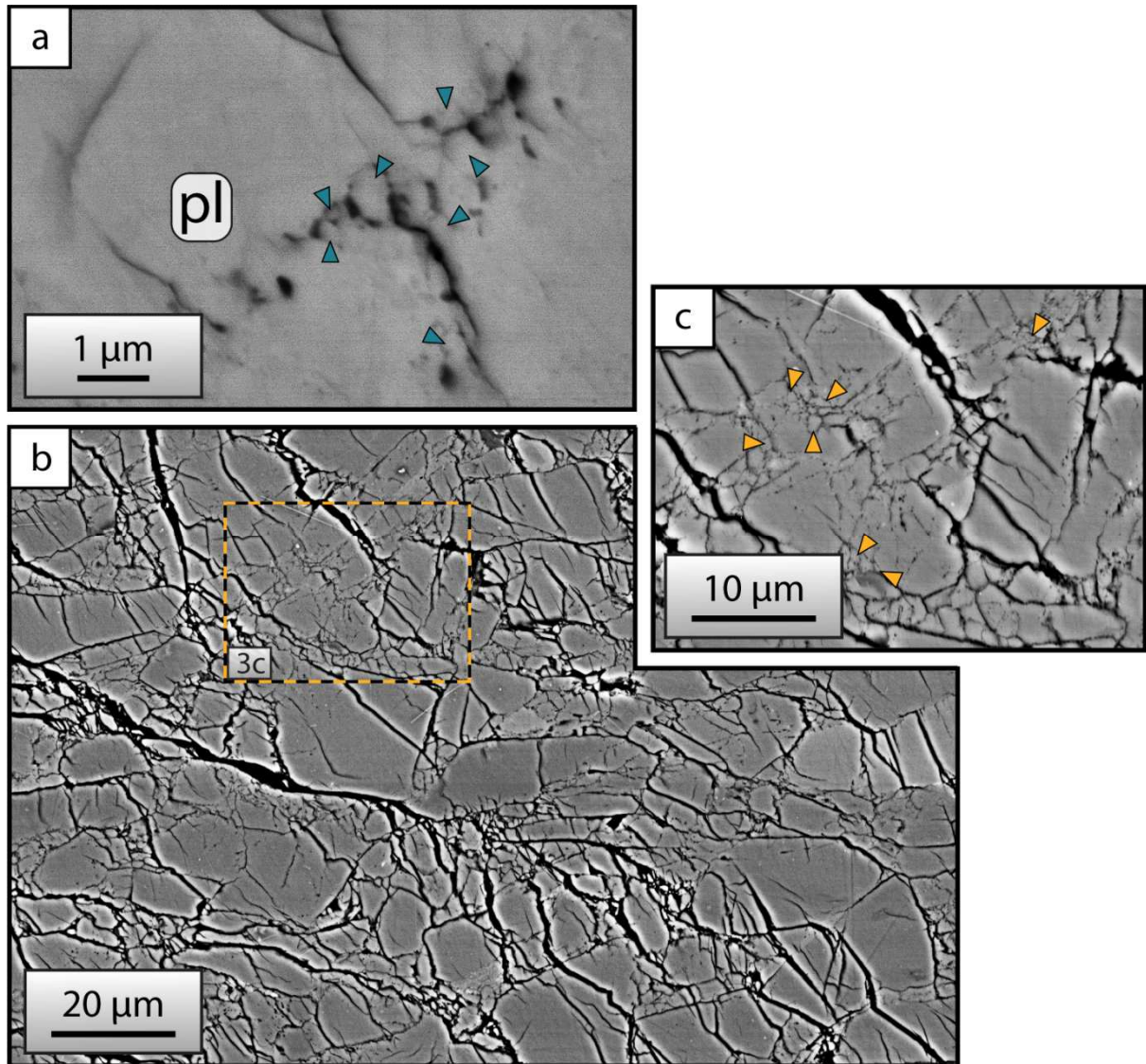


Fig. 3.

