

1                   **Validation of subgrain-size piezometry as a tool for**  
2                   **measuring stress in polyminerale rocks**

3                   **Rellie M. Goddard<sup>1\*</sup>, Kathryn M. Kumamoto<sup>2</sup>, Lars N. Hansen<sup>3</sup>, David Wallis<sup>4</sup>,**  
4                   **Andrew J. Cross<sup>1</sup>, and Christopher A. Thom<sup>5</sup>**

5                   <sup>1</sup>Department of Geology and Geophysics, Woods Hole Oceanographic Institution, Woods Hole, MA, USA

6                   <sup>2</sup>Lawrence Livermore National Laboratory, Livermore, CA, USA

7                   <sup>3</sup>Department of Earth and Environmental Sciences, University of Minnesota, Minneapolis, MN, USA

8                   <sup>4</sup>Department of Earth Sciences, University of Cambridge, Cambridge, UK

9                   <sup>5</sup>Rhenium Alloys, Inc., North Ridgeville, OH, USA

10                   **Key Points:**

- 11                   • Subgrain sizes are insensitive to the presence of secondary phases in polyminerale  
12                   rocks.
- 13                   • The grain size of a rock influences what portion of the deformation history the sub-  
14                   grain size records.
- 15                   • Stresses estimated from individual phases in a mixture do not necessarily record the  
16                   bulk stress applied to the rock.

---

\*Current address, Department of Earth, Environmental and Geographic Sciences, The University of British Columbia, 4237 University Way, Kelowna, BC V1V 1V7, Canada

Corresponding author: Rellie M. Goddard, [rellie.goddard@gmail.com](mailto:rellie.goddard@gmail.com)

**Abstract**

We deformed samples with varied proportions of olivine and orthopyroxene in a deformation-DIA apparatus to test the applicability of subgrain-size piezometry to polymineralic rocks. We measured the stress within each phase *in situ* via X-ray diffraction during deformation at a synchrotron beamline. Subgrain-size piezometry was subsequently applied to the recovered samples to estimate the stress that each phase supported during deformation. For olivine, the final *in-situ* stresses are consistent with the stresses estimated via subgrain-size piezometry, both in monomineralic and polymineralic samples, despite non-steady state conditions. However, stress estimates from subgrain-size piezometry do not reliably record the *in-situ* stress in samples with grain sizes that are too small for extensive subgrain-boundary formation. For orthopyroxene, subgrain boundaries are typically sparse due to the low strains attained by orthopyroxene in olivine-orthopyroxene mixtures. Where sufficient substructure does exist, our data supports the use of the subgrain-size piezometer on orthopyroxene. These results do, however, suggest that care should be taken when applying subgrain-size piezometry to strong minerals that may have experienced little strain. Stresses estimated by X-ray diffraction also offer insight into stress partitioning between phases. In mixtures deformed at mean stresses  $> 5$  GPa, orthopyroxene supports stresses greater than those supported by olivine. This stress partitioning is consistent with established theory that predicts a slightly higher stress within a 'strong' phase contained in a material consisting of interconnected weak layers. Overall, these results demonstrate that subgrain-size piezometry is a valuable tool for quantifying the stress state of polymineralic rocks.

**Plain Language Summary**

To measure the stresses previously supported by exhumed lower-crustal and upper-mantle rocks, we rely on examining features in the rocks that have a calibrated relationship with stress. One such metric is based on the size of subgrains, which are distinct crystallographic regions within individual grains that comprise a rock. Experiments on rocks composed of a single mineral have shown the subgrain size to have an inverse relationship with stress. However, the majority of rocks are composed of multiple minerals. In addition, subgrain sizes generated in the experiments used in the calibrations each record a single, steady-state stress, whereas natural rocks are often subject to complex stress histories.

48 We test the applicability of using subgrain size to estimate stress in rocks containing  
49 more than one mineral through high-temperature, high-pressure experiments paired with  
50 *in-situ* X-ray diffraction. Results indicate that stresses estimated from subgrain sizes in  
51 rocks composed of more than one mineral are a good indication of the final stress experi-  
52 enced by a sample regardless of the deformation history. However, additional parameters,  
53 such as the amount of strain and the grain size relative to the subgrain size, need to be  
54 considered when applying this method to natural rocks.

## 55 **1 Introduction**

56 Paleostress estimates from exhumed rocks provide critical insight into the mechanical  
57 state of Earth's lithosphere (e.g., Kohlstedt & Weathers, 1980), strain localisation along plate  
58 boundaries (e.g., Ambrose et al., 2018; Boutonnet et al., 2013), and elastic loading of the  
59 mid-crust through the earthquake cycle (e.g., Trepmann & Seybold, 2019). Such stresses  
60 can be estimated via paleopiezometry, that is, the relationship between stress and specific  
61 microstructural features (e.g., Nicolas, 1978; Tullis, 1979). The most widely implemented  
62 paleopiezometer is based on the mean size of dynamically recrystallised grains, which  
63 follows an inverse power law with differential stress in monomineralic rocks (e.g., Karato et  
64 al., 1980; Luton & Sellars, 1969; Shimizu, 2008; Stipp & Tullis, 2003; Twiss, 1986). However,  
65 grain sizes in a polymineralic rock can be influenced by the presence of secondary phases  
66 that pin migrating grain boundaries (Cross & Skemer, 2017; Evans et al., 2001; Hiraga et  
67 al., 2010; Smith, 1948; Tasaka et al., 2017). Consequently, grain-size piezometry tends to  
68 overestimate paleostresses when applied to well-mixed polymineralic rocks (e.g., Hansen  
69 & Warren, 2015).

70 An alternative proxy for paleostress in polymineralic rocks is subgrain size (Twiss,  
71 1986). Subgrains are crystallographic regions enclosed by low-angle (typically  $< 10\text{--}15^\circ$ )  
72 boundaries within individual grains (Karato, 2012, page 94). As with grain size, an inverse  
73 relationship exists between differential stress and the spacing of subgrain boundaries (e.g.,  
74 Durham et al., 1977; Karato et al., 1980; Raleigh & Kirby, 1970; Servi et al., 1952; Toriumi,  
75 1979). However, subgrain-size piezometry offers three potential advantages over grain-  
76 size piezometry. First, low-angle boundaries have lower mobilities than grain boundaries  
77 (e.g., Huang et al., 2000) and are therefore more resistant to post-kinematic static annealing  
78 (Qin et al., 2003). Second, mean subgrain sizes exhibit the same relationship with stress in  
79 both relict and recrystallized grains (see Figure 8 in Ross et al., 1980, and Figure 6 in Trimby

80 et al., 1998) meaning no distinction is required between the two grain populations. Finally,  
81 for a given stress, subgrain size is thought to be unaffected by the presence of secondary  
82 minerals (Hansen & Warren, 2015; White, 1979).

