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1 Porosity of metamorphic rocks and fluid migration within subduction interfaces

2

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9 **Abstract:**

10 Large earthquakes break the subduction interface to depths of 60 to 80 km. Current
11 models hold that seismic rupture occurs when fluid overpressure builds in link with porosity
12 cycles, an assumption still to be experimentally validated at high pressures. Porosities of
13 subduction zone rocks are experimentally determined under pressures equivalent to depths of
14 up to 90 km with a novel experimental approach that uses Raman deuterium-hydrogen
15 mapping. Natural rocks (blueschists, antigorite serpentinites, and chlorite-schists)
16 representing a typical cross-section of the subduction interface corresponding to the deep
17 seismogenic zone are investigated. In serpentinite, and to a smaller extent blueschist, porosity
18 increases with deformation, whereas chlorite-rich schists remain impermeable regardless of
19 their deformation history. Such a contrasting behavior explains the observation of over-
20 pressurized oceanic crust and the limited hydration of the forearc mantle wedge. These results
21 provide quantitative evidence that serpentinite, and likely blueschist, may undergo porosity
22 cycles making possible the downdip propagation of large seismic rupture to great depths.

23

24 **Keywords:** subduction interface, porosity, permeability, seismic cycle, fluid overpressure

25 **1. Introduction**

26

27 The interface between a subducting oceanic plate and overriding lithosphere and
28 mantle wedge (the subduction interface) is an area of intense seismic activity. Models of the
29 earthquake cycle have invoked fluid pressure variations to explain faulting behavior. Fluid
30 pressure progressively increases to near lithostatic values during the inter-seismic period
31 before it is released during an earthquake rupture (Sleep and Blanpied, 1992). Fluid pressure
32 variations may be explained either by upward fluid movement through a rigid matrix or by
33 compaction of the deformable fault zone (Sleep and Blanpied, 1992), both associated with
34 permeability changes. The seismic cycle is associated with a “porosity” cycle, with porosity
35 decreasing during the inter-seismic phase, and increasing through brittle deformation during
36 an earthquake. Porosity and permeability are also fundamental parameters in modeling of
37 fluid circulation, and its influence on frictional behavior (Khoshmanesh and Shirzai, 2018).

38 Permeability and fluid pressure variations during the seismic cycle have been
39 confirmed by in-situ measurements of heat flow and residual temperature on the subduction
40 interface (Fulton et al., 2013). Variations of the hydrological properties of crustal rocks
41 between seismic and interseismic periods is also inferred from hydraulic measurements
42 (Ingebritsen and Manning, 2010). Fluid overpressures in the subducted oceanic crust are
43 imaged by seismology (Audet et al., 2009; Shiina et al., 2013), and require contrasted
44 hydrological properties in the different lithologies of the oceanic crust and overlying mantle
45 (Peacock et al., 2011). Porosity and permeability are also essential for modeling fluid flow in
46 the various lithological layers dipping along the megathrust interface (Morishige and van
47 Keken, 2018).

48 Experimental values of rock porosity and permeability need to be determined as
49 critical parameters for numerical simulations of subduction dynamics (Audet et al., 2009;

50 Peacock et al., 2011). Direct porosity or permeability measurements based on fluid flow
51 measurements are restricted to confining pressures up to 150 MPa (Katayama et al., 2012;
52 Kawano et al., 2011; Shmonov et al., 1995), a pressure range that addresses the upper five
53 kilometers of the seismogenic zone, where clay minerals dominate the interface (Chester et
54 al., 2013; Ujiie et al., 2013). Extrapolation to greater depths is uncertain. Use of the Kozeny-
55 Carman relationship on ambient temperature experiments in the 50-100 MPa range
56 (Katayama et al., 2012; Kawano et al., 2011) yields permeabilities below 10^{-21} m² at
57 subduction zone pressures, a threshold below which rocks are impermeable. Similar
58 experiments up to 150 MPa (Shmonov et al., 1995) indicate stabilization of the permeability
59 values in the 100-150 MPa range instead of a continuous decrease with increasing pressure,
60 and a complex behavior upon heating and reaching dehydration of hydrated rocks around
61 300-400°C for serpentines.

62 At higher temperatures, permeability and fluid connectivity have been determined
63 under conditions (800 - 1200°C, 1 - 5 GPa) pertinent to magmas under volcanic arcs, and
64 where minerals can reach textural equilibrium with fluids (Mibe et al., 1999; Wark and
65 Watson, 1998). These textural equilibrium experiments may capture the long-term behavior
66 of polycrystalline aggregates with dry minerals of moderate anisotropy (olivine, quartz) but
67 they are not relevant to highly anisotropic materials such as amphiboles and phyllosilicates
68 stable in forearc rocks, and at high deformation rates.

69 In order to bridge the gap between the low- and high-pressure high-temperature range,
70 we developed a novel experimental technique that uses Raman deuterium-hydrogen mapping
71 to determine the porosity of metamorphic rocks (blueschists, antigorite serpentinites, and
72 chlorite-schists) representing a typical cross-section of the subduction interface corresponding
73 to the deep seismogenic zone. They represent, respectively, the metamorphosed oceanic crust
74 at the footwall of the subduction megathrust, the hydrated forearc mantle wedge at the

75 hanging wall, and the metasomatic rocks or sediments at the interface between the subducting
76 plate and the mantle wedge (Spandler et al., 2008). Experiments were conducted up to 3 GPa
77 corresponding to depths of about 90 km. This covers the portion of the subduction interface
78 that requires mechanical decoupling for matching the heat flux observations in geodynamic
79 thermal models (Syracuse et al., 2010; Wada et al., 2008).

80

81 **2. Methods**

82

83 **2.1 Starting material**

84 We used four different natural rocks, all dominated by one or two hydrated mineral:
85 two serpentinites (SB: BCS16a from Baja California, Mexico, and SC: Cu12 from Escambray
86 Massif, Central Cuba) dominated by antigorite, one chlorite-schist (C: SZ24 from the Saas-
87 Zermatt unit, Western Alps) dominated by chlorite, and one blueschist (G: from Groix Island,
88 French Armorican Massif) dominated by glaucophane and epidote. These rocks are
89 representative of high-pressure low-temperature fossil subduction rocks, i.e. with mineral
90 assemblages, compositions, and strength corresponding to subduction zones.

91 The two serpentinites are dominated by antigorite, the high-temperature form of
92 serpentine (Schwartz et al., 2013), with minor amount of chlorite, magnetite and carbonate
93 (less than 5% in both rocks). SC also contains traces (<5% each) of lizardite and chrysotile,
94 the two other varieties of serpentine. Chemical formulas for the $m = 14$ modulation of
95 antigorite are $(\text{Mg}_{2.52}\text{Fe}_{0.24}\text{Al}_{0.01})_{\Sigma=2.77}(\text{Al}_{0.06}\text{Si}_{1.94})_{\Sigma=2}\text{O}_5(\text{OH})_{3.57}$ for SB (Pilorgé et al., 2017),
96 and $(\text{Mg}_{2.54}\text{Fe}_{0.16}\text{Cr}_{0.01}\text{Al}_{0.09})_{\Sigma=2.8}(\text{Al}_{0.1}\text{Si}_{1.9})_{\Sigma=2}\text{O}_5(\text{OH})_{3.7}$ for SC (Reynard et al., 2011).
97 Antigorite grains are up to 100-150 μm long and 10-20 μm -thick, with grain size being
98 slightly lower in SB than in SC. SB is heterogeneously deformed and exhibits narrow zones
99 of low-temperature semi-brittle deformation surrounding zones with high-temperature

100 schistosity. We refer to it as the deformed serpentinite. SC has a high-temperature schistosity
101 of strong to very strong intensity (Bezacier et al., 2010) with well-recrystallized individual
102 grains and does not exhibit significant retrograde deformation. We refer to it as the
103 undeformed serpentinite.

104 The chlorite-schist (C) is composed mainly of chlorite (>95%) with minor magnetite
105 and carbonate (<5%). The chemical formula of chlorite grains (100-500 μm -long and 10-50
106 μm -thick) is $(\text{Mg}_{8.93}\text{Fe}^{2+}_{0.16}\text{Al}_{0.18}, \text{Al}_{1.98})(\text{Si}_{6.37}\text{Al}_{1.63})\text{O}_{20}\text{OH}_{16}$ (Ganzhorn et al., 2018). They are
107 well recrystallized and almost free of deformation.

108 The blueschist (G) exhibits a mineralogical foliation with glaucophane-rich layers
109 (600-1000 μm thick) alternating with epidote-rich layers (200-1000 μm thick). The
110 glaucophane-rich layers were selected for experiments. They mainly contain glaucophane
111 (>95%) with minor epidote, quartz, garnet, and titanite (<5%). Chemical formulas are
112 $\text{Na}_{1.8}\text{Ca}_{0.2}\text{Fe}_{1.0}\text{Mg}_{2.28}\text{Al}_{1.9}\text{Si}_{7.7}\text{O}_{22}(\text{OH})_2$ for glaucophane and $\text{Ca}_{1.9}\text{Al}_{2.5}\text{Fe}_{0.5}\text{Si}_3\text{O}_{12}(\text{OH})$ for
113 epidote. Glaucophane and epidote grains are ca. 30 μm long. Grains are well recrystallized
114 and show little evidence of internal deformation in the form of subgrain boundaries.