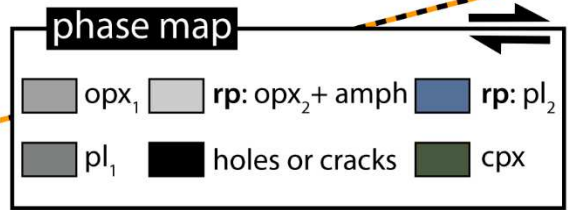
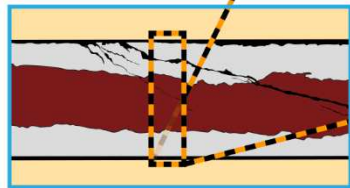
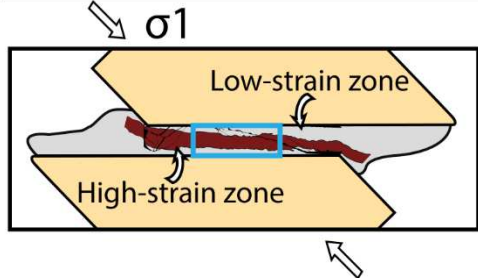
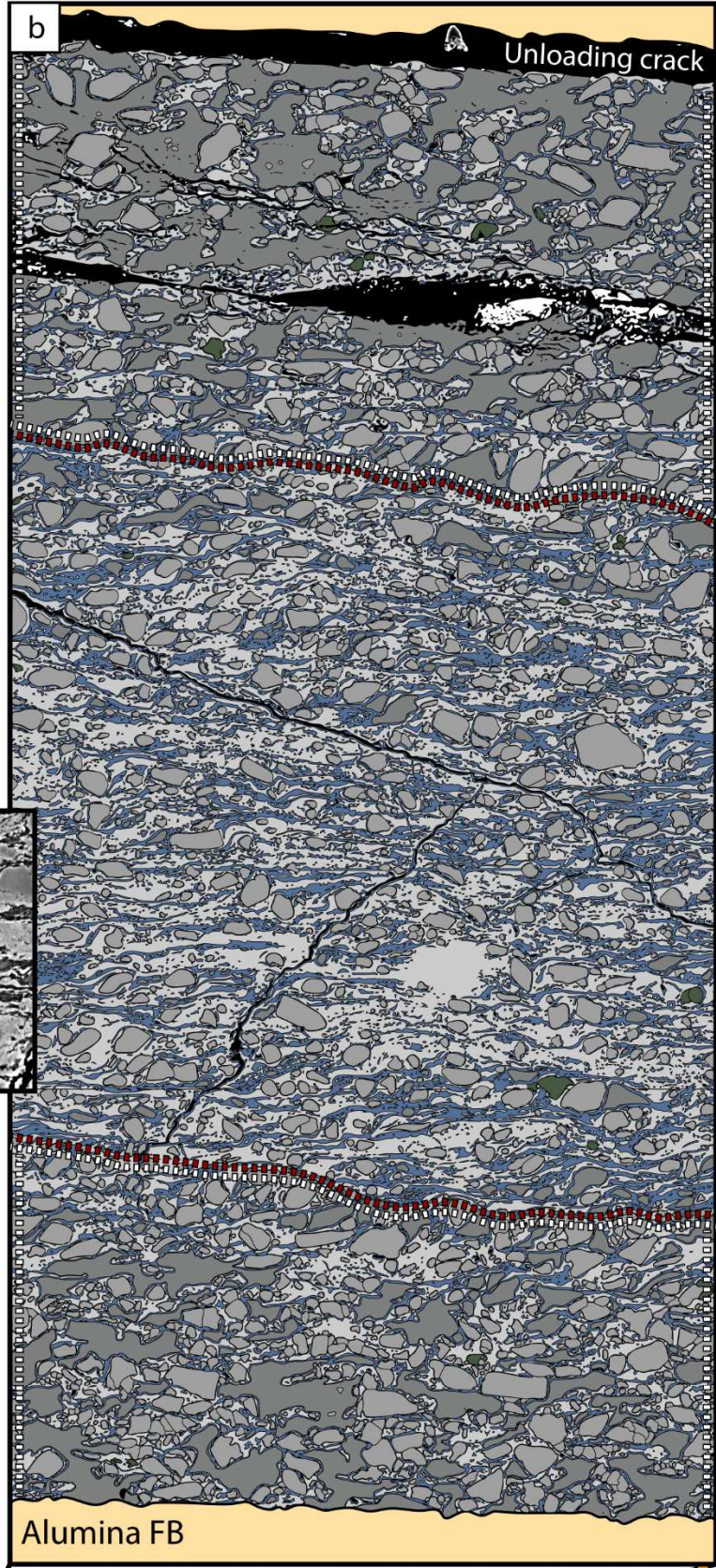
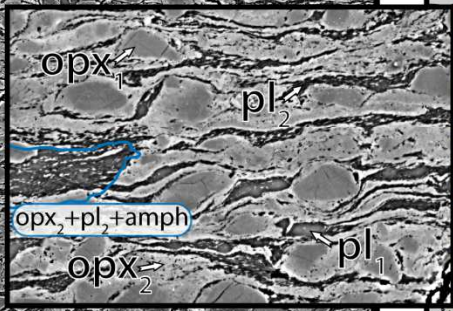
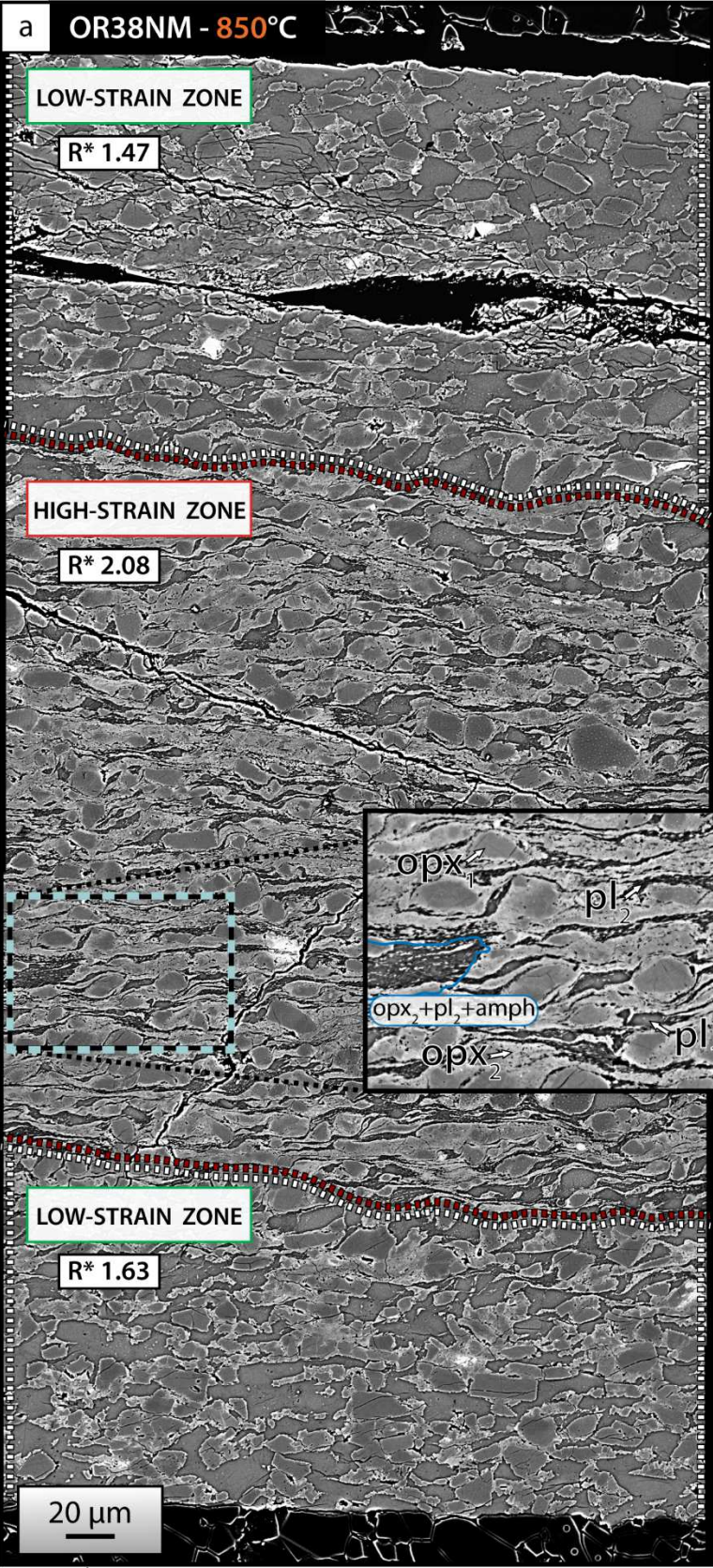


Fig. 4.