83 Recently Goddard et al. (2020) calibrated a new subgrain-size piezometer using elec-  
84 tron backscatter diffraction (EBSD), a widely used technique for characterising microstruc-  
85 tural features. This new piezometer was calibrated using both olivine and quartz, resulting  
86 in a generalised relationship that could potentially be applied to other minerals without  
87 the need for additional calibration. Subgrain size therefore provides a powerful tool for  
88 estimating stress from natural polymineralic rocks. Nevertheless, two key uncertainties  
89 remain. First, although subgrain sizes should be unaffected by the presence of secondary  
90 phases as they are intragranular features (Hansen & Warren, 2015; White, 1979), no study  
91 has directly tested this idea. Second, it is unclear how subgrain size relates to stress under  
92 non-steady-state conditions. Nearly all piezometers are calibrated using experiments in  
93 which stress has reached a steady state (e.g., Karato et al., 1980; Stipp & Tullis, 2003), and  
94 thus the statistics of microstructural features are likewise assumed to be invariant with fur-  
95 ther strain. Recent work has been conducted to explore how grain size responds to changes  
96 in stress (e.g., Kidder et al., 2016; Soleymani et al., 2020), however, the equivalent published  
97 work on subgrain-size piezometry is limited and inconclusive. As natural rocks often un-  
98 dergo significant stress changes, for example during exhumation (Behr & Platt, 2011) or  
99 during the seismic cycle (e.g., Campbell & Menegon, 2019; Menegon et al., 2021), under-  
100 standing how subgrain sizes respond to changing stresses is vital to accurately interpreting  
101 the rock record.

102 To address these questions, we conducted experiments on synthetic polymineralic  
103 samples of olivine and orthopyroxene using a deformation-DIA (D-DIA) apparatus at  
104 beamline 6-BM-B of the Advanced Photon Source synchrotron, Argonne National Labo-  
105 ratory, Illinois. To explore the extent to which subgrain size tracks stress in non-steady-  
106 state systems, we incorporated temperature and strain-rate steps into most experiments.  
107 We compared stresses measured *in situ* via X-ray diffraction to those estimated from EBSD  
108 maps of recovered samples and the subgrain-size piezometer of Goddard et al. (2020). In  
109 addition, we explored how stress partitioning between phases in the polymineralic aggre-  
110 gates compared to Handy's (1994) model for two-phase flow.

111 Our results support the hypothesis that subgrain sizes are unaffected by the presence  
112 of secondary phases. The stresses estimated from subgrain-size piezometry are consistent  
113 with the final stresses measured *in-situ*, irrespective of the stress histories that preceded  
114 them. Stress partitioning within individual phases is also found to be consistent with that  
115 expected from models of like microstructures. As such, this study broadens the applica-  
116 bility of subgrain-size piezometry to polymineralic natural rocks. However, we also cau-  
117 tion that careful consideration should be taken when using the subgrain-size piezometer  
118 to infer the bulk strength of polymineralic rocks, especially in low-strain or fine-grained  
119 samples.

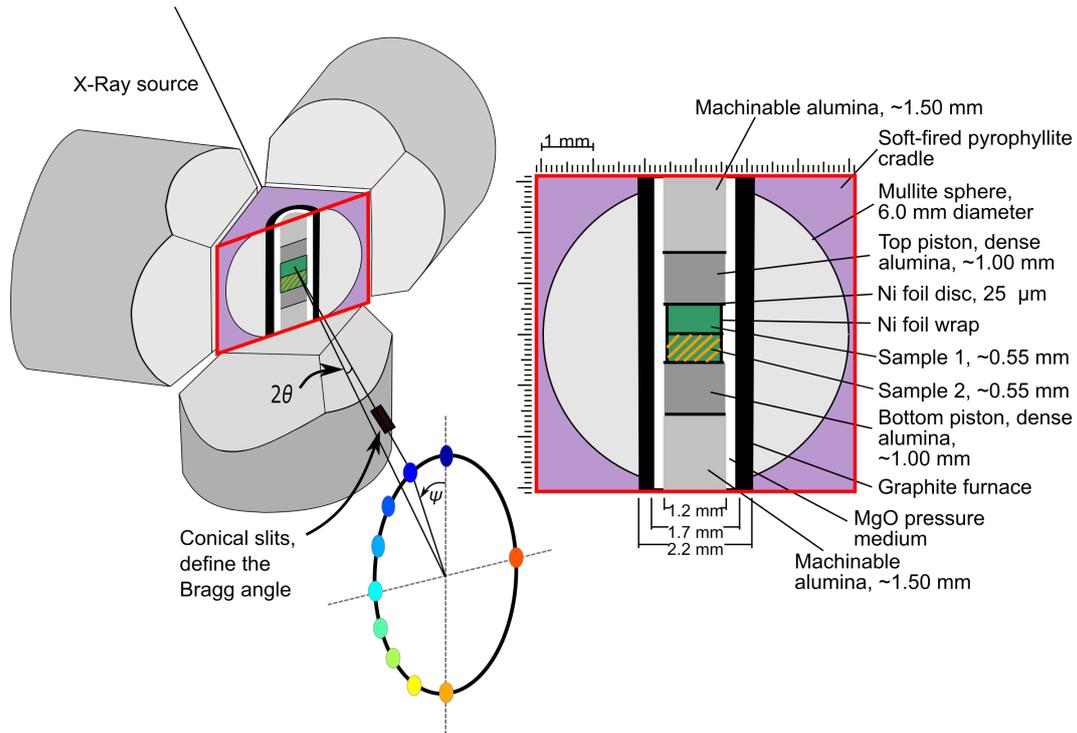
## 120 2 Methods

### 121 2.1 Sample Synthesis

122 D-DIA experiments consisted of a stack of two samples deformed in series. Figure 1 il-  
123 lustrates a typical sample assembly. One of the samples was commonly olivine, which was  
124 used as a stress sensor in the experiments. Given the use of olivine to calibrate the original  
125 subgrain-size piezometer, these monomineralic samples were also used as a control when  
126 comparing stress measurements from X-ray diffraction to those from piezometry. Other  
127 samples consisted of olivine orthopyroxenite or harzburgite, which contained 85 vol.%  
128 and 30 or 50 vol.% of orthopyroxene, respectively.