115 The same preparation protocol was applied to all natural rocks. Hand-scale sample
116 was crushed separately in an agate mortar and the different fractions were separated by a
117 series of sieves. Two different grain sizes of powder were used for experiments on
118 serpentinite (<125-100> μm sieves and <50 μm sieve) and only a single grain size was used
119 for the other natural rocks (<125-100> μm sieves for chlorite-schist and <80 μm sieve for
120 blueschist). Two grain-sizes of powder were used in the first experiments in order to test for
121 compaction, water fraction and kinetic effects, after which the largest grain size of powder
122 was preferred. This allowed increasing the amount of initial deuterated water from 3-10 wt%
123 to 10-20 wt%. Sub-micrometric particles were left in the fraction as they dissolve more

124 readily in water during the high-temperature stage, saturating the fluid so that it is in
125 equilibrium with the rock, limiting as far as possible potential dissolution within it.

126 Intact rock pieces were introduced in the experimental charge along with powder to
127 simulate pristine rocks (Fig. 1). When possible, cores of about 1.5 mm diameter were used.
128 When it was not possible to core the rock due to schistosity and brittle behavior, chips of
129 several hundreds of micrometers were used.

130

131 **2.2. High-pressure experiments**

132 Experiments were performed in a belt apparatus at the University of Lyon 1 (Fig. 1).
133 The belt press is a high-volume press with typical sample size of 70 mm³. The powders and
134 one core, or one to three chips of natural rocks were loaded in a gold capsule (6 mm-long and
135 4 mm in diameter). Powders were soaked with D₂O (purity 99 atom%, Sigma-Aldrich) that
136 filled the porosity.

137 Capsules are designed to seal in the belt apparatus while pressure is increased at
138 ambient temperature (Pilorgé et al., 2017). In order to limit D₂O leakage at ambient pressure
139 and temperature, D₂O was added and capsules were closed only a few hours (3-5) before
140 running experiments. Additionally, they were wrapped in paraffin film and kept frozen until
141 used in the belt apparatus. Experiment names, *e.g.* C-XX-315, are built as follow: the first
142 letter indicates the protolith (SB: serpentinite BSC16a, SC: serpentinite Cu12, C: chlorite-
143 schist, G: glaucophane-rich blueschist), XX is the number of the experiment, and the last
144 number indicates the experimental temperature of isotopic exchange.

145 Belt experiments were run at several temperatures and 1.5 - 3 GPa for 12 - 38 hours.
146 Each gold capsule was placed in an insulating boron nitride gasket inside a cylindrical
147 graphite furnace which itself was surrounded by a pyrophyllite gasket. Pressure was
148 calibrated against the room temperature fixed-point metal transitions and the high-temperature

149 quartz to coesite transition. Due to the design of the belt press, temperature cannot be directly
150 monitored with a thermocouple but is calculated from the calibrated relationship between
151 input power and capsule temperature. Input power – capsule temperature was calibrated at 1
152 and 5 GPa over a broad range of temperatures (25-1000°C), and temperature uncertainty is
153 estimated to be $\pm 40^\circ\text{C}$ (Koga et al., 2005).

154 The sample was pressurized at room temperature before heating (Fig. 1) for 12 to 48
155 hours at constant pressure and temperature, the only stage at which isotopic exchange
156 between fluids and minerals is possible through diffusion. Samples were then quenched to
157 ambient temperature before decreasing the pressure within a few hours (0.02 GPa/min).
158 Recovered capsules were deformed with a shape ratio (length/diameter) ca. 0.5 to be
159 compared with the initial ratio of 1.4. Deformation occurred due to the soft pressure media
160 (i.e. the boron nitride) and the high D₂O content and high porosity of the powder.

161 The weight of deformed capsules was monitored before and after piercing and drying
162 at 120°C for 10 minutes in a sterilizer. Weight loss after drying indicates that capsules still
163 contained water after the experiments. It was demonstrated that with this type of capsule and
164 assemblage, the loss of D₂O during an experiment and the hydrogen loss and water
165 dissociation were low (Ganzhorn et al., 2018; Pilorgé et al., 2017). Thus capsules are assumed
166 to be closed systems.

167 Quenched capsules were mounted in epoxy resin and cut lengthwise in two parts with
168 a wire diamond saw. Both parts were then entirely remounted in epoxy to properly fix the
169 loose crystals in the powder. They were polished with silicon carbide abrasive paper down to
170 a grain size of 2.5 μm , and then with a solution of alumina down to 0.5 μm . Cores (or chips)
171 were sometimes lost during the preparation due to poor mechanical integrity of the powder.

172

173 **2.3. Raman spectroscopy**

174 We used a Horiba™ LabRam HR Evolution and a 532 nm continuous wave laser
175 source. This confocal setup coupled with an objective of x100 magnification and a high-
176 resolution stage for the sample movements allowed us to acquire hyperspectral images with
177 spatial resolutions between 0.5 to 1.5 μm depending on the map size. Each spectrum was
178 acquired in 2 accumulations of 2 seconds with a laser power of 30 mW on the sample.

179 No deuterium was detected in the characteristic Raman spectra of the hydrous mineral
180 of the studied natural rocks, owing to the low natural deuterium abundance (< 0.02 atom%),
181 well below the detection limit ca. 0.1% at most. OD vibrations are observed only after
182 interaction with D_2O at high pressure and temperature. For all investigated minerals, OD and
183 OH bonds have different Raman shifts in the regions $2440 - 2745 \text{ cm}^{-1}$ and $3300 - 3720 \text{ cm}^{-1}$,
184 respectively, allowing good discrimination between the two. Epidote has the lowest Raman
185 shifts ($2440 - 2560, 3301 - 3426 \text{ cm}^{-1}$), glaucophane shows thin bands ($2671 - 2712, 3613 -$
186 3674 cm^{-1}), antigorite has several bands in the $2600-2730$ and $3560-3700 \text{ cm}^{-1}$ range, and
187 chlorite shows the broadest bands ($2460 - 2730, 3310 - 3700 \text{ cm}^{-1}$).

188 For each mineral, the OD bands are strictly homologous to the OH bands with a
189 frequency ratio of 1.356 ± 0.001 , which correspond to the theoretical harmonic reduced mass
190 ratio (1.374) and to minor anharmonic effects (Reynard and Caracas, 2009). This indicates
191 that deuterium substitutes for hydrogen on their crystallographic sites through a lattice
192 diffusion process without modifications of the mineral chemistry or structure, and that
193 dissolution and precipitation processes, if any, are not detected by Raman or observed by
194 SEM. By integrating the area below OD and OH bands, it is possible to calculate the
195 $\text{D}/(\text{D}+\text{H})$ ratio for each spectrum and convert the Raman map into a $\text{D}/(\text{D}+\text{H})$ map (Ganzhorn
196 et al., 2018; Pilorgé et al., 2017).

197

198 **2.4. Quantification of porosity from $\text{D}/(\text{D}+\text{H})$ maps**

199 The method of porosity quantification was designed to account for and take advantage
200 of lattice diffusion. Because fluid is lost after the opening of the capsule, the only trace of
201 fluid-mineral interactions is an increase of D/(D+H) ratio in the mineral with diffusion
202 profiles starting from grain boundaries and smoothly decreasing towards the interior of the
203 grains (Pilorgé et al., 2017). Typical diffusion patterns can be compared with actual grain
204 boundaries and fractures from SEM observations (Fig. 2). In this example, a thin blade of
205 antigorite that displays a continuous crystallographic orientation throughout the image
206 displays two sectors affected by intra-crystalline deuterium diffusion. On close inspection by
207 SEM (Fig. 2c), the limit between the two sectors shows nano-porosity that was as active fluid
208 path during the high-temperature stage because diffusion occurred along it. Other cracks that
209 are easily seen on SEM images crosscut the isotopic zoning and postdate it. They were likely
210 formed during decompression of the sample. This shows that isotopic exchange is a much
211 more pertinent tool to locate "active" porosity, i.e. open fluid paths, during the high-pressure
212 high-temperature experiments than SEM image analysis. In the following, instead of using the
213 isotopic profiles to obtain lattice diffusion coefficients, we use isotopic maps to infer active
214 porosity during the high-pressure high-temperature stage of the experiment when deuterium-
215 hydrogen exchange between the fluid and the rock occurs.