Journal Pre-proof



Mixture Opx + Plag - High-shear-strain experiments - All at 850°C

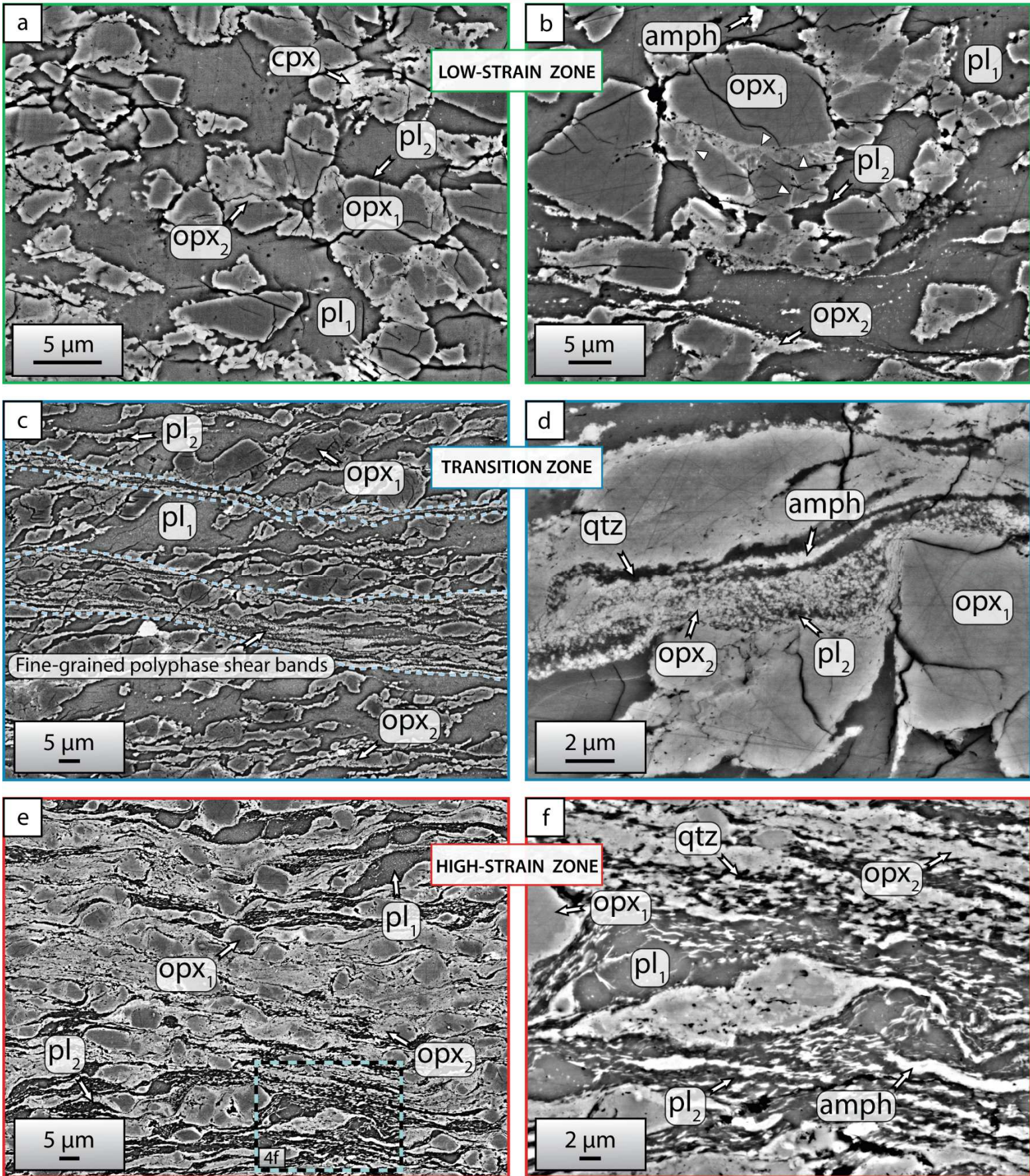


Fig. 5.



High-strain zones in *Opx* + *Plag* assemblages - 850 - 900°C

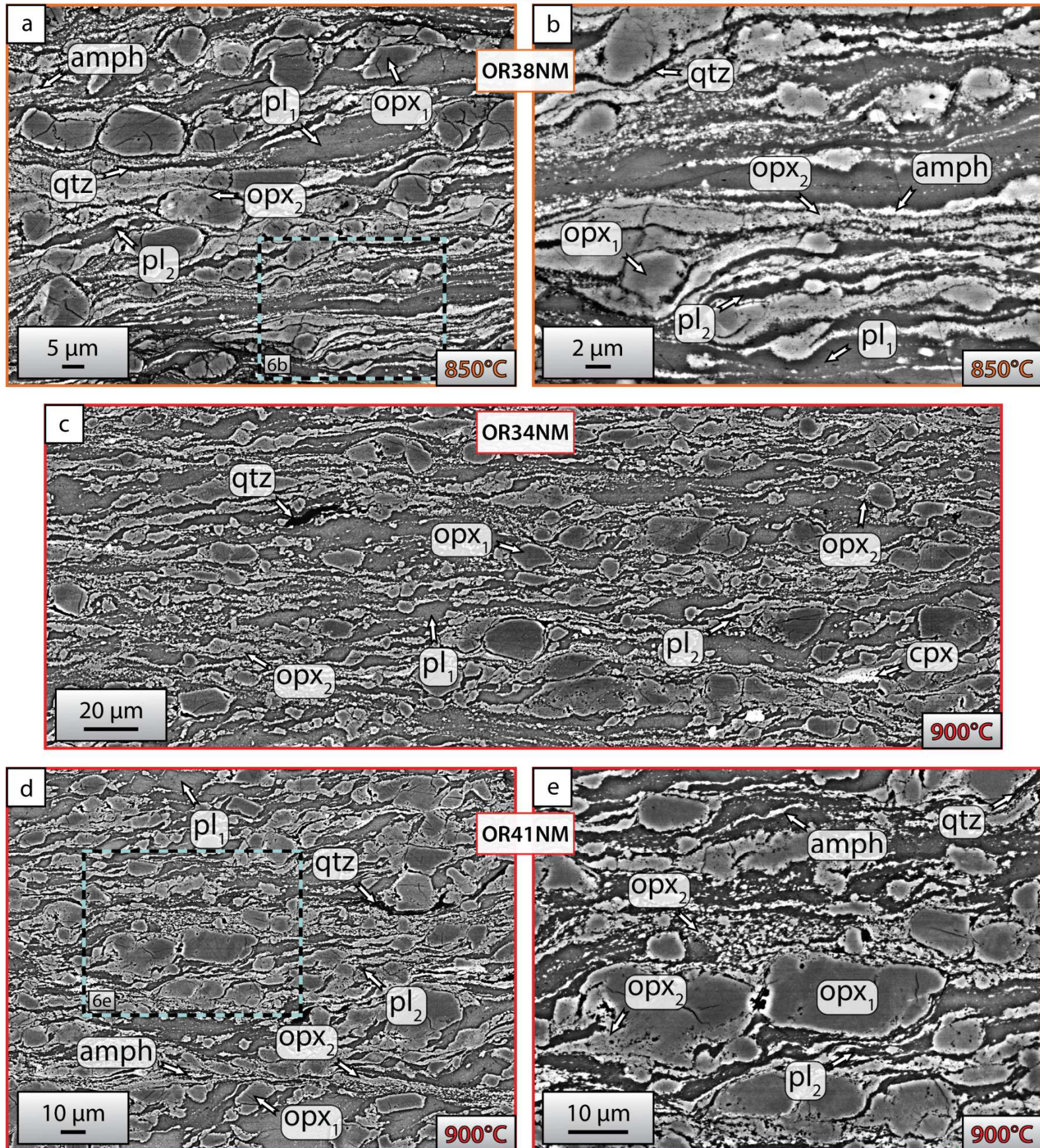


Fig. 6.