129 Samples were synthesised either at Brown University or the University of Minnesota.  
130 LT-DrySC, a dry olivine sample, was synthesised at Brown University from San Carlos  
131 olivine with particle sizes 10–32  $\mu\text{m}$ . This dry olivine was made from a ‘wet olivine’, which  
132 had  $\sim 0.5\text{--}0.7$  wt% deionised water added to the olivine powder prior to hot pressing. Pow-  
133 ders were sealed in a nickel jacket and hot pressed at a confining pressure of 1.3 GPa and  
134 temperature of 900°C for 24–26 hours in a triaxial, Tullis-modified Griggs apparatus, us-  
135 ing solid NaCl as the confining medium. To dehydrate, the sample was placed in a 1 atm  
136 CO/CO<sub>2</sub> furnace at 1000°C and a  $P_{\text{O}_2}$  of  $\sim 10^{-10}$  atm for 10 hours.

137 At the University of Minnesota, samples were synthesised from powders of Bamble  
138 enstatite and San Carlos olivine, with particle sizes  $< 10$   $\mu\text{m}$ . For samples 33 and PT-1166,  
139 powders of olivine were combined with 1% enstatite to buffer the silica activity. For sample  
140 PT-1299HiFe, enstatite was mixed in ethanol with olivine powder in a 30:70 ratio. For PI-  
141 2056, powders of enstatite and pure olivine were similarly prepared, as well as 50:50 and



**Figure 1.** Schematic illustration of a typical D-DIA sample assembly, modified from Durham et al. (2002) and Hansen et al. (2019).

142 85:15 ratios of enstatite to olivine. Powders were tumbled for at least 12 hours in a plastic  
 143 bottle with plastic-coated iron balls and subsequently dried on a hot plate at 60°C until all  
 144 the ethanol had evaporated. Powders were then placed in a 1 atm CO/CO<sub>2</sub> furnace, with  
 145 a  $P_{O_2}$  of  $\sim 10^{-10}$  atm, in which temperature was ramped up to 1000°C over 10 hours and  
 146 then held constant for 12 hours before cooling. The sample powders were cold pressed  
 147 at  $\sim 100$  MPa into a Ni can on top of a layer of NiO powder, which was present to buffer  
 148 oxygen fugacity. For sample PI-2056, the cold press consisted of roughly equal layers of  
 149 the aforementioned different compositional ratios. Samples were vacuum hot-pressed in a  
 150 gas-medium apparatus at a vacuum pressure on the interior of the jacket of  $\sim 10$  Pa and at a  
 151 confining pressure of 300 MPa (Meyers et al., 2017). Hot presses were carried out at 1250°C  
 152 under a small uniaxial load ( $\sim 1$  MPa) to aid compaction. For PT-1299HiFe and PI-2056, the  
 153 hot press was continued until compaction effectively ceased, which took between 0.5 and  
 154 1.3 hours. Samples 33 and PT-1166 were part of a series that were hot-pressed for between  
 155 3 and 60 hours to produce different grain sizes.

## 2.2 Sample Assembly

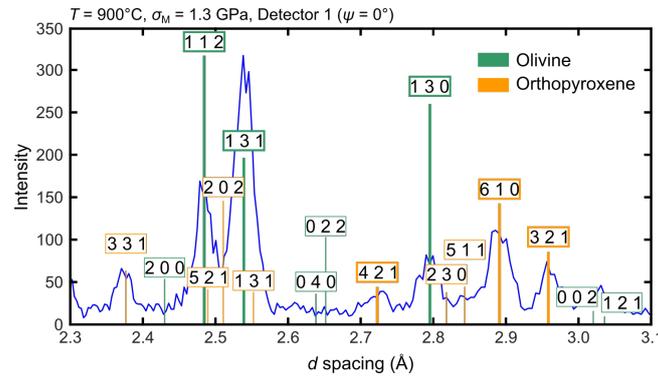
Hot presses were cored using a diamond coring drill to produce right-cylindrical samples of diameters 1.05–1.19 mm, which were subsequently ground to a height of  $0.55 \pm 0.05$  mm. For each experiment, a pair of samples were stacked between dense alumina pistons and then machinable alumina. Discs of nickel foil were placed between each component within the assembly to act as strain markers and also control the oxygen fugacity at the Ni/NiO buffer. The circumference of the samples was also wrapped in Ni foil. The assembly stack was placed within concentric sleeves of MgO and graphite, which provided an insulator and a resistive heater, respectively (See Figure 1). All components were placed inside a mullite sphere, which acted as the solid confining medium, itself residing within a pyrophyllite cube. The pyrophyllite cells were soft-fired for 3 hours at 900°C prior to sample assembly, which enabled them to flow during initial compression to form a gasket between the six anvil cells. Completed assemblies were sealed using a zirconia cement, ground to be square, and then left in an oven at  $\sim 120^\circ\text{C}$  for  $\geq 2$  hours to ensure an anhydrous state.

## 2.3 Experimental Procedure

We generated the desired stress state in the sample in the same manner as previous investigations implementing D-DIA experiments (Durham et al., 2009; Mei et al., 2010; Wang et al., 2003; Weidner et al., 2010). Pressure was applied to the pyrophyllite assembly cube through a hydraulically-loaded anvil in contact with each cube face. Pressurisation from the main loading ram applied equal load to all six anvils simultaneously, generating a hydrostatic compressive stress. Temperature was controlled using a calibrated relationship with heater power, as the inclusion of a thermocouple tends to degrade the mechanical stability of the sample assembly (Dixon & Durham, 2018). Once target pressures, here equivalent to mean stress  $((\sigma_v + 2\sigma_h)/3)$ , and temperatures were reached, differential stress was generated by independently advancing the vertical anvils, each controlled by driving a hydraulic syringe pump at a constant rate. Due to compressibility of the syringe fluid, the experimental boundary conditions are neither truly constant strain rate nor constant load. Moreover, changes in heater power (i.e., temperature) led to minor changes in the strain rate of the sample during an experiment. Nevertheless, nominally constant strain rates were reached after an initial loading phase. Meanwhile, the main ram was servo controlled to maintain a nominally constant mean stress (Durham et al., 2002).