216 When diffusion coefficients are around 10^{-20} m²/s, the distance over which lattice
217 diffusion occurs is of the order of tens of nanometers, smaller than the Raman beam size (ca.
218 500 nm) used to map D/(D+H) ratios. In these conditions, deuterium-bearing zones observed
219 in hydrated mineral on Raman maps are identified with locations of fluid-mineral interface at
220 the time of experiments. D/(D+H) ratios are proportional to the active, i.e. fluid-filled, grain
221 boundary density (Fig. 3). We then make the assumption that higher D/(D+H) ratios
222 correspond to the creation of numerous new grain boundaries that are identified as cracks on
223 SEM images of a serpentinite experiment (Fig. 4). Corrugated cracks or grain boundaries may

224 occur and could invalidate the assumption of straight boundaries made here because of
 225 overlapping diffusion profiles. This is not the case when diffusion length is small as in the
 226 present experiments with low diffusion coefficients. SEM images indicate little corrugation
 227 and tortuosity of grain boundaries. Zones with foliation orientation subperpendicular to the
 228 sample section are dominant because of shape orientation of the rock chip or core, ensuring
 229 that a majority of grain boundaries are subperpendicular to the studied sections.

230 Conversion of $D/(D+H)$ ratios into grain boundary density per unit area is achieved by
 231 calculating synthetic $D/(D+H)$ ratios corresponding to given numbers of grain boundaries (n)
 232 in one pixel (Fig. 3b). We then use the solution of the diffusion equation for a plane sheet in
 233 contact with an infinite reservoir, considering small time (i.e. duration of experiment is small
 234 in comparison to a characteristic diffusion time) from (Cranck, 1975), to calculate diffusion
 235 profile between two grain boundaries bounding a planar element of mineral (our plane sheet):

$$236 \quad \frac{C(x)-C_0}{C_1-C_0} = \sum_{m=0}^{\infty} (-1)^m \operatorname{erfc}\left(\frac{(2m+1)l-x}{2\sqrt{Dt}}\right) + \sum_{m=0}^{\infty} (-1)^m \operatorname{erfc}\left(\frac{(2m+1)l+x}{2\sqrt{Dt}}\right) \quad (1),$$

237 with $C(x)$ the mineral $D/(D+H)$ ratio at the position x , C_0 the initial mineral $D/(D+H)$ ($=0$ in
 238 our case), C_1 the fluid $D/(D+H)$ ratio at fluid-mineral interfaces ($x=-l$ and $x=l$, i.e. at crack
 239 location, Fig. 3c), l the half thickness of the plate sheet (in m), D the diffusion coefficient (in
 240 m^2/s), and t the experiment duration (in s). This solution converges rapidly and the infinite
 241 sums may be approximated with the three first terms ($m=0$ to 3). The synthetic $D/(D+H)$ ratio
 242 of one pixel for a known amount of grain boundary (n) is then:

$$243 \quad \frac{D}{D+H} = \frac{2n}{Y} \int_{x=0}^l C(x) dx \quad (2),$$

244 with Y the pixel size (in m) and $C(x)$ the $D/(D+H)$ profile for a half space plane sheet
 245 calculated with equation (1) for $m = 0$ to 3. The integral was approximated with the trapeze
 246 method. We then calculate the evolution of $D/(D+H)$ for one pixel with the number of grain
 247 boundary per unit length using equation (2) (Fig. 3d). This relationship was then fitted with a
 248 linear function over a restricted range of grain boundary number above which the chosen

249 solution to the diffusion equation is not valid anymore (*e.g.* the last two points in Fig. 3d).
250 Actual experimental values were such that this hypothesis is verified. A different function was
251 generated for each experiment taking into account the Raman map pixel size (Y), the
252 corresponding diffusion coefficient, experiment duration and average effective C_1 (Pilorgé et
253 al., 2017). Finally, $D/(D+H)$ Raman maps are converted in grain boundary density maps using
254 the corresponding function (Fig. 3e).

255 2D porosity was calculated as the mean grain boundary density of the map multiplied
256 by the grain boundary thickness δl . δl is of the order of 0.4 nm in olivine aggregates (Hiraga
257 et al., 2004) with lattice parameters in the 0.5 - 0.8 nm range. The present minerals have more
258 complex structures, and this value is likely higher. Scaling δl with lattice parameters in the 0.5
259 - 4 nm range, we estimate δl in the 0.5 - 2 nm range, which is consistent with HRTEM images
260 of both naturally and experimentally deformed serpentine samples (Amiguet et al., 2014).

261 From experiments at various temperatures, the inter-diffusivity of deuterium-hydrogen
262 was determined in the different hydrous minerals. For antigorite and chlorite deuterium-
263 hydrogen inter-diffusivity is within the required range at 315°C ($10^{-20.6}$ and $10^{-20.7}$ m²/s
264 respectively (Ganzhorn et al., 2018; Pilorgé et al., 2017). In the case of glaucophane,
265 deuterium-hydrogen inter-diffusivity at 315°C is so low that almost no incorporation of
266 deuterium is seen in glaucophane grains after high-pressure experiments. To overcome the
267 very slow diffusion in glaucophane, we set the temperature to 500°C (G-28-500) and 450°C
268 (G-31-450) where the diffusion coefficients for glaucophane of $10^{-18.7}$ and $10^{-20.3}$ m²/s,
269 respectively, are estimated from preliminary room pressure exchange experiments between D₂
270 gas and glaucophane. Estimated uncertainties on diffusion coefficients are of one order of
271 magnitude and are taken into account in error propagation on porosity determination.

272 As a result, among some thirty experiments, only five (Table 1) could be selected
273 because they fall in the range where deuterium-hydrogen diffusion in minerals can be used to

274 estimate porosity in the rock core or chip, at temperatures of 315°C (experiments SB-4-315,
275 SC-24-315, C-25-315) for chlorite- and antigorite-dominated rocks, and of 450-500°C
276 (experiments G-28-500 and G-31-450) for the glaucophane-dominated rock. The good
277 agreement between porosity determinations at 450 and 500°C for the blueschist (Table 1)
278 with one and a half order of magnitude difference in diffusion coefficient validates the
279 approach, although the explored temperature range is small due to constraints on diffusion
280 coefficients and thermal stability of hydrated minerals.

281

282 **3. Results**

283 The present experiments simulate the deformation sequence of the seismic cycle in
284 water-saturated rocks: a short and intense pulse of cold compression representing an initial
285 earthquake is followed by a rest period with lower stress and slower deformation. Cores or
286 chips and powder of selected rocks are enclosed in a gold capsule and loaded in a belt press
287 (Fig. 1a). The first stage is a pressure and temperature increase (< 900 s) that models the
288 deformation event, with 85±5% of the deformation occurring during it as recorded by piston
289 displacement in the press (Fig. 1b). Associated strain rates are on the order of 10^{-3} s^{-1} , and
290 most of the deformation is taken up by compaction and deformation of the water-saturated
291 rock powder around rock cores or chips, which results in a large shape change of the gold
292 capsule observed after the experiments (Fig. 1a). Stresses are high enough to induce
293 deformation in the presence of excess water when rock cores are used. Texture and
294 deformation heterogeneities were also inherited from those of the starting material in the case
295 for thin rock chips embedded in rock powder. Deformation results in smallest grain sizes
296 identified with optical microscopy or SEM.

297 The second stage corresponds to a pressure and temperature plateau during which the
298 rocks interact with ambient aqueous solutions. With maximum strain rates of 10^{-5} s^{-1} during

299 this stage, stresses are constrained by plastic flow of boron nitride, the deformable material
300 surrounding the capsule, to values below 10 MPa at 315°C, and to even lower values at 500°C
301 (Pezzotti et al., 1997). These values are lower than the plastic or brittle yield stress of the
302 minerals studied here. The second stage is a suitable representation of metamorphic
303 conditions, and of the beginning of the inter-seismic relaxation period, during which samples
304 exchanged hydrogen with deuterated water during the high-temperature plateau. Isotopic
305 exchange is limited to the high-temperature stage because diffusion is negligible at ambient
306 temperature.

307 Raman mapping of the isotopic exchange observed in the recovered samples is used to
308 constrain porosity with deuterium-hydrogen inter-diffusivity in minerals close to 10^{-20} m²/s.
309 In these conditions (see above), Raman measurements of the D/(D+H) ratio are proportional
310 to the density of grain boundaries that experienced fluid-rock interaction during the
311 experiment, a method validated by comparison with SEM images (Fig. 4). D/(D+H) maps are
312 converted to grain boundary densities, and then to 2D porosities by multiplying grain
313 boundary density by grain-boundary thickness, which we assume to be in the 0.5 - 2
314 nanometer range (Amiguet et al., 2014; Hiraga et al., 2004). 2D porosities are converted in 3D
315 porosities assuming a ratio close to unity for the platelet and elongated geometry of the
316 phyllosilicates and inosilicates studied here.