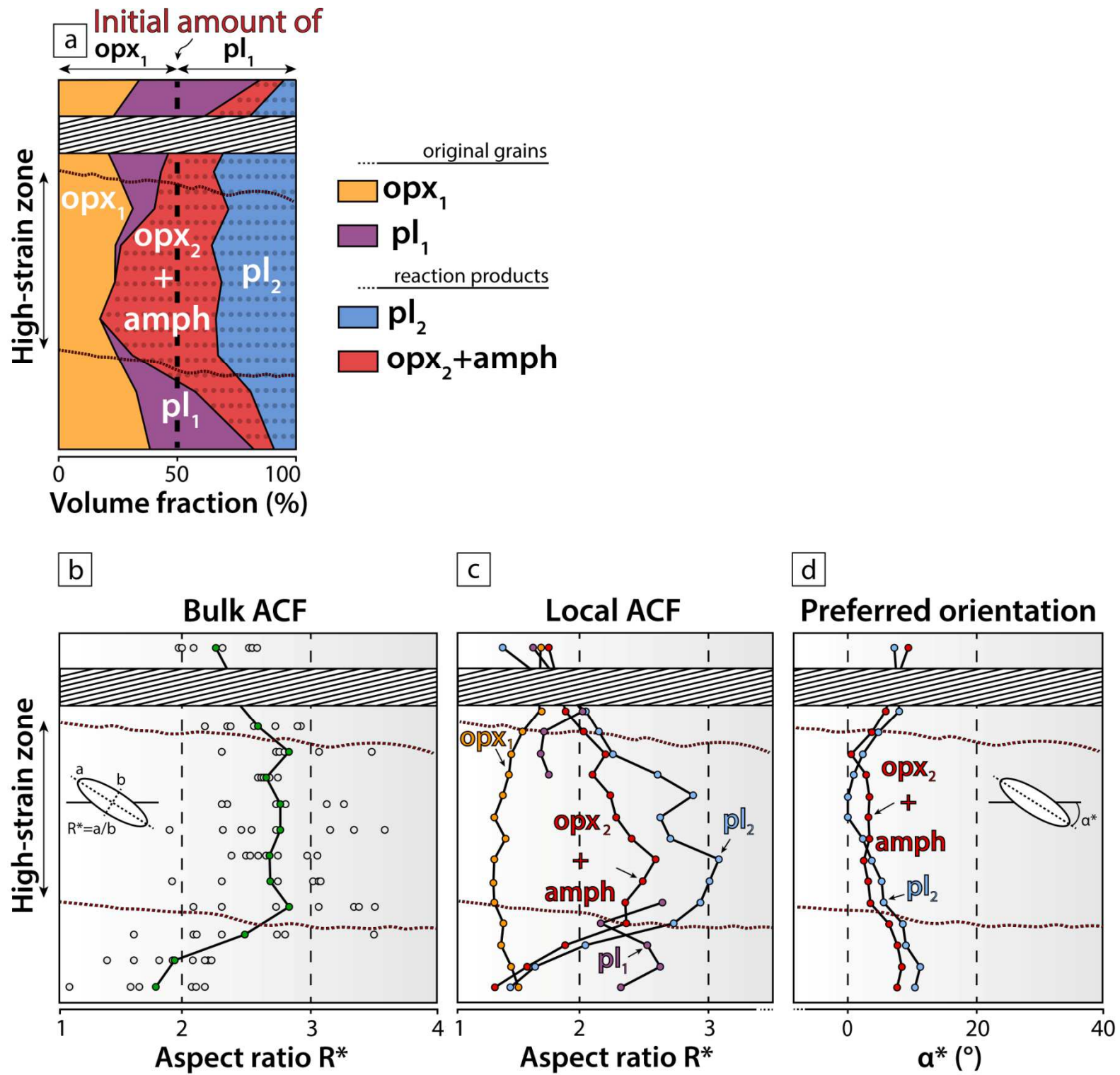


Fig. 7.

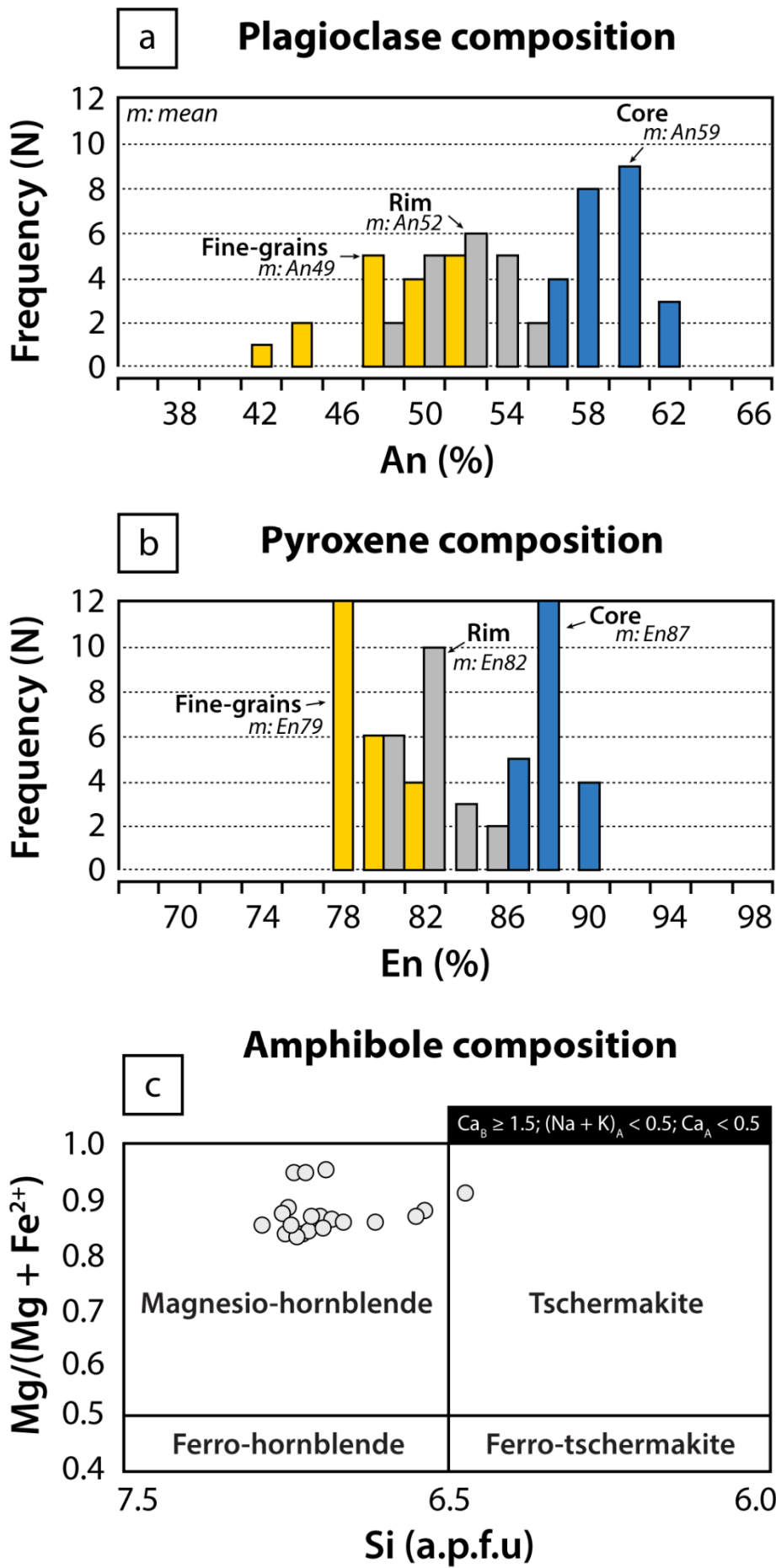


Fig. 8.