188 During each experiment, a high-energy, white X-ray source was used to provide *in-situ*  
 189 stress and strain measurements via energy-dispersive X-ray diffraction and X-ray radiog-  
 190 raphy, respectively. Ten solid-state detectors were arranged downstream of the sample at  
 191 a variety of fixed azimuths ( $\psi$ ) and at an angle of  $6.5^\circ$  ( $2\theta$ ) to the beam direction (Figure  
 192 1). The Bragg angle of  $6.5^\circ$  was fixed through the use of conical slits and provided a col-  
 193 limated signal that ensured X-rays arriving at the detectors were diffracted from within  
 194 the sample, and not from the surrounding assembly components. Each analysed peak  
 195 within the diffraction spectrum corresponds to an  $\{hkl\}$  plane whose atomic spacing, or '*d*-  
 196 spacing', was calculated through Bragg's law and the fixed diffraction angle. This energy-  
 197 to-*d*-spacing conversion, as well as the Bragg angle, was calibrated at least once every seven  
 198 experiments (typically every two days) using an alumina-powder standard.

199 Diffraction spectra were collected, alternating between the top and the bottom sam-  
 200 ples, for durations of 20 to 60 seconds depending on the clarity of the peaks. Peaks used  
 201 in this analysis are highlighted in Figure 2 and correspond to the  $\{130\}$ ,  $\{131\}$ , and  $\{112\}$   
 202 planes in olivine and the  $\{421\}$ ,  $\{610\}$ , and  $\{321\}$  planes in orthopyroxene.



**Figure 2.** X-ray diffraction spectrum collected for 20 seconds in a harzburgite sample. The measured data are plotted as a blue line. Green and orange lines represent key diffracting planes in olivine and orthopyroxene, respectively. Peak labels outlined in bold are those used to measure stress in this experimental series.

203 Mean stress and differential stress were calculated from the measured *d*-spacing of  
 204  $\{hkl\}$  peaks following previous work (e.g., Durham et al., 2009; Hansen et al., 2019; Mei et  
 205 al., 2010; Wang et al., 2013; Weidner et al., 2010). Mean stress ( $P$ ) was measured from the  
 206  $\{130\}$  peak of olivine, calculated using the third-order Birch-Murnaghan equation of state  
 207 for olivine,

$$P = \left( \frac{3(K_0 + K'_T \Delta T)}{2} \right) \left[ \left( \frac{V_{0,T}}{V} \right)^{\frac{7}{3}} - \left( \frac{V_{0,T}}{V} \right)^{\frac{5}{3}} \right] \left\{ 1 + \frac{3}{4} (K'_P - 4) \left[ \left( \frac{V_{0,T}}{V} \right)^{\frac{2}{3}} - 1 \right] \right\}, \quad (1)$$

208 where  $\Delta T$  is the difference in temperature relative to the temperature at which the thermal  
 209 expansion measurements were made,  $K_0$  is the bulk modulus and  $K'_T$  and  $K'_P$  are the  
 210 temperature and pressure derivatives of  $K_0$ , respectively. Values of 129.4 GPa for  $K_0$  and  
 211 4.29 for  $K'_P$  were taken from Abramson et al. (1997). For  $K'_T$ , a value of  $-0.0224 \text{ GPa K}^{-1}$   
 212 was taken from Kumazawa and Anderson (1969). The unit-cell volume,  $V$ , was calculated  
 213 for each diffraction pattern during the experiment through

$$V = \left[ d_p (h^2 + k^2 + l^2)^{\frac{1}{2}} \right]^3, \quad (2)$$

214 where  $d_p = \frac{d_v + 2d_h}{3}$ ,  $d_v$  is the  $d$ -spacing measured at the vertical azimuth, and  $d_h$  is the  
 215  $d$ -spacing measured at the horizontal azimuth. To calculate  $d_h$  and  $d_v$ ,  $d$ -spacing was plotted  
 216 against  $\psi$  and then a sine wave was fit to the data.  $d_h$  and  $d_v$  were taken to be the maximum  
 217 and minimum of the sine wave, which sit  $90^\circ$  apart.  $V_{0,T}$  is the unit-cell volume at the  
 218 experiment temperature and ambient pressure, and is calculated as

$$V_{0,T} = V_0 \exp \left( 2.55 \times 10^{-5} \times (T - T_0) + \frac{1}{2} \times 1.15 \times 10^{-8} \times (T^2 - T_0^2) \right), \quad (3)$$

219 where  $V_0$  is the unit-cell volume at ambient temperature and pressure,  $T$  is the temperature,  
 220 and  $T_0$  is the temperature at which the measurements of thermal expansion were made.

221 Differential stress,  $\sigma$ , was calculated using the difference in  $d$ -spacing between the  
 222 horizontal and vertical directions,

$$\sigma = \left( \frac{1}{2G_R} \right)^{-1} \frac{d_h - d_v}{d_p}, \quad (4)$$

223 where  $G_R$  is the X-ray shear modulus calculated assuming the Reuss (isostress) condition  
224 (Singh et al., 1998) and using the elastic constants from Abramson et al. (1997) and Isaak  
225 (1992).

226 Prior to fitting peaks in the diffraction data, the intensity spectra were smoothed using  
227 a Savitzky-Golay filter and interpolated with a spline function. Due to the complicated  
228 diffraction patterns present in mixtures of olivine and orthopyroxene, peak locations were  
229 identified by fitting Gaussians to the highest intensity portions of each individual peak,  
230 rather than by fitting a mixing model of multiple Gaussians. When compared to the peak-  
231 fitting model (peakfit.m, O'Haver, 2018, page 340–358) used by Hansen et al. (2019) on  
232 a representative set of diffraction patterns for monophasic olivine, stress and mean stress  
233 measurements were similar.