317 The five studied samples show different intensities of fluid-rock exchange (Fig. 5),
318 hence different porosities of 0.2 - 2.6×10^{-2} for the blueschist, $0.1 - 6 \times 10^{-3}$ and $1 - 16 \times 10^{-3}$ for
319 serpentinite in the undeformed sample SC and deformed sample SB, respectively, and $1 -$
320 19×10^{-4} for the chlorite-schist. In sample SB, deformation is heterogeneous, and deformed
321 areas detected by SEM (P2 in Fig. 5b) displays higher porosities ($4 - 16 \times 10^{-3}$) than
322 undeformed areas ($1 - 3 \times 10^{-3}$, P1 in Fig. 5b). The largest porosities of the deformed areas of
323 the rock core are similar to the porosity of the deformed powder surrounding it (Fig. 6).

324 Values given above include uncertainties on pore width in the 0.5-2 nm range, and on
325 diffusion coefficient of one order of magnitude (Table 1). Chlorite-schist retains low porosity
326 even in the powdered part of the sample (Fig 5d). The blueschist chip shows measurable
327 porosity heterogeneity associated with deformation heterogeneity in the starting material (0.2
328 $- 1.0 \times 10^{-2}$ vs. $0.6 - 2.6 \times 10^{-2}$, Fig. 6), although smaller than observed for serpentinite SB.

329

330 **4. Discussion**

331 Differences in mechanical properties of the major rock-forming mineral can explain
332 the variety of porosity increase on deformation. Plastic yield stress exceeds 700 MPa in
333 antigorite (Hilairret et al., 2007) because of corrugated structure that hinders basal slip
334 (Amiguet et al., 2014), and 100 - 300 MPa in amphibole, *e.g.* glaucophane (Ko and Jung,
335 2015). In the presence of excess water, the yield stress of amphiboles drops down to about
336 60 ± 20 MPa because brittle failure is favored (Ko and Jung, 2015). This typical brittle yield
337 stress in the presence of excess water is commensurate with stresses expected at the
338 subduction interface of a few tens of MPa (Fagereng and den Hartog, 2016). Chlorite yields
339 plastically at similar stresses as micas, lizardite and talc, all phyllosilicates with easy slip on
340 continuous basal plane planes (Amiguet et al., 2014), in the 20 - 100 MPa range (Amiguet et
341 al., 2012; Kronenberg et al., 1990). Chlorite, micas, and talc, with their low plastic yield for
342 basal slip, are the only class of mineral capable of deforming plastically in deep subduction
343 zone conditions, therefore maintaining low porosity even for large strain and strain rates.
344 Optical and SEM observations on the recovered samples confirm that chlorite shows
345 extensive plastic deformation with smoothly bent crystals.

346 Porosities are similar in the undeformed serpentinite sample SC and in chlorite-schist,
347 and increases by one order of magnitude in the deformed areas of serpentinite sample SB. The
348 porosity difference between deformed serpentinite and chlorite-schist may be explained by

349 their different deformation mode. Bending and kinking of chlorite grains prevent the creation
350 of new porosity and chlorite-schists remain impermeable during deformation. Likewise, low
351 porosities are also expected for other phyllosilicates with continuous layers and low yield
352 stress such as micas and talc (Amiguet et al., 2014). In contrast, deformation in antigorite or
353 in glaucophane grains generates new grain boundaries, which act as open porosity, explaining
354 the one order-of-magnitude difference in porosity between deformed and undeformed
355 serpentinite (Fig. 6), and of half-an-order of magnitude in blueschists.

356 Porosities obtained in this study (Fig. 7) are similar to those of gabbro and serpentinite
357 at 100 MPa (Katayama et al., 2012), and at least two orders of magnitude higher than
358 extrapolated at 1 GPa by these authors from the trend of their low-pressure measurements.
359 Present values of porosities are about one order of magnitude lower than those measured in
360 textural equilibrium experiments on olivine or quartzite (Mibe et al., 1999; Wark and Watson,
361 1998). Once porosities are determined, permeabilities may be inferred from porosity values
362 using the Kozeny-Carman relationship (Walder and Nur, 1984):

363
$$k = k_0 \left(\frac{\phi}{\phi_0} \right)^n \quad (1),$$

364 where ϕ is the porosity corresponding to the permeabilities k , with ϕ_0 and k_0 being
365 reference values. The exponent n in equation (1) take different values depending on rock type
366 and pore geometry. Values for reference porosity ($\phi_0=0.005$) and permeability ($k_0=10^{-20.1}$) and
367 $n=2$ were anchored on those from previous studies on similar rocks as those studied here
368 (Katayama et al., 2012). Different values and exponent of $n=3$ are obtained in synthetic
369 quartzite (Fig. 7a) because pores have well-equilibrated shapes at triple junctions and can be
370 described by a wetting angle (Wark and Watson, 1998). This is not the case in the present
371 study and in that of Katayama et al. (2012) where sharp and straight grain boundaries
372 dominate due to the easy cleavage of minerals and low temperatures that do not allow textural

373 equilibration. Permeabilities are estimated in the 10^{-24} - 10^{-18} m² range (Fig. 7a), with
374 uncertainties of the same order of magnitude as those obtained from modeling of fluid or heat
375 flow in the continental crust (Ingebritsen and Manning, 1999; Ingebritsen and Manning,
376 2010) or from measurements on rock cores from deep drilling (Shmonov et al., 2003).

377 Rock permeabilities of subduction zone rocks from the present study can be compared
378 with those of crustal rocks as a function of depth (Fig. 7b) in stable metamorphic conditions
379 and after earthquakes (Ingebritsen and Manning, 1999; Ingebritsen and Manning, 2010;
380 Shmonov et al., 2003). Values for blueschists fall within the trend for stable continental crust,
381 and those for antigorite serpentinite SB and chlorite schist are lower than 10^{-21} m², indicating
382 that these rocks are practically impermeable. Low permeabilities were also inferred from
383 hydraulic studies in metamorphic terrains (Sanford, 2017). Values for highly deformed areas
384 in serpentinite SB are similar to those of the blueschist, indicating that porosity and
385 permeability increase by about one and two orders of magnitude, respectively, with
386 deformation. This difference is similar to that inferred between stable continental crust rocks
387 and fault zones after earthquakes (Ingebritsen and Manning, 2010), but absolute values are
388 lower for serpentinites than for continental crust.

389 The present high-pressure determination of porosity and permeability offers a new
390 perspective on the distribution of seismicity in subduction zones at forearc depths (35 - 90
391 km). Low frictional coefficient or high transient pore-fluid pressure required in order to
392 generate earthquakes call for a control of seismic activity by a porosity cycle (Sleep and
393 Blanpied, 1992) with porosity in the range of 1×10^{-3} - 1×10^{-2} depending on the model. The
394 present determinations of porosity show that serpentinites experience the largest porosity
395 variations with deformation compatible with the range for seismic rupture defined above. In
396 blueschist, porosity variation is less pronounced, but sufficient to facilitate seismic activity.
397 Hence deformed serpentinite, and possibly blueschist, may facilitate the propagation of

398 rupture near the plate interface down to depths of 60-80 km, as observed for large shocks (e.
399 g. Tohoku-oki 3/2011, Sumatra 12/2004) and aftershocks (Lay et al., 2012).

400 Chlorite schists do not show significant porosity variation with deformation, and their
401 low porosity makes the metasomatic plate interface a likely barrier for fluid migration. Low
402 permeability layers at the interface hamper massive upward migration of the aqueous fluids
403 produced by dehydration the subducted crust and limit the serpentinization of the mantle
404 wedge (Fig. 8). The thin layer acting as a seal is expected to break up during large
405 earthquakes, which would allow transient fluid migration from the subducted crust into the
406 forearc mantle wedge and local serpentinization (Fig. 8). Mantle wedge porosity and
407 permeability are expected to vary with the deformation of serpentinites (Fig. 7), especially
408 near the deforming interface where it should increase.

409 Observations of low-velocity layers in subducted crust suggest that the oceanic crust is
410 over-pressurized down to a depth of 35 - 40 km in hot subduction zones (Audet et al., 2009;
411 Peacock et al., 2011) and 90 km in cold subduction zone (Shiina et al., 2013). The porosity of
412 the subducted oceanic crust deduced from seismic-wave velocities is in the range $1 - 4 \times 10^{-2}$
413 (Peacock et al., 2011; Shiina et al., 2013), in agreement with the value of up to 2.6×10^{-2}
414 obtained in the present experiments on blueschists.

415 Over-pressure in subducted crust requires the presence of a low permeability seal
416 ($10^{-24} - 10^{-21} \text{ m}^2$) at the plate interface (Audet et al., 2009; Peacock et al., 2011). According to
417 these authors, such a seal forms by grain-size reduction during shearing or by precipitation of
418 platy minerals such as clay, serpentine or micas. Permeabilities inferred for the sealed
419 subduction interface are similar to those assessed here both for chlorite-schist ($k \approx 10^{-24} -$
420 $10^{-21.5} \text{ m}^2$) and undeformed serpentinite ($k \approx 10^{-24} - 10^{-20.5} \text{ m}^2$; Fig. 7). Serpentinite is likely
421 deformed near the subduction interface, hence its porosity would be too high to seal the
422 interface, unless sealing by pressure-solution occurs at timescales larger than those of the

423 present experiments. We infer that the seal may correspond to a one to several meter-thick
424 chlorite-rich layer presents down to around 45 km in hot subduction zones (such as Cascadia)
425 and down to 90 km in cold ones (such as NE Japan, Fig. 8), consistent with the predicted
426 stability limits of chlorite along the subduction interface for the two different thermal regimes
427 (Abers et al., 2013). Chlorite-schist layers are commonly observed in ophiolites at the
428 metasomatic contact between serpentinites and metamorphosed oceanic crust (Spandler et al.,
429 2008). Pelitic metasediments and metasomatic talc-schists are lithologies with weak
430 phyllosilicates behaving like chlorite that could also contribute to seal the subduction
431 interface.