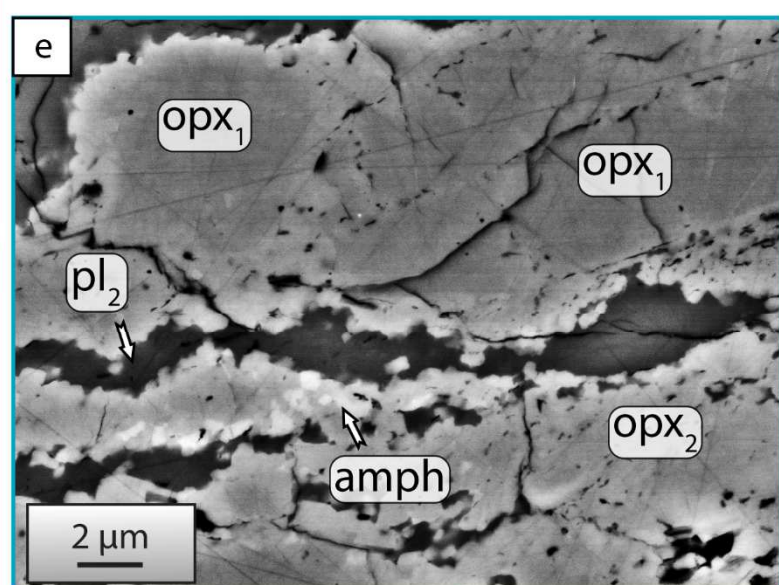
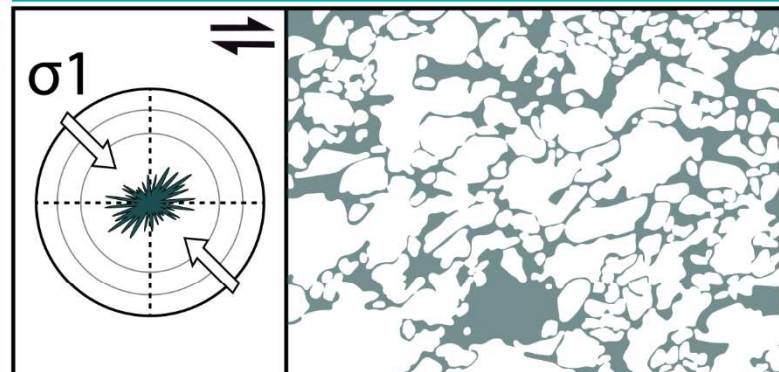
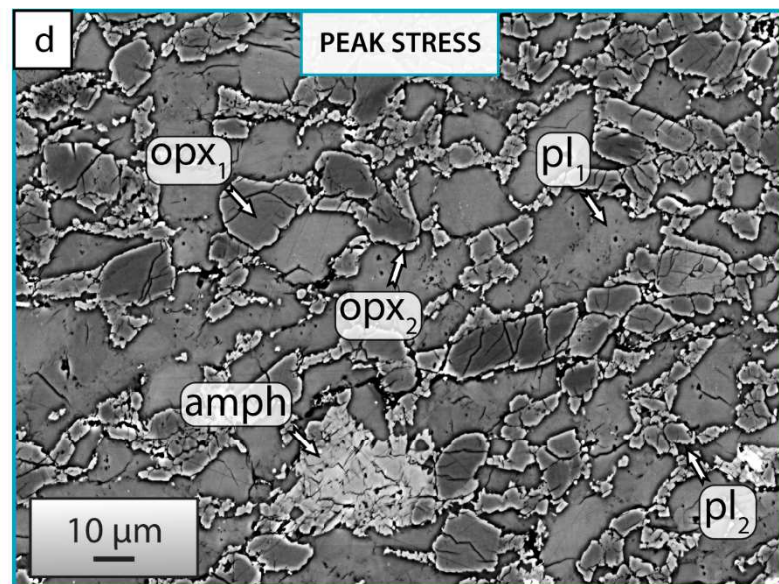
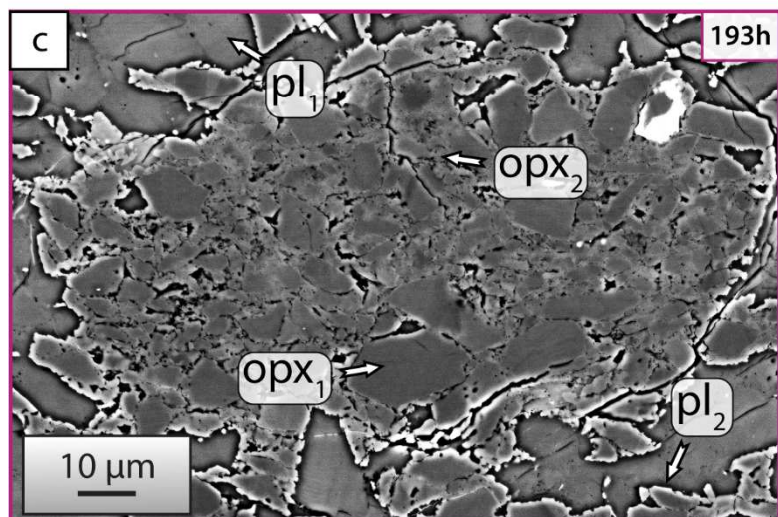
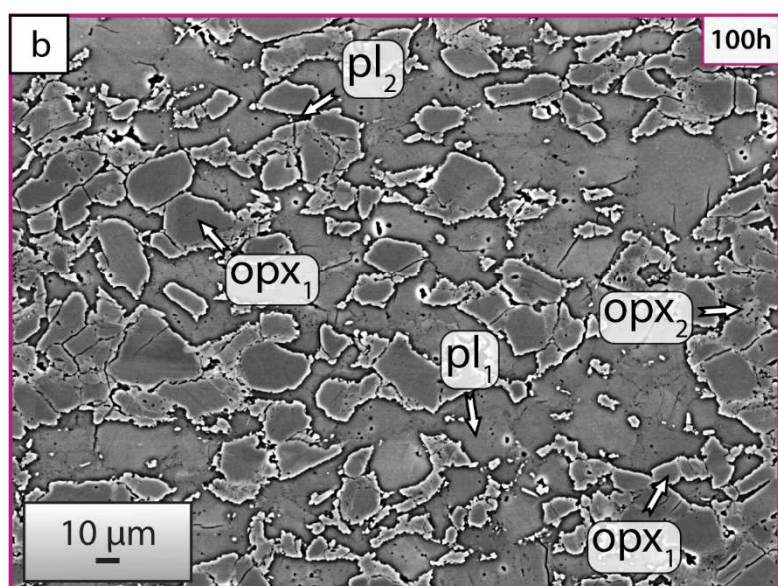
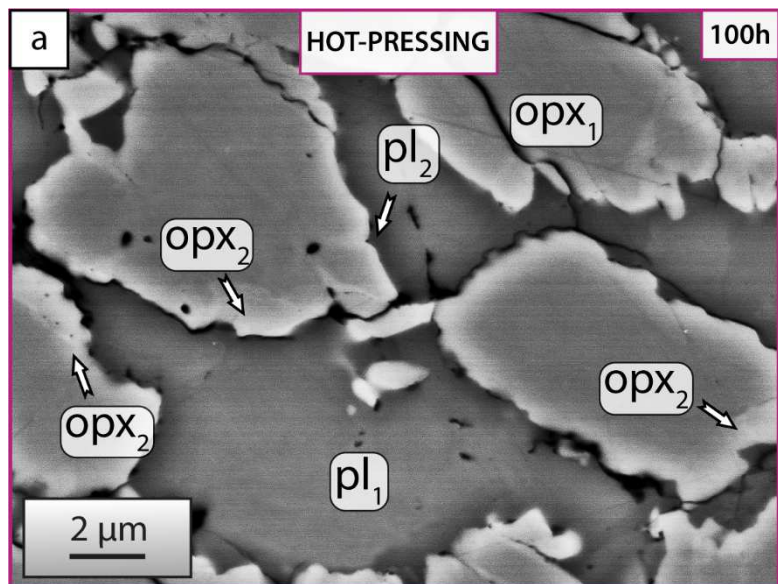


Fig. 9.