234 Axial strain was obtained by using digital image cross-correlation to precisely mea-  
235 sure the positions of the nickel-foil strain markers in X-ray radiographs. By applying this  
236 technique, we were able to resolve subpixel shifts in foil location, resulting in precision in  
237 axial strain measurement of  $10^{-5}$  to  $10^{-4}$  (Hansen et al., 2019).

238 Deformation experiments were conducted at temperatures and mean stresses of  
239 800–1270°C and 1.6–9.7 GPa, respectively. Prior to deformation, each sample was annealed  
240 at temperatures of 800–1100°C for 5–20 minutes to relax any internal stress heterogeneity  
241 introduced during pressurisation (see Figure 1 of Wallis et al., 2020). To test which portion  
242 of the stress history the subgrain size records, all except one experiment were subjected to  
243 either steps in temperature, steps in strain rate, or both. At the end of each experiment,  
244 samples were quenched, the vertical anvils were withdrawn, and mean stress from the  
245 main loading ram was decreased slowly under approximately hydrostatic conditions.

## 246 **2.4 Microstructural Analysis**

247 To prepare the samples for microstructural analysis after deformation, the graphite  
248 furnace and all components within were extracted from the sample assembly and mounted  
249 on a glass slide using Logitech Epothin resin. Thick sections were formed by grinding the  
250 experimental column down on a Buehler Petrothin, at the University of Oxford, until it was  
251 a thickness of 1.2–1.8 mm. The ground surface was subsequently remounted on a fresh  
252 glass slide, the old glass slide removed, and the new free surface ground until a central  
253 section of the sample remained. At this stage, blobs of UV resin were placed around the

254 edges of the slides to help stabilise the samples during final polishing. Samples were then  
255 reground on the Petrothin to thicknesses in the range 0.2–0.6 mm with a flat central section  
256 exposed for polishing. All surfaces were polished with a Planocloth polishing cloth, 0.3  
257  $\mu\text{m}$  alumina powder, and water for 2–4 hours. Samples were subsequently polished with  
258 colloidal silica, as necessary, for 0.3–2.5 hours. Prior to EBSD data collection, samples were  
259 coated with 5–8 nm of carbon to minimise charging in the scanning electron microscope.

260 EBSD data were collected on a field-emission gun scanning-electron microscope (FEG-  
261 SEM) either at the Department of Earth Sciences, University of Oxford; the Marine Biolog-  
262 ical Laboratory (Woods Hole); or the Characterization Facility, University of Minnesota.  
263 At the University of Oxford, data were collected on an FEI Quanta 650 E-SEM equipped  
264 with Oxford Instruments AZtec acquisition software and a NordlysNano EBSD camera. At  
265 the Marine Biological Laboratory, data were collected using a Zeiss Supra 40VP FEG-SEM  
266 equipped with an Oxford Instruments Symmetry EBSD detector. At the University of Min-  
267 nesota, EBSD data were collected using a JEOL 6500 FEG-SEM with an Oxford Instruments  
268 Symmetry S2 EBSD detector. In all three systems, samples were tilted to  $70^\circ$  and mapped  
269 at either low-vacuum (50–70 Pa  $\text{H}_2\text{O}$  or  $\text{N}_2$ ) or high-vacuum conditions, at an accelerating  
270 voltage of 20–30 kV, with a step size of 0.075–0.5  $\mu\text{m}$ . Collection conditions for individual  
271 samples can be found in Supplementary Table S1.

272 Data were processed using a combination of Oxford Instruments' Channel5 or AZtec-  
273 Crystal software and the MTEX toolbox (version 5.7) for MATLAB<sup>®</sup> (Bachmann et al., 2010).  
274 First, spurious olivine pixels with systematic misindexing due to pseudosymmetry were  
275 corrected by applying a rotation of  $60^\circ$  around [100] (Bystricky et al., 2006) in Channel5  
276 or AZtecCrystal. EBSD data were then processed with the MTEX toolbox. Isolated pixels  
277 and "grains" comprised of  $< 3$  pixels were removed. Pixels with mean angular deviation  
278 (MAD) values  $> 1^\circ$  were also removed. Subsequently, all non-indexed pixels, which com-  
279 prise 2–38% (average 21%) of each map, were filled in MTEX using a nearest-neighbour  
280 method, whereby empty pixels are assigned the phase and orientation of the nearest in-  
281 dexed pixel. Interpolation was required for subgrain-size analysis as the centre of non-  
282 indexed regions are set to be boundaries in the current line-intercept MATLAB script, and  
283 could therefore result in inaccurate stress estimates. Figure S1 in the supplementary infor-  
284 mation presents phase maps of EBSD data prior to the infill of non-indexed regions.

285 To test the effect of indexing rate on the piezometric stress measurements, samples  
 286 were further polished with colloidal silica and new maps were collected for the olivine-  
 287 orthopyroxenite sample in San502 and for both the olivine stress sensor and harzburgite  
 288 samples in San508, two key samples where the original maps contained high proportions  
 289 of non-indexed pixels (25–40%). Despite the indexing rate being increased by up to 27%,  
 290 the change in estimated stress was generally negligible (between 0–15%), and has no effect  
 291 on the overall conclusions of this work.

## 292 2.5 Subgrain-size Piezometry

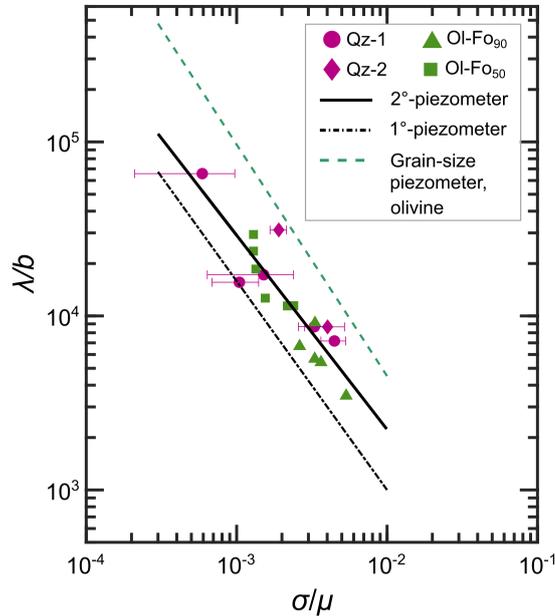
293 We conducted subgrain size piezometry following the procedures laid out by Goddard  
 294 et al. (2020). Goddard et al. (2020) presented a single subgrain-size piezometer calibrated  
 295 from EBSD maps of deformed quartz aggregates and olivine aggregates, in which subgrain  
 296 size was normalised by the Burgers vector, and differential stress was normalised by the  
 297 shear modulus. Subgrain size was defined as the average spacing between boundaries with  
 298 misorientation angles  $\geq 1^\circ$ . As such, it is important to ensure that EBSD maps have angular  
 299 precision  $< 1^\circ$  (ideally  $< 0.5^\circ$ ) to avoid the creation of spurious  $1^\circ$  boundaries. Here, we  
 300 find that some EBSD maps contain isolated pixels misoriented from their neighbours by  
 301  $\geq 1^\circ$  (e.g., Supplementary Figure S2), which we attribute to two sources. First, angular  
 302 uncertainties arising from application of the Hough transform for automated indexing,  
 303 commonly on the order of  $0.5^\circ$  (Maitland & Sitzman, 2007). Second, high concentrations of  
 304 isolated geometrically necessary dislocations, not contained within subgrain boundaries,  
 305 given the relatively high differential stresses ( $> 1$  GPa) experienced by samples in this  
 306 study.