432 The presence of a low permeability seal at the interface down to about 80 km in the
433 cold Tohoku subduction zone accounts for three additional seismological observations.
434 Firstly, the high seismic velocities observed in the forearc mantle indicate limited
435 serpentinization and effective plate sealing (Abers et al., 2017). Secondly, release of fluids to
436 the mantle wedge is detected below this depth (Kawakatsu and Watada, 2007). Thirdly, updip
437 fluid migration from the blueschist-eclogite dehydration transition in the oceanic crust can
438 account for high seismicity in the shallower blueschist facies conditions (Kita et al., 2006).
439 Updip fluid migration and overpressured crust may favor the occurrence of large aseismic slip
440 preceding the magnitude 9.0 Tohoku-oki earthquake (Kato et al., 2012).

441 In young and hot subduction zones such as Cascadia or Shikoku, the presence of
442 thicker metasomatic zones with low-porosity chlorite-type phyllosilicates at depths around 30
443 to 40 km may favor the occurrence and observation of tremors (Ide, 2012). The high seismic
444 velocities observed worldwide in the forearc mantle of subduction zones hint at limited
445 serpentinization and effective plate sealing, with the two notable exceptions of Cascadia and
446 Costa Rica (Abers et al., 2017). This is an indication of intense fluid flow through the
447 interface of Cascadia, in apparent contradiction with thicker seal than in cold subduction

448 zone. The large fluid production in hot Cascadia may efficiently break the seal, leading to
449 complex zonation of the mechanical behavior of the interface (Audet and Schaeffer, 2018)
450 and serpentinization of the mantle wedge. In any case, the present determinations of porosities
451 and permeabilities provide experimental values for the essential parameters of mechanical and
452 geodynamical models describing fluid flow in dipping lithologic layers of subduction zones
453 (Morishige and van Keken, 2018).

454

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466

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578 **Figure captions**

579

580 Fig. 1. Experimental procedure. a. Cartoon of the experimental procedure. b. Piston
581 displacement (blue), temperature (red) and pressure (green) curves as recorded during the C-
582 25-315 experiment. Step 1: Pressure increase at ambient temperature. Step 2: Increase of
583 temperature at high-pressure. 80-90% of the strain occurs during steps 1 & 2 (blue dashed line
584 for C-25-315 and shaded grey area for all experiments), mimicking large strain rates
585 associated with an earthquake. Step 3: Pressure and temperature plateau where deuterium-
586 hydrogen exchange occurs between rock and fluid. Step 4: Piston displacement is only
587 slightly recovered after decompression, demonstrating the large irreversible deformation.

588

589 Fig. 2. Isotopic zonation by Raman mapping and texture by SEM of an antigorite sample
590 exchanged with D₂O at 540°C and 3 GPa for 38 hours (redrawn from Pilorgé et al., 2017). a.
591 Raman map of the D/(D+H) ratio, the dashed lines indicate the approximate outline of a large
592 single crystal of antigorite. b. SEM image, ellipses show the approximate location of diffusion
593 zoning observed in a. The two diffusion domains are separated by very fine porosity (green
594 line). c. high magnification SEM image showing nanoporosity that was active during isotopic
595 exchange at high temperature. In the opposite, cracks visible in b. that cross-cut the isotopic
596 zonation (red lines) were created after quenching, most likely during decompression.

597

598 Fig. 3. Determination of the grain boundary density. a. Initial D/(D+H) data. b. Schematic
599 geometry of one pixel with several new grain boundaries. c. Corresponding schematic
600 diffusion profile. C_1 is the D/(D+H) ratio of the fluid. $C_0 \sim 0$ is the initial ratio in the mineral. l
601 is the half thickness of the plane sheet model used to calculate the diffusion profile. d.
602 Example of synthetic relationship between D/(D+H) and the number of grain boundaries (n)
603 for one pixel of size Y (for conditions of experiment SB-4-315), and the linear fit used. e.

604 Final map of grain boundary density calculated for SB-4-315. White on the color scale
605 corresponds to discarded spectra for saturated signals.

606

607 Fig. 4. BSE images of heterogeneously deformed antigorite laths in experiment SB-4-315. a.
608 Zone of deformed antigorite laths. b. Zoom-in on a deformed area showing numerous parallel
609 cracks forming new grain boundaries. c. Zoom-in on an undeformed area. d. Zoom-in on a
610 deformed area showing fan-shaped cracks. e. Corresponding $D/(D+H)$ map from Fig. 3.

611

612 Fig. 5. $D/(D+H)$ and grain boundary density maps (first and second image, respectively) for
613 blueschist G (a), deformed serpentinite SB (b, P1: almost undeformed zone; P2: highly
614 deformed zone), undeformed serpentinite SC (c), and the chlorite schist C (d). The grain
615 boundary density and $D/(D+H)$ color scales are the same for all maps. White pixels are
616 discarded from analysis due to low signal/noise ratio. (e) Histograms corresponding to grain
617 boundary maps for the four experiments. Red dashed line: mean density used to calculate
618 porosity.

619

620

621 Fig. 6. Maps of grain boundary density at the capsule scale. Dashed lines: cores contours. The
622 color scale is the same as in Fig. 5. a. blueschist (G-28-500). White arrow indicates a band of
623 higher porosity zone in the blueschist chip. Porosity is the highest in the powder. b.
624 serpentinite (SB-4-315). Serpentinite chip shows heterogeneities due to localized
625 deformation. Powder appears dark due to low signal associated with poorly polished surface.
626 Location of high-resolution maps in Fig. 5 is shown.

627

628 Fig. 7. a) Permeability-porosity diagram, with present porosity data in blue: blueschist; dark
629 green: chlorite schist; medium green: antigorite serpentinite SC; light green: deformed
630 antigorite serpentinite SB. Dashed arrows: trend low-pressure data for gabbro (orange) and
631 serpentinite (green), respectively, define a Kozeny-Carman relationship with typical exponent
632 $n=2$ (Katayama et al., 2012; Kawano et al., 2011) used to estimate permeabilities from the
633 present porosity determinations. Permeabilities of the different rocks are slightly offset for
634 readability. Red: high-pressure data on textural equilibrium experiments on quartzite with
635 typical exponent $n=3$ (Wark and Watson, 1998). Grey area: impermeable rock domain. b)
636 Comparison of estimated permeabilities in a) with those based on hydraulic and heat flow
637 measurements in stable continental crust (Ingebritsen and Manning, 1999), solid and long-
638 dashed black lines, after earthquakes (Ingebritsen and Manning, 2010), grey lines, and
639 measurements on deep core rocks (Shmonov et al., 2003), short-dashed black line.
640 Uncertainties are shown as vertical bars on the right of the diagram.

641
642 Fig. 8. Schematic model of water circulation in hot (a) and cold (b) subduction zones taking
643 into account the present porosity and permeability determinations. a. Down to 40 km, the
644 subduction interface corresponds to a low porosity layer (*e.g.* chlorite schists) that act as a
645 seal leading to overpressure within the subducted crust and hampering fluid flow in the
646 mantle corner. Fluids, generated in large quantities especially at the eclogite (E) – blueschist
647 (BS) transition, can penetrate the cold corner of the mantle wedge at greater depths leading to
648 serpentinization of the mantle, as evidenced in the Cascadia subduction zone. b. The
649 subduction interface is sealed down to 80-90 km hampering fluid flow in the mantle corner
650 (seal thickness of a few meters to a few tens of meters is exaggerated). Fluid flows upward
651 within the high porosity blueschist causing earthquakes (Kita et al., 2006) and fluid
652 overpressure (Shiina et al., 2013) in the crust as observed in NE Japan. Fluid flow across the

653 subduction interface may occur only after earthquake that eventually break the seal, leading to
654 weak localized serpentinization of the mantle corner, or to fluid release to the mantle below
655 80-90 km depth (Kawakatsu and Watada, 2007). GS: greenschist facies; BS: blueschist facies;
656 E: eclogite facies.