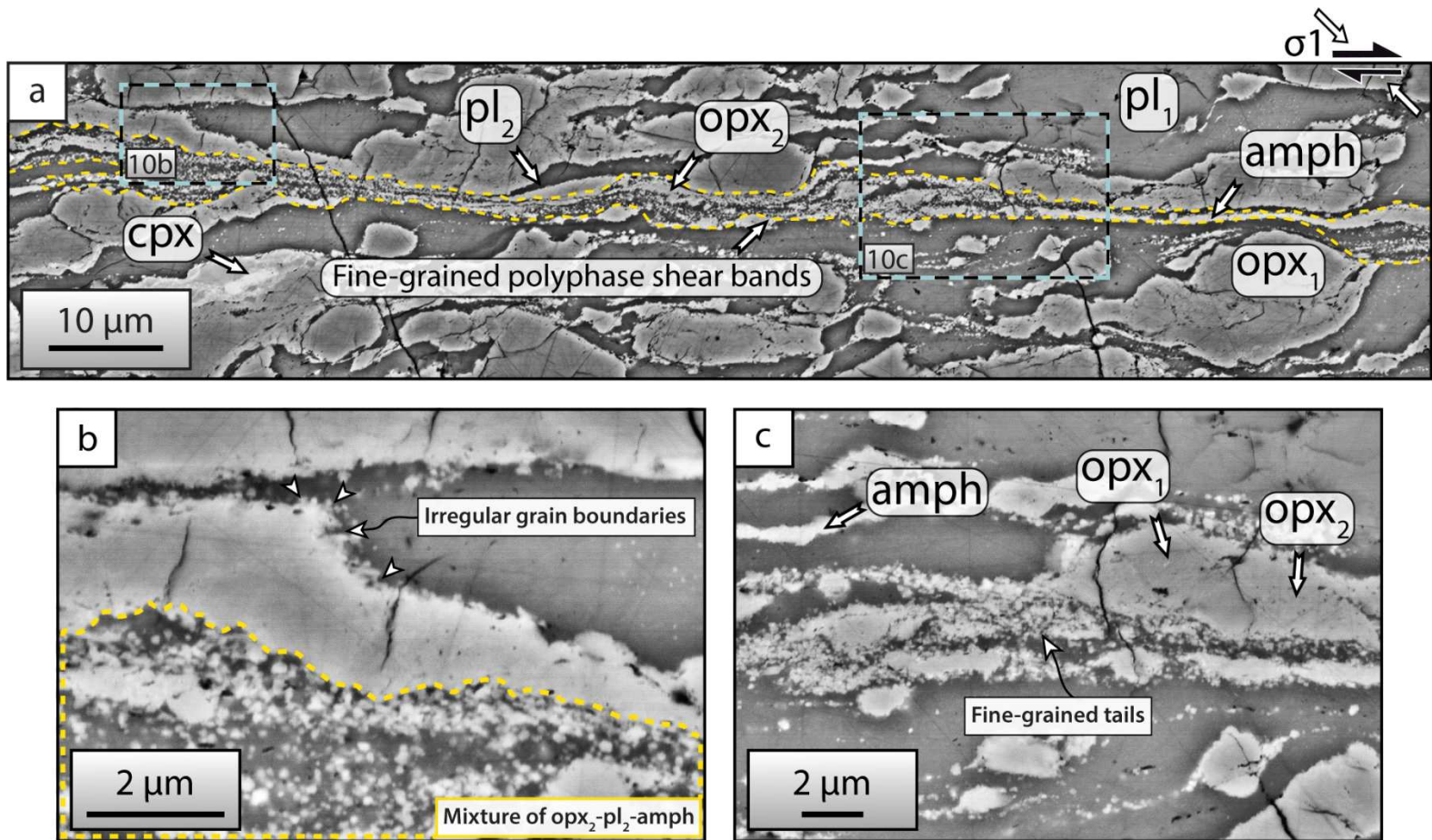


Fig. 10.

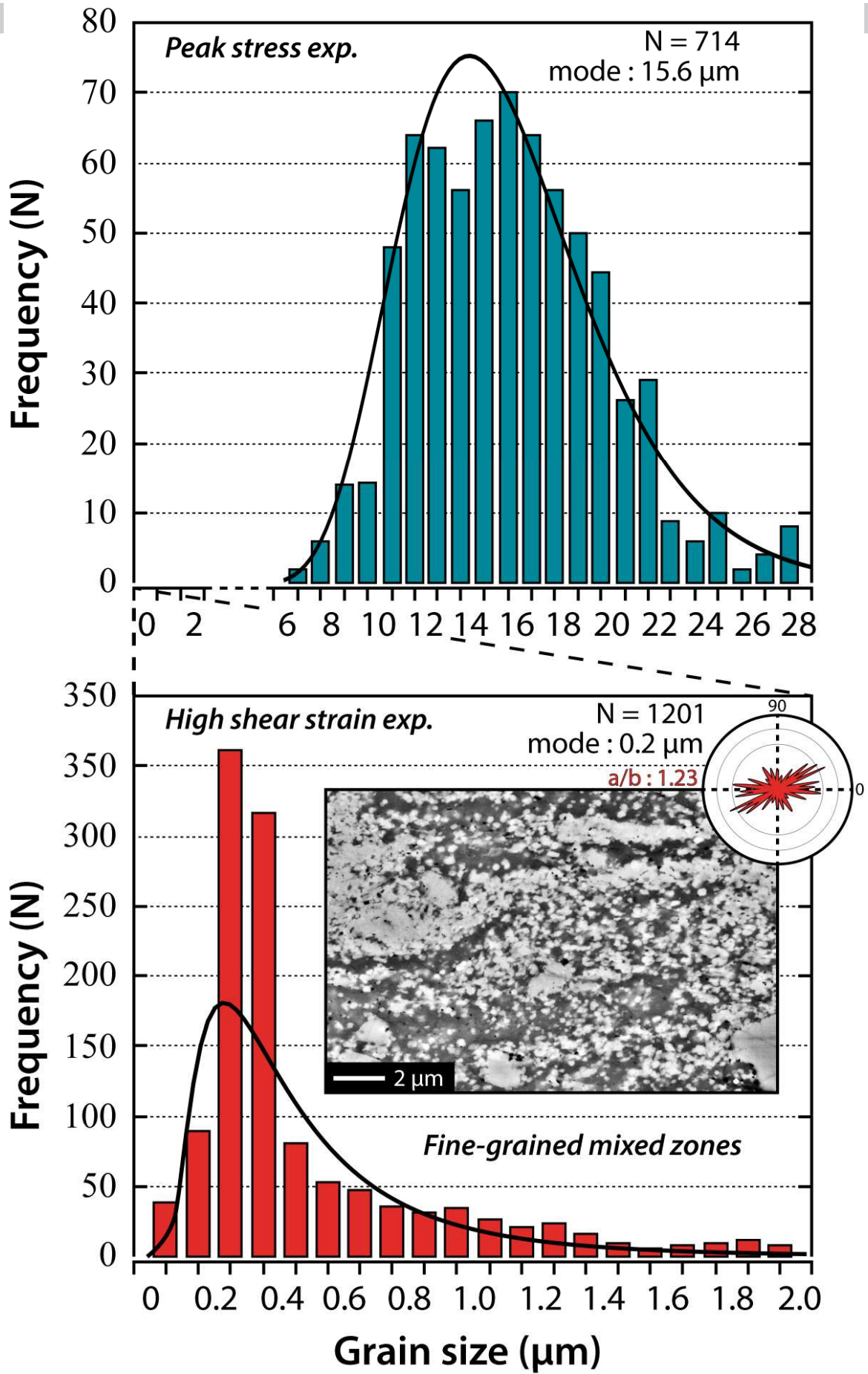


Fig. 11.