307 To avoid the inclusion of spurious  $1^\circ$  boundaries, we instead use a modified subgrain-  
 308 size piezometer (Figure 3), recalculated for a critical misorientation angle of  $2^\circ$  from the  
 309 EBSD maps used in the original calibration by Goddard et al. (2020) :

$$\frac{\lambda}{b} = 10^{1.1 \pm 0.3} \left( \frac{\sigma}{\mu} \right)^{-1.1 \pm 0.1} \quad (5)$$

310 where  $\lambda$  is the mean line-intercept length,  $b$ , is the Burgers vector,  $\sigma$ , is the equivalent stress,  
 311 and  $\mu$  is the shear modulus. This new calibration also includes an additional cleaning step  
 312 of removing pixels with mean angular deviation of  $> 1^\circ$ . Note that the stresses applied

313 to quartz in Figure 3 have also been corrected for friction in accordance with Holyoke and  
 314 Kronenberg (2010).



**Figure 3.** Recalibration of the subgrain-size piezometer based on boundaries with misorientation angles of  $\geq 2^\circ$ . Mean line-intercept length,  $\lambda$ , normalised by the Burgers vector,  $b$ , is plotted as a function of the equivalent stress,  $\sigma$ , normalised by the shear modulus,  $\mu$ . The original  $1^\circ$ -piezometer by Goddard et al. (2020) has been added for comparison (dashed black line) as well as the grain-size piezometer for olivine (van der Wal et al., 1993). Currently, no equivalent grain-size piezometer exists for orthopyroxene. For further explanation of the different data subsets, see Goddard et al. (2020).

315 For consistency with Goddard et al. (2020), subgrain size was measured using the  
 316 line-intercept method. To ensure that we used an adequate number of intercepts for robust  
 317 measurement of the subgrain size, we systematically increased the number of intercepts  
 318 until the line-intercept length became essentially invariant (varying  $< 2.5\%$  from the previ-  
 319 ous measurement). The mean-line intercept length was calculated as the arithmetic mean  
 320 of intercepts measured both parallel and perpendicular to  $\sigma_1$ . The mean grain size of each  
 321 phase,  $\lambda_{gs}$ , was also measured using the line-intercept method with the minimum mis-  
 322 orientation angle of a grain boundary defined as  $15^\circ$ , and with no geometric correction  
 323 applied.

324 For the initial calibration of the subgrain-size piezometer, temperature and pressure  
 325 had a negligible impact on the shear modulus and Burgers vector values used to normalise

326 the piezometer, at least over the range of temperature-pressure conditions used in the cal-  
 327 ibration experiments (Goddard et al., 2020). However, the olivine samples used for the  
 328 original piezometer calibration were deformed at a pressure of 0.3 GPa, which is signifi-  
 329 cantly lower than the mean stresses used here (1.6–9.7 GPa). These elevated mean stresses  
 330 have a particularly significant impact on the shear modulus of olivine, which increases  
 331 by 4–16% relative to its value at room pressure. Therefore, we chose to correct the shear  
 332 moduli and Burgers vectors of olivine and orthopyroxene using the pressure derivatives  
 333 given in Table 1. The corrected Burgers vectors and shear moduli give stresses that, on av-  
 334 erage, are 7% greater than those based on using the uncorrected values, with a maximum  
 335 difference of 12%.

336 For each piezometric stress estimate, an error was calculated using a Monte-Carlo-  
 337 based approach. We assumed that the probability distribution of the subgrain size, pre-  
 338 exponential constant, and exponent all follow lognormal distributions with standard de-  
 339 viations (in log units) of 0.13, 0.3, and 0.1, respectively, and with means given by the mea-  
 340 sured or best-fit values. These standard deviations were determined in the initial calibra-  
 341 tion of the subgrain piezometer, as described in Appendix A. We then randomly drew val-  
 342 ues from these distributions and used them to predict the differential stress. This process  
 343 was repeated 10,000 times, and the standard deviation in the distribution of differential  
 344 stresses was taken to be the error in the stress measurement. A full description of this  
 345 method can be found in Appendix A.

346

**Table 1.** Pressure dependence of shear moduli and Burgers vectors for olivine and orthopyroxene

Mineral	$G_o$ (GPa)	$\partial G/\partial P$	$\partial^2 G/\partial^2 P$ (GPa <sup>-1</sup> )	$b$ ( $\mu\text{m}$ )	$\partial b/\partial P$ ( $\mu\text{mGPa}^{-1}$ )	$\partial^2 b/\partial^2 P$ ( $\mu\text{mGPa}^{-2}$ )
Ol	77.8 <sup>a</sup>	1.71 <sup>b</sup>	-0.054 <sup>b</sup>	<a> 4.75 <sup>c</sup> $\times 10^{-4}$	-8.377 <sup>b</sup> $\times 10^{-3}$	2.088 <sup>b</sup> $\times 10^{-4}$
Opx	78.1 <sup>d</sup>	1.45 <sup>d</sup>	0	<c> 5.20 <sup>e</sup> $\times 10^{-4}$	-1.548 <sup>f</sup> $\times 10^{-6}$	8.2180 <sup>f</sup> $\times 10^{-8}$

<sup>a</sup>Mao et al. (2015)<sup>b</sup>Abramson et al. (1997)<sup>c</sup>Deer et al. (1988, page 4)<sup>d</sup>Chai et al. (1997)<sup>e</sup>Deer et al. (1988, page 158)<sup>f</sup>Angel and Hugh-Jones (1994)

### 3 Results

#### 3.1 Mechanical Data

We performed seven experiments in total: one with an olivine stress sensor stacked in series with an olivine-orthopyroxenite sample, five with an olivine stress sensor stacked in series with a harzburgite sample, and one with two samples of harzburgite stacked in series. Table 2 lists the temperatures, strain rates, mean stresses, and final strains for each sample. The final stress indicated by each diffraction peak can be found in Table 3, and the peak and average stresses can be found in Supplementary Tables S2 and S3, respectively.