657

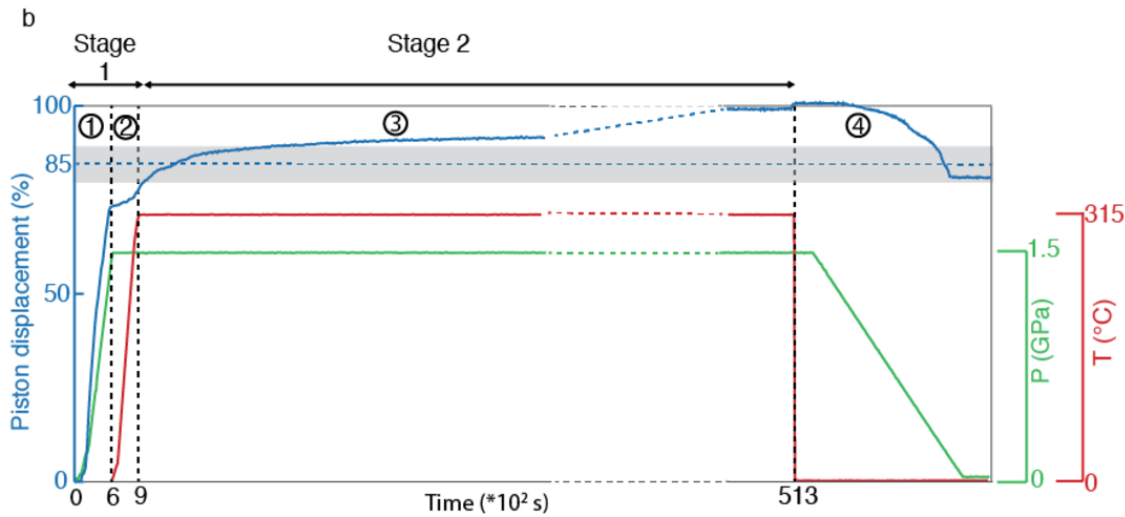
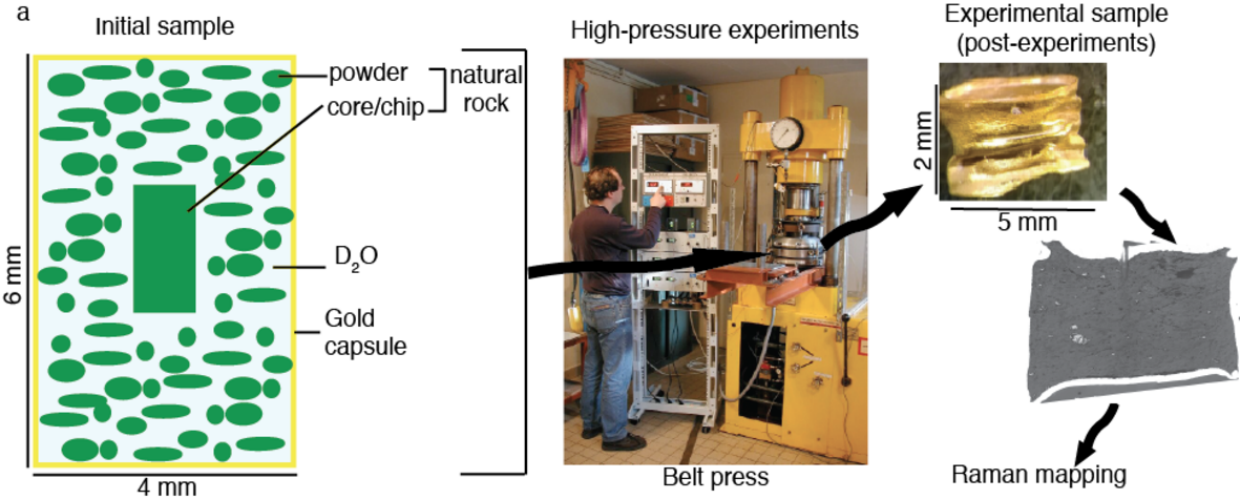
658

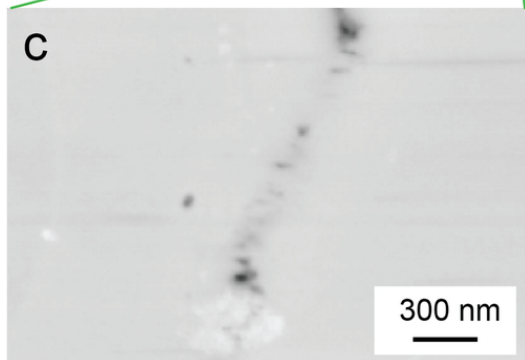
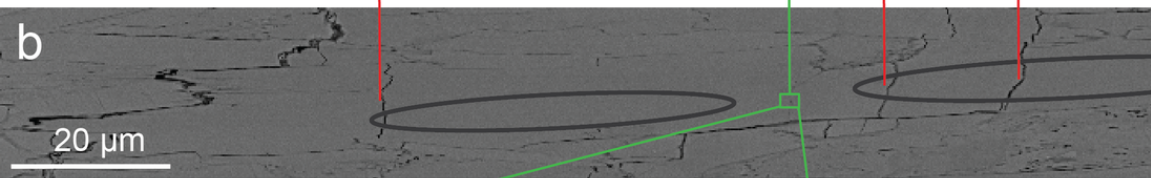
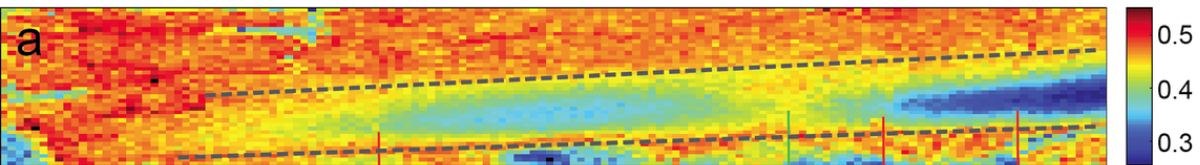
659 Table 1. Experimental conditions for the experiments used for porosity quantification
 660 assuming pore width in the range 0.5-2 nanometers and one order of magnitude uncertainty in
 661 diffusion coefficients.

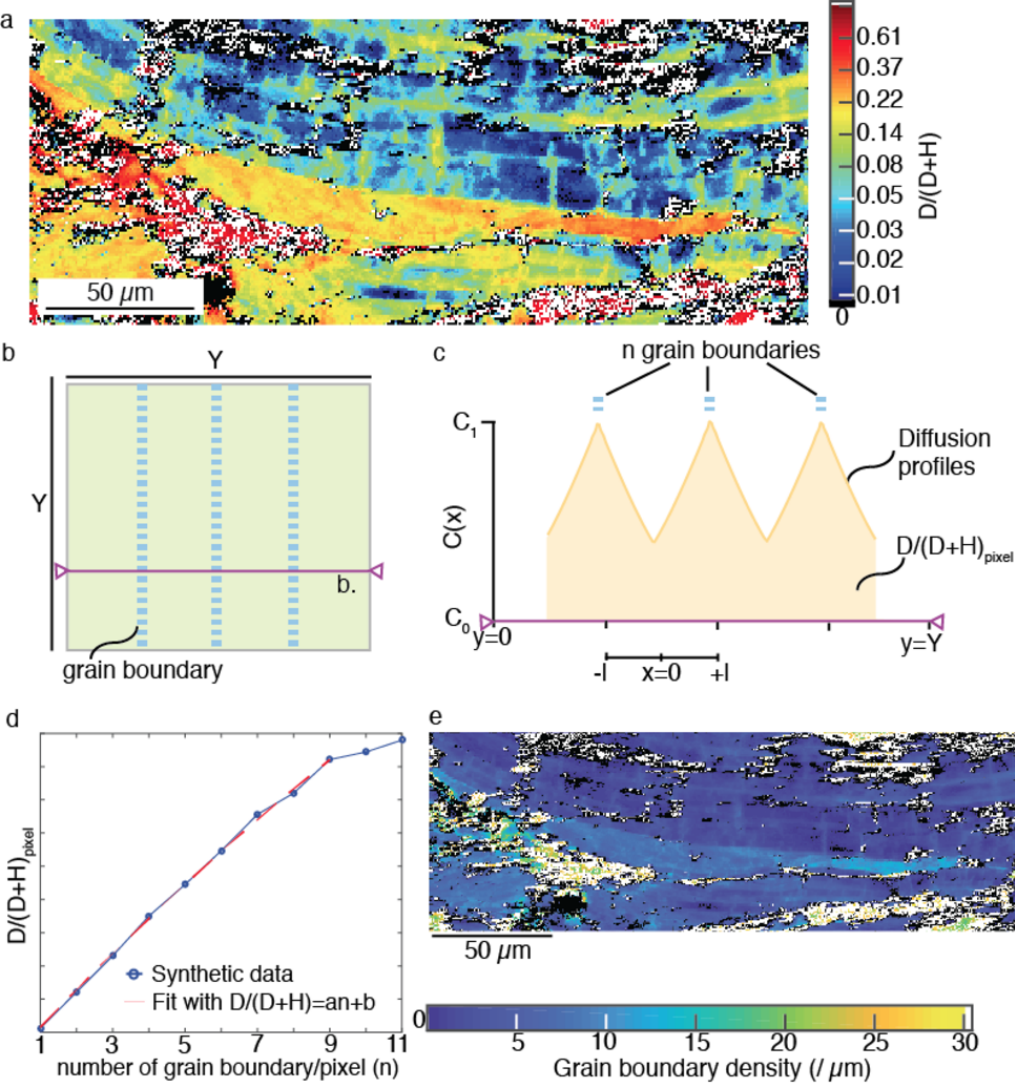
Name	Starting material	T (°C)	P (GPa)	Duration (h)	□□□□□□□□ (%)
SB-4-315	Atg (SB)	315	3	12	0.1-1.6
SC-24-315	Atg (SC)	315	1.5	14	0.01-0.6
C-25-315	Chl (SZ24)	315	1.5	14	0.01-0.2
G-28-500	Gl, Ep (G)	500	2.5	14	0.2-2.6
G-31-450	Gl, Ep (G)	450	2.5	48	0.3-2.1