Hot-pressing Peak stress Intermediate shear strain High shear strain

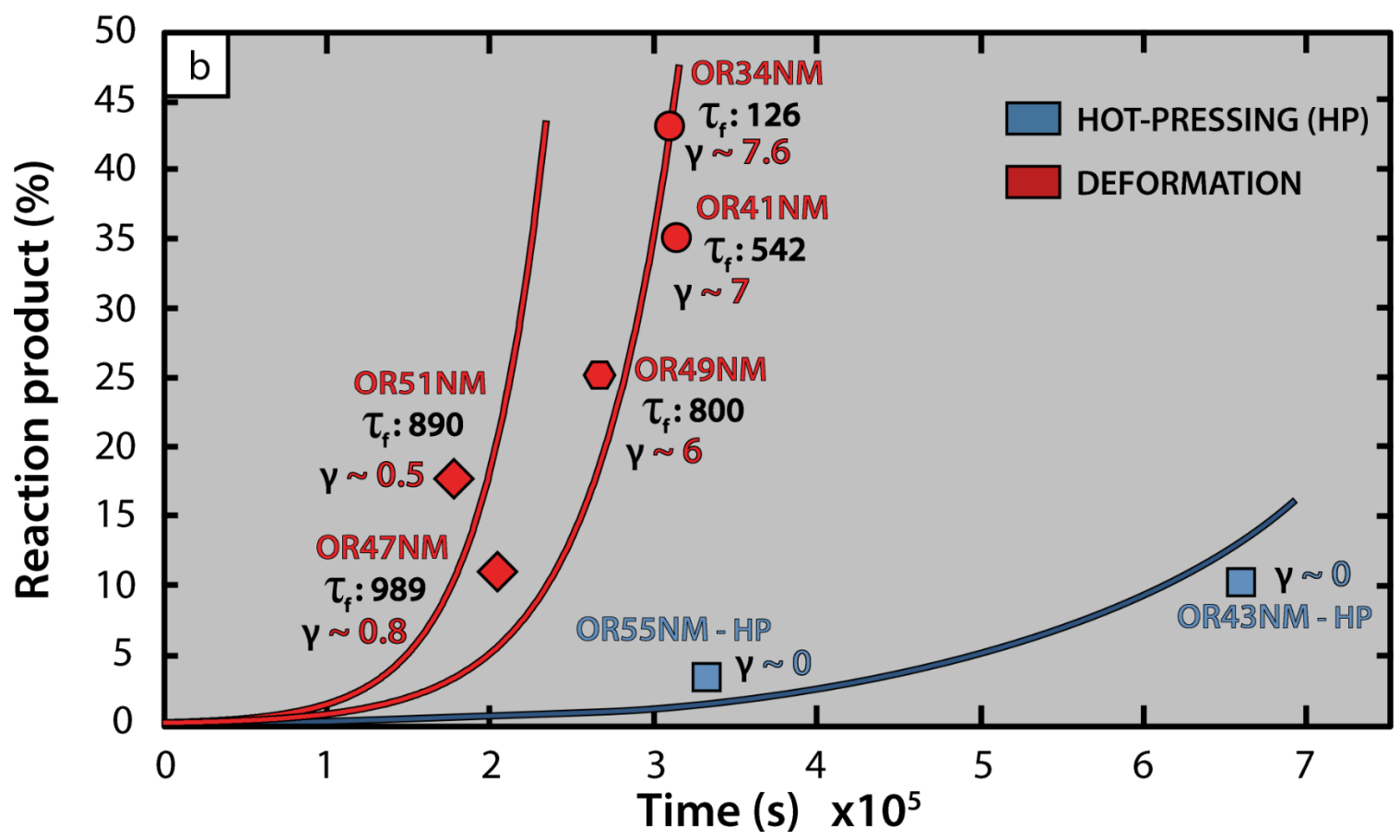
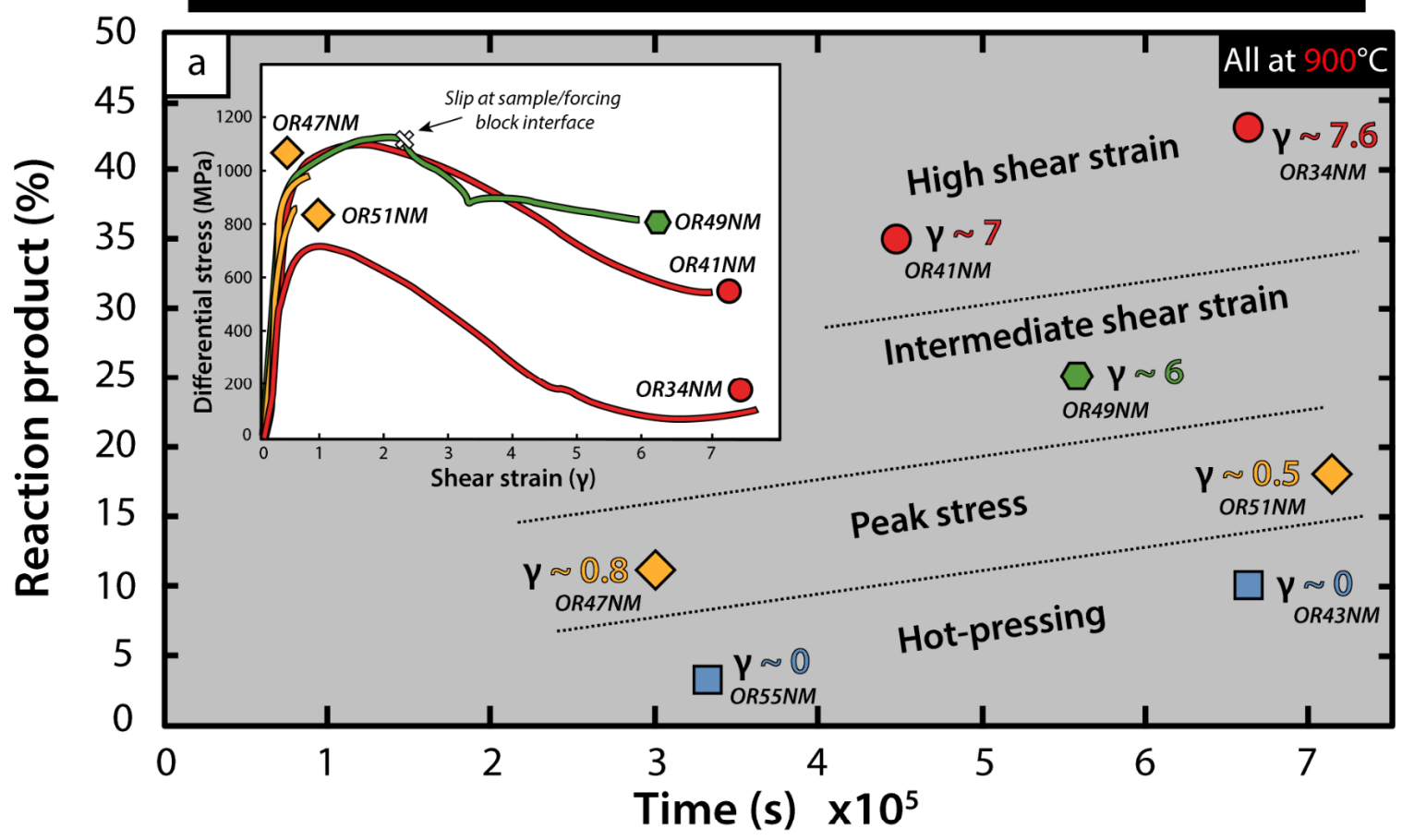


Fig. 12.