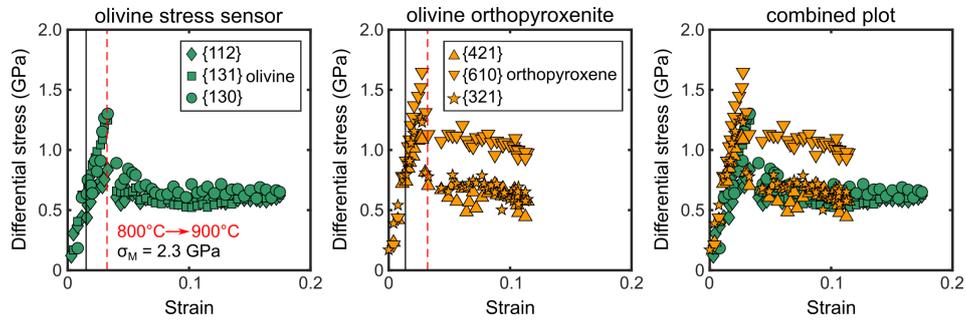
##### 3.1.1 Olivine Stress Sensor Stacked Against Olivine-Orthopyroxenite

Mechanical data for San502, which consisted of an olivine-orthopyroxenite sample stacked against an olivine stress sensor, are displayed in Figure 4. Due to the low proportion of olivine (15%) within the olivine-orthopyroxenite sample, stresses were only calculated for orthopyroxene. Initially, differential stress increased with strain. The apparent Young's modulus of  $\sim 40$  GPa from this period is too low to represent purely elastic deformation, which is associated with an elastic modulus of  $\sim 200$  GPa for olivine. As we measure stresses from the sample directly, we can rule out machine-compliance effects. Therefore, we infer that our experiments yielded early during loading and progressively hardened.

Samples were first deformed at a temperature of  $800^{\circ}\text{C}$  and at strain rates of  $2.9 \times 10^{-5} \text{ s}^{-1}$  and  $2.5 \times 10^{-5} \text{ s}^{-1}$ , for the olivine stress sensor and olivine-orthopyroxenite sample, respectively until a strain of 0.016 was reached in the olivine and a strain of 0.014 in the olivine orthopyroxenite. At this point, the displacement rate was decreased resulting in strain rates of  $1.9 \times 10^{-5} \text{ s}^{-1}$  in the olivine and  $1.4 \times 10^{-5} \text{ s}^{-1}$  in the olivine orthopyroxenite. After a further  $\sim 0.02$  strain, temperature was increased to  $900^{\circ}\text{C}$  so that deformation could continue at a lower differential stress. After the change in temperature, the olivine stress sensor and olivine-orthopyroxenite sample deformed at strain rates of  $4.4 \times 10^{-5} \text{ s}^{-1}$  and  $2.3 \times 10^{-5} \text{ s}^{-1}$ , respectively.

During the final deformation step, the average flow stresses in the olivine stress sensor (590–690 MPa) were similar to those measured from the  $\{421\}$  and  $\{321\}$  peaks in orthopyroxene within the olivine-orthopyroxenite sample (620 MPa and 670 MPa, respectively).

377 However, stresses measured from the {610} peak in orthopyroxene were considerably  
 378 greater, averaging 1050 MPa.



**Figure 4.** Stress as a function of strain for experiment San502, which consists of an olivine stress sensor paired against an olivine orthopyroxenite. Stresses from olivine and orthopyroxene are in green and orange, respectively. The red dashed vertical line represents a change in temperature, the black vertical line represents a change in displacement rate, and  $\sigma_M$  refers to the mean stress during the final conditions of the experiment (see Table 2 for values).