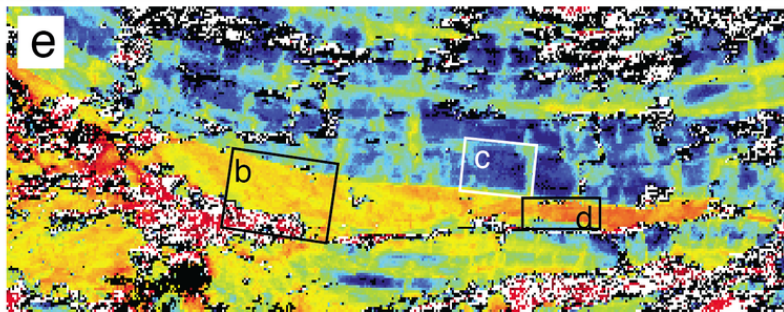
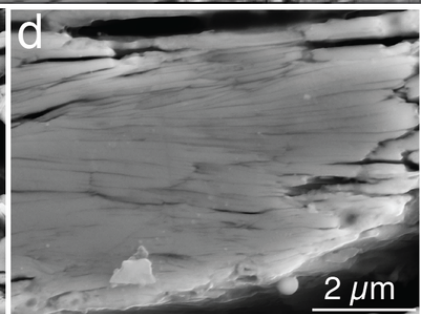
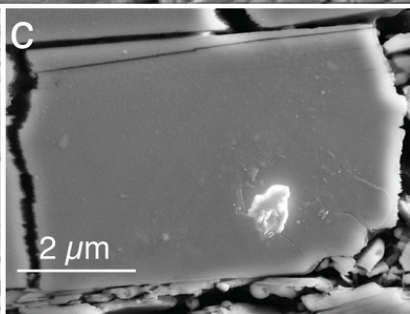
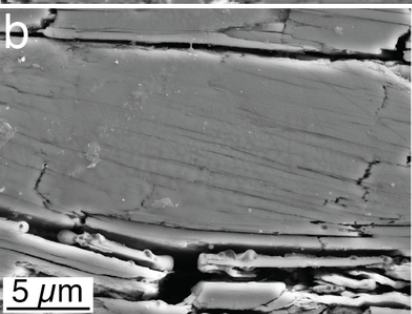
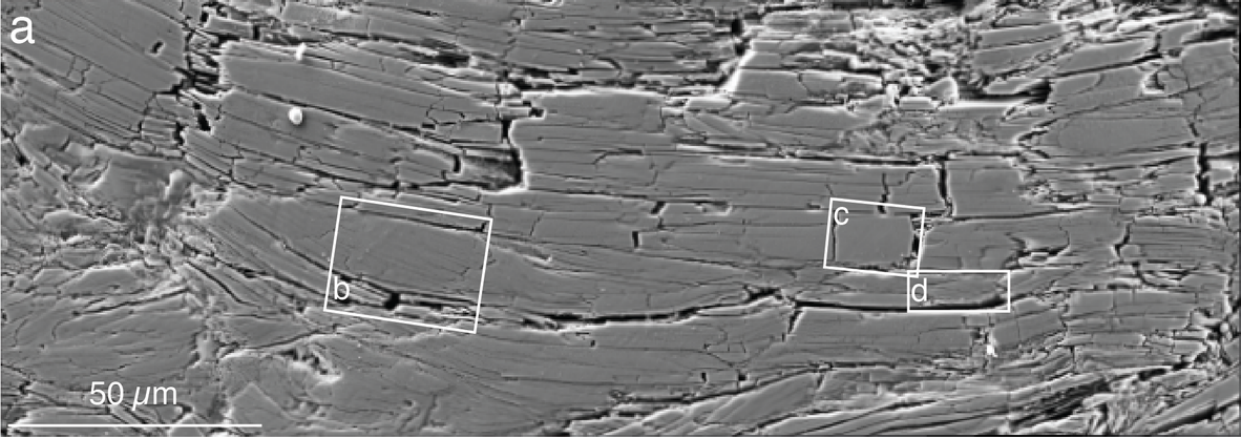
662 Atg: antigorite. Chl: Chlorite. Gl: glaucophane. Ep: Epidote.