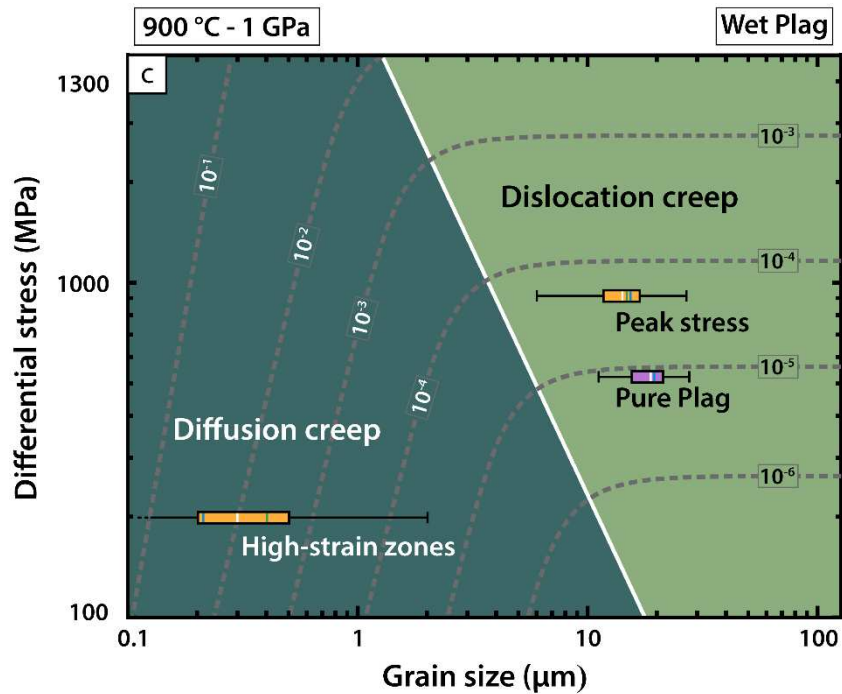
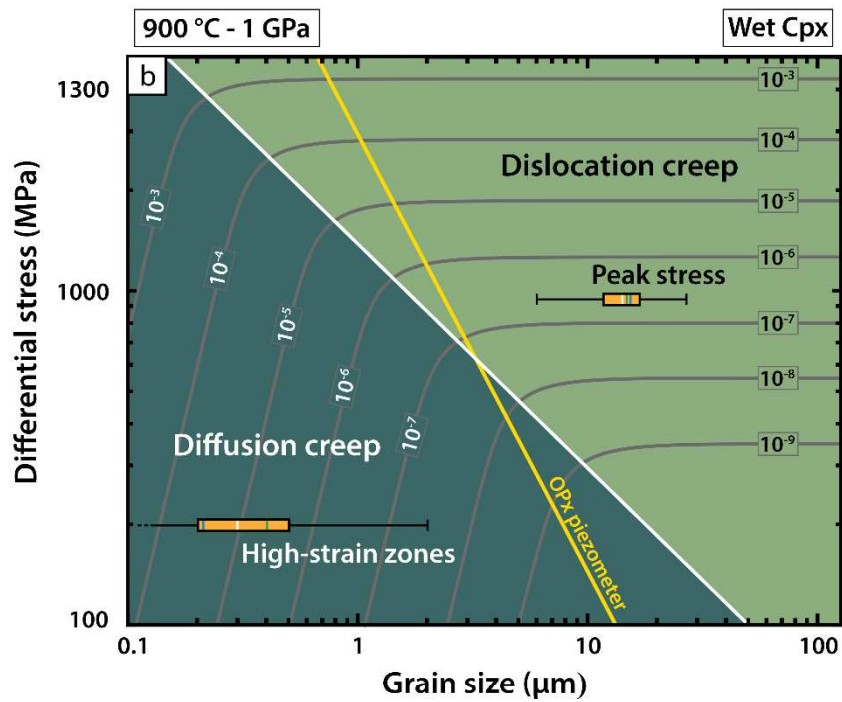
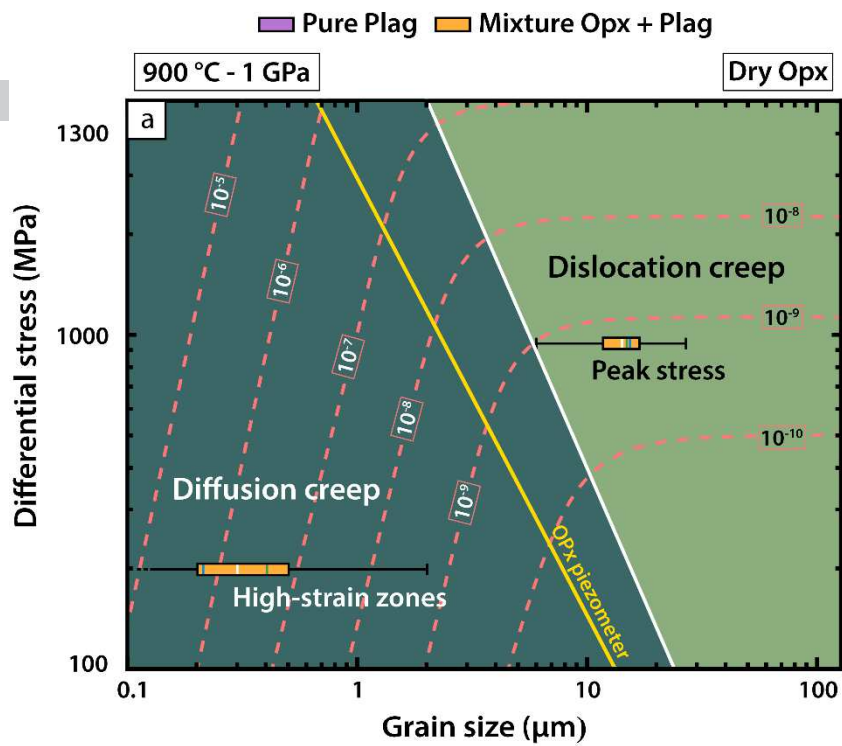


Fig. 13.

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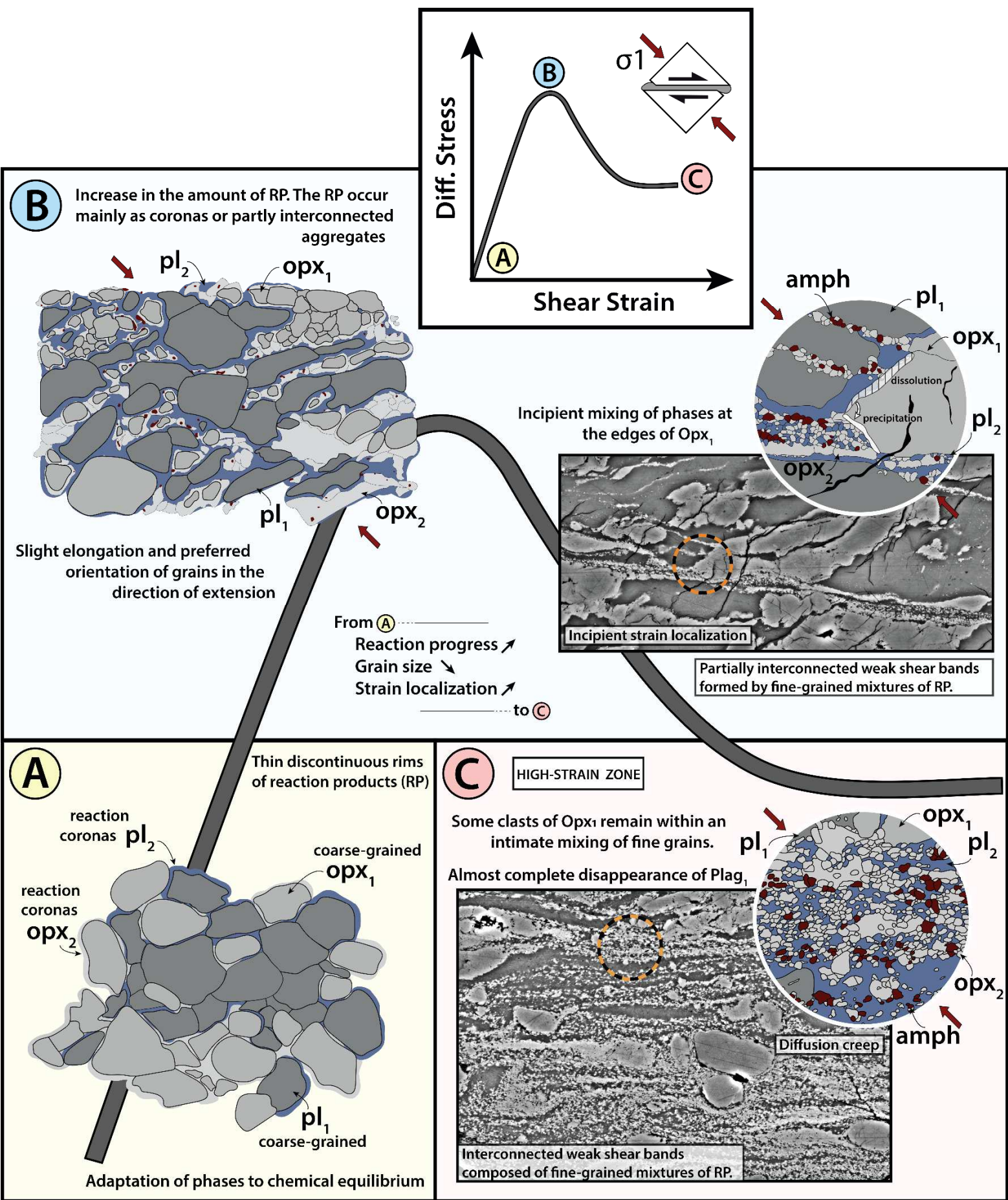


Fig. 14.

- Deformation considerably enhances the kinetics of mineral reactions.
- Diffusion creep is dominant in fine grained aggregates.
- Strain localization is due to the interplay between deformation and reaction.
- The degree of connectivity of the reacted material controls the bulk rheology.

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Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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