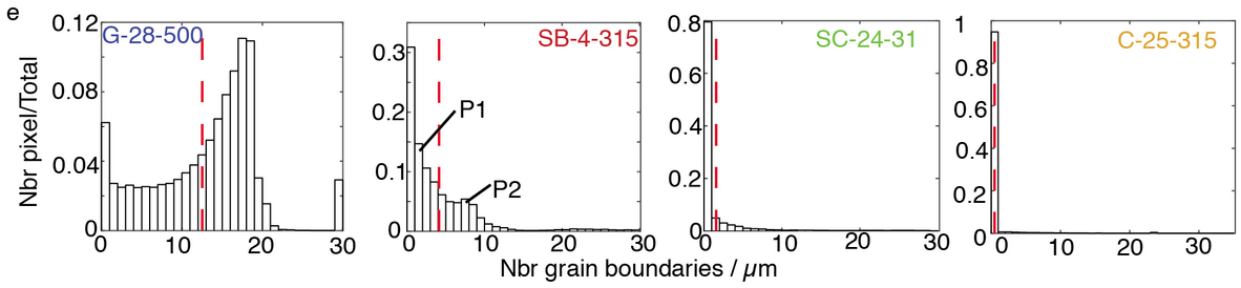
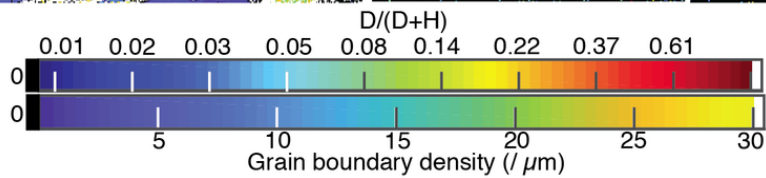
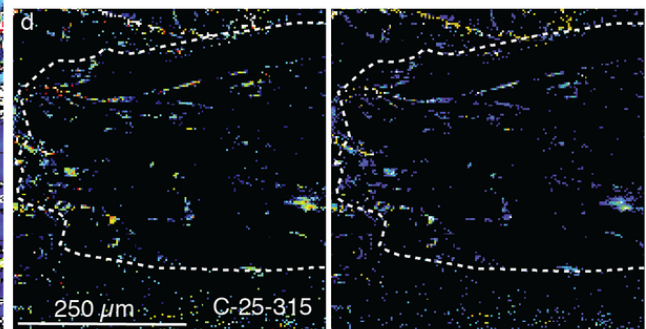
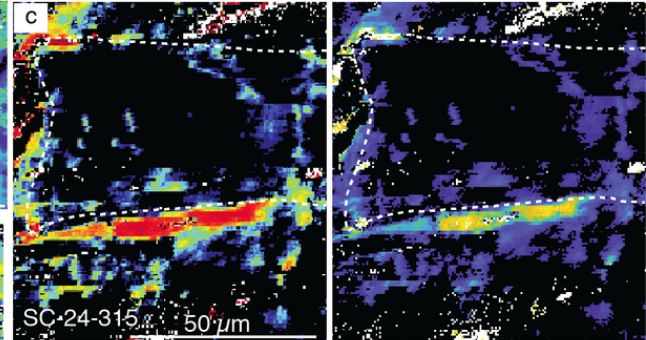
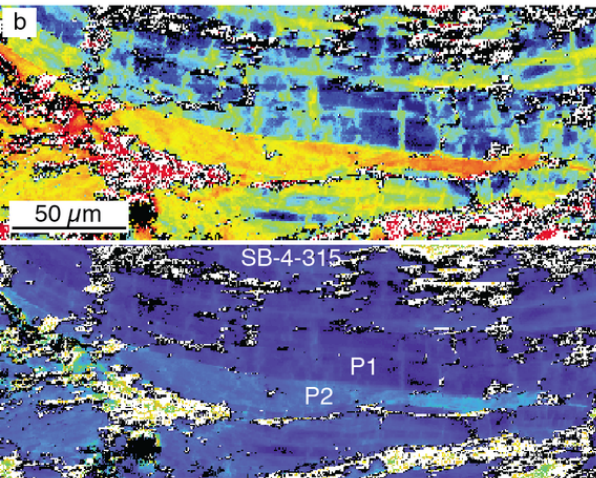
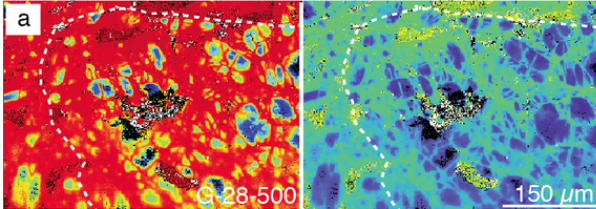
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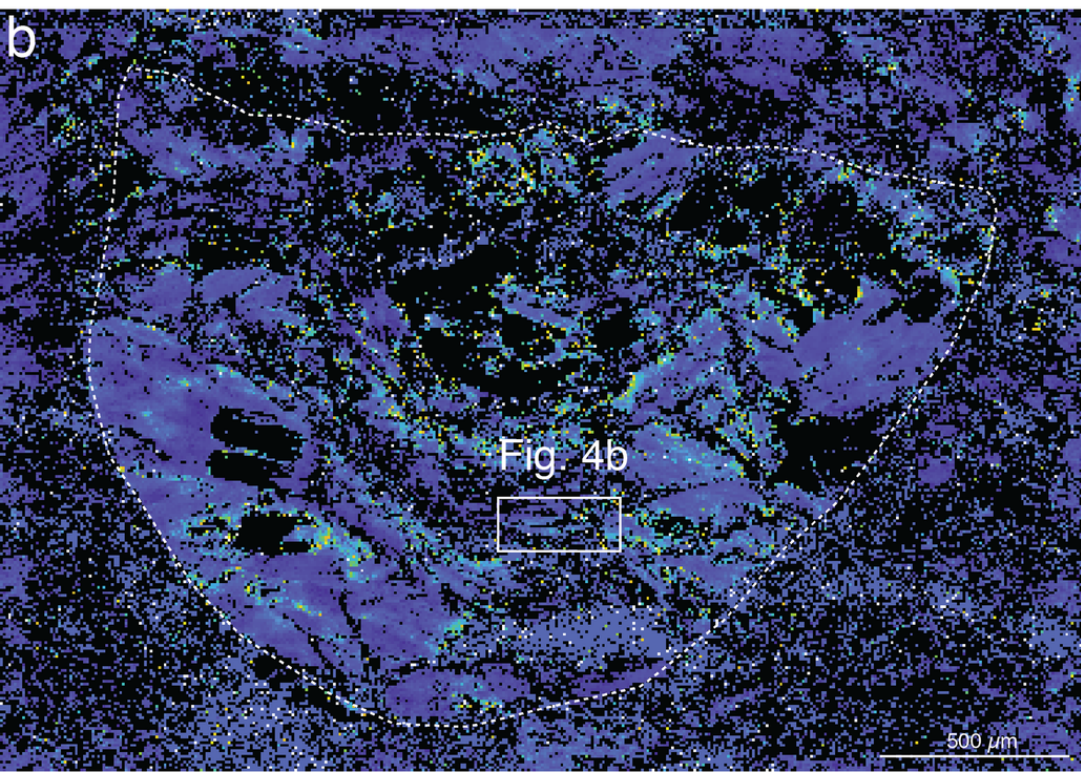
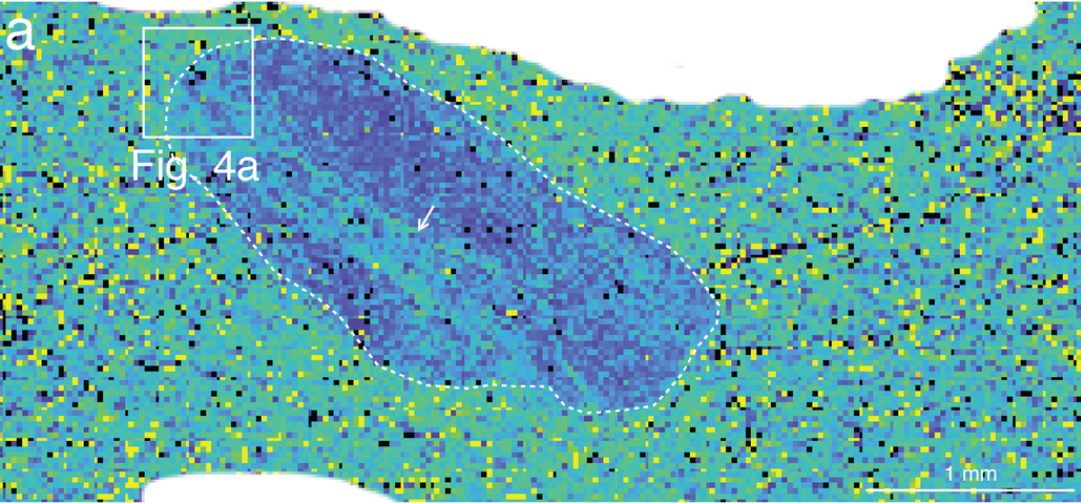


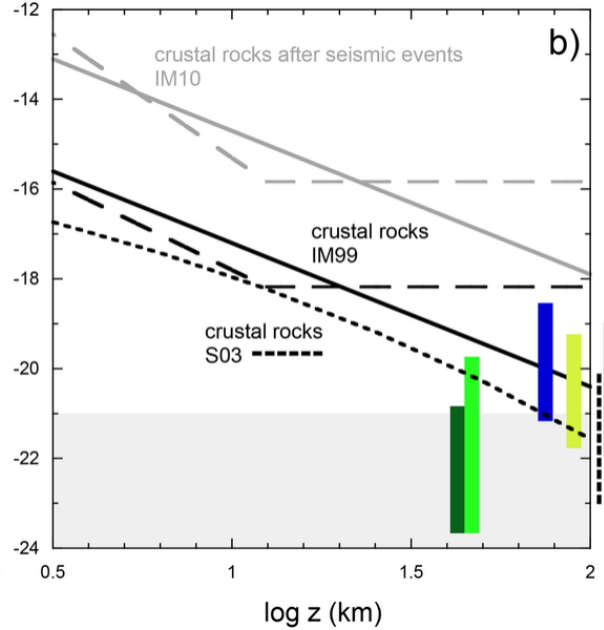
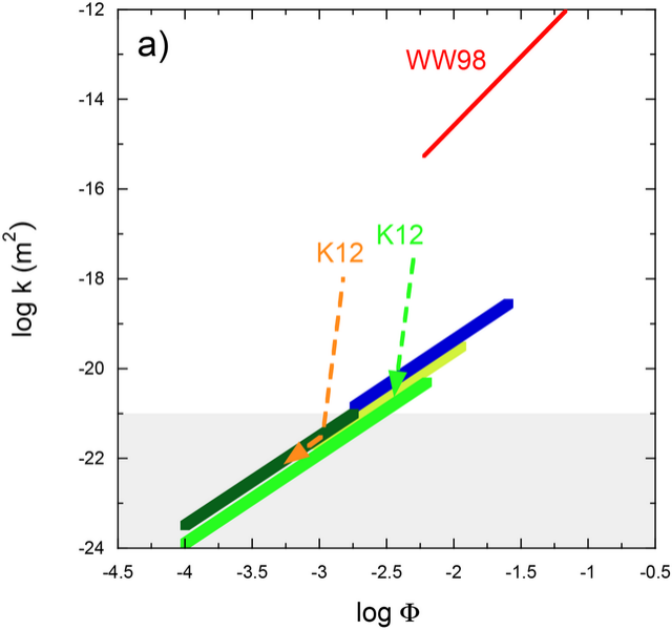




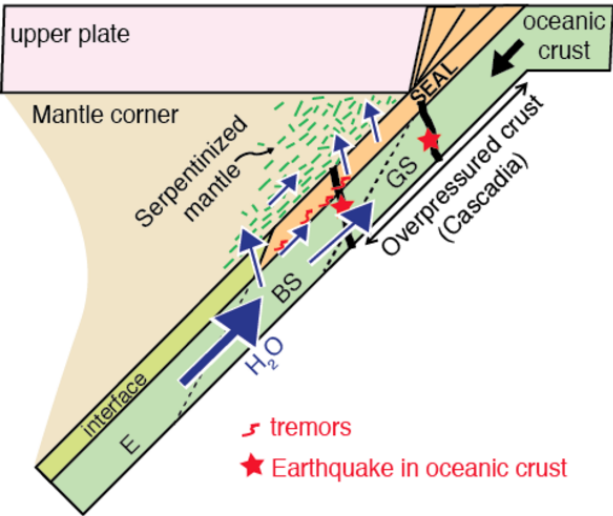








a Hot subduction zones



b Cold subduction zones