### 3.1.2 Olivine Stress Sensor Stacked Against Harzburgite

379

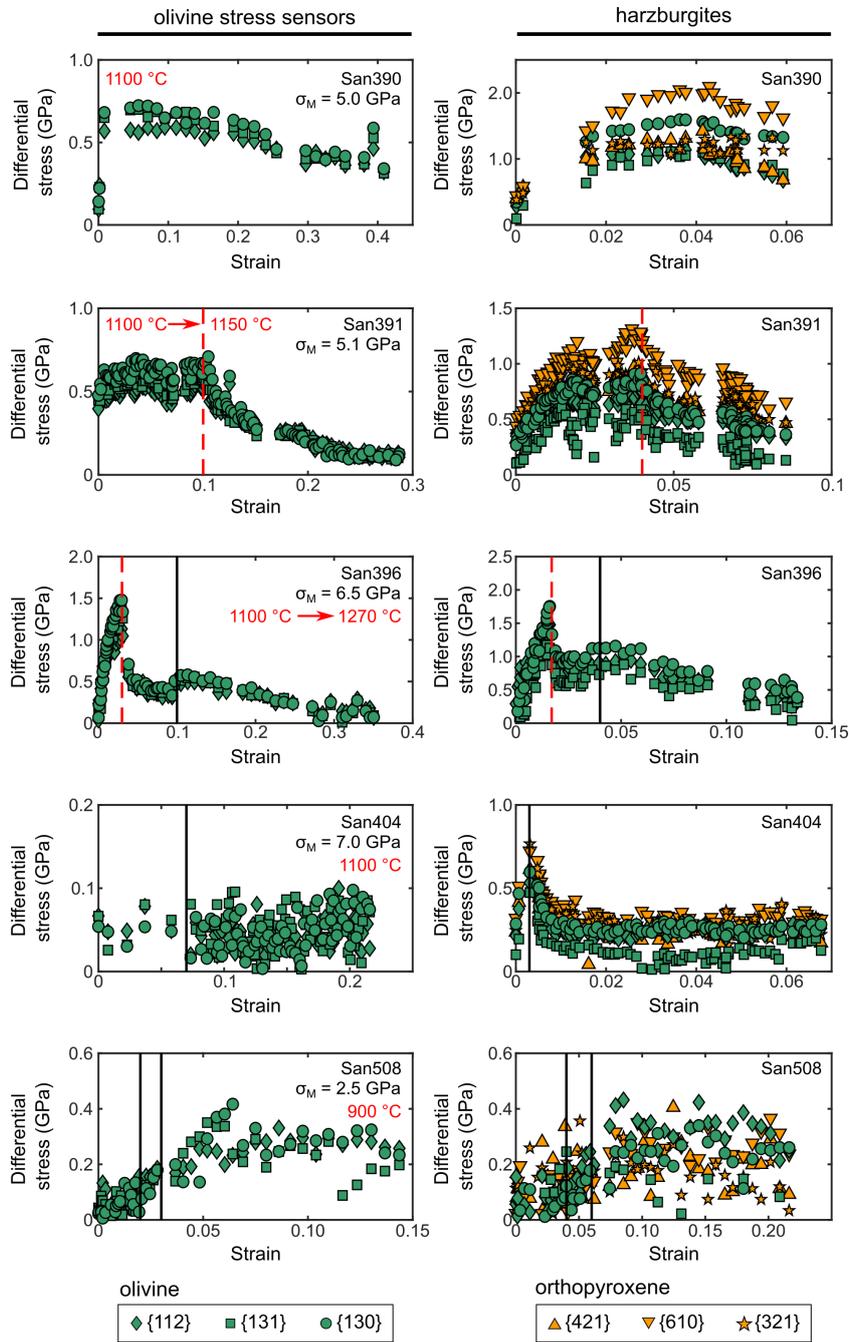
380 Mechanical data for the five experiments consisting of a harzburgite sample and olivine  
 381 stress sensor are presented as stress-strain curves in Figure 5. Four of these experiments  
 382 were deformed at temperatures in the range 1100–1270°C and average mean stresses in  
 383 the range 4.7–9.4 GPa. Experiment San508 was deformed at 900°C and at average mean  
 384 stresses of 1.6–2.5 GPa.

385 In all samples except the stress sensors in San391 and San404, stress initially increased  
 386 with strain. Low apparent moduli from these portions of the stress-strain curves suggest  
 387 that yielding was instantaneous within the temporal resolution of our experimental mea-  
 388 surements. Steeper gradients at < 1% strain in the mixtures in San390 and San404 suggest  
 389 deformation may have initially been elastic, but data from these segments of loading are  
 390 sparse.

391 In most experiments, temperature and/or the displacement rate of the vertical rams  
 392 were modified during deformation to change the differential stress. During experiments  
 393 San391 and San396, the temperature was raised to decrease the differential stress. In ex-  
 394 periment San404, the displacement rate of the vertical rams was reduced, also to decrease  
 395 the differential stress. In experiments San396 and San508, the ram displacement rate was

396

instead increased. These changes in displacement rate and temperature are indicated in Table 2, and the stress-strain curves are presented Figure 5.



**Figure 5.** Stress as a function of strain for olivine stress sensors (left) paired against harzburgites (right). Stresses from olivine and orthopyroxene are in green and orange, respectively. The red dashed vertical lines represent changes in temperature, the black vertical lines represent changes in displacement rate, and  $\sigma_M$  refers to the mean stress during the final conditions of the experiment (see Table 2 for values).

397

398 Stresses in samples in San404 and San508 evolved to constant values whereas stresses  
399 in the other harzburgite-bearing experiments decreased progressively over time. This re-  
400 duction in stress may be due to an increase in temperature as a result of changes to the  
401 furnace during deformation (e.g., changes in furnace shape or movement of the sample  
402 relative to the hot zone). As stated above, temperature is calculated based on a calibrated  
403 relationship between furnace power and temperature (Dixon & Durham, 2018). Therefore,  
404 deviations in the furnace shape or the position of a sample relative to the hot zone from that  
405 of the calibration assembly would lead to discrepancies between the calculated and actual  
406 temperature. As temperature is an input into the equation for mean stress (see Eqs 1 & 3),  
407 comparing the differential-stress and mean-stress curves can give an indication of whether  
408 temperature increased. An artificially low apparent temperature (i.e., when the tempera-  
409 ture based on the furnace calibration is lower than the actual temperature experienced by  
410 the samples) leads to an artificially low apparent mean stress. Thus, a decrease in both  
411 mean stress and differential stress during an experiment may be indicative of an increase  
412 in temperature. The simultaneous drop in the calculated mean stresses (see Figure S3) and  
413 differential stresses suggests that temperature did increase throughout experiments San404  
414 and San508. However, as mean stress and temperature only play a minor role in the calcu-  
415 lation of the X-ray shear modulus and thus the calculation of the differential stress—and  
416 as piezometers are thought to be temperature independent (e.g., Stipp & Tullis, 2003)—the  
417 precise cause of the drop in stress is not directly relevant to this study.

418 In the olivine stress sensors, average stresses from each of the three diffraction peaks  
419 are typically consistent to within 20 MPa, with the exception of San390, which is consistent  
420 to within 70 MPa. For olivine within harzburgites, stresses from the three peaks differ by up  
421 to 450 MPa, with the {130} peak typically giving the greatest stress. For orthopyroxene in  
422 harzburgite, the difference in stresses calculated from the {421} and {321} peaks is within  
423 40 MPa, with the exception of San390, where calculated stresses differ by up to 110 MPa.  
424 If stresses from the {610} peak are included, the difference in the stress increases up to 690  
425 MPa.

426 For four of the experiments involving harzburgite, strain in the olivine stress sensor  
427 (0.22–0.41) was significantly greater than that in the harzburgite (0.06–0.13), indicating that  
428 harzburgite was stronger than olivine at the high pressures (4.7–9.4 GPa) and tempera-  
429 tures (1100–1270°C) of these experiments. Stresses measured in the harzburgite sample by  
430 X-ray diffraction were also, in all cases, greater in orthopyroxene than in olivine. In exper-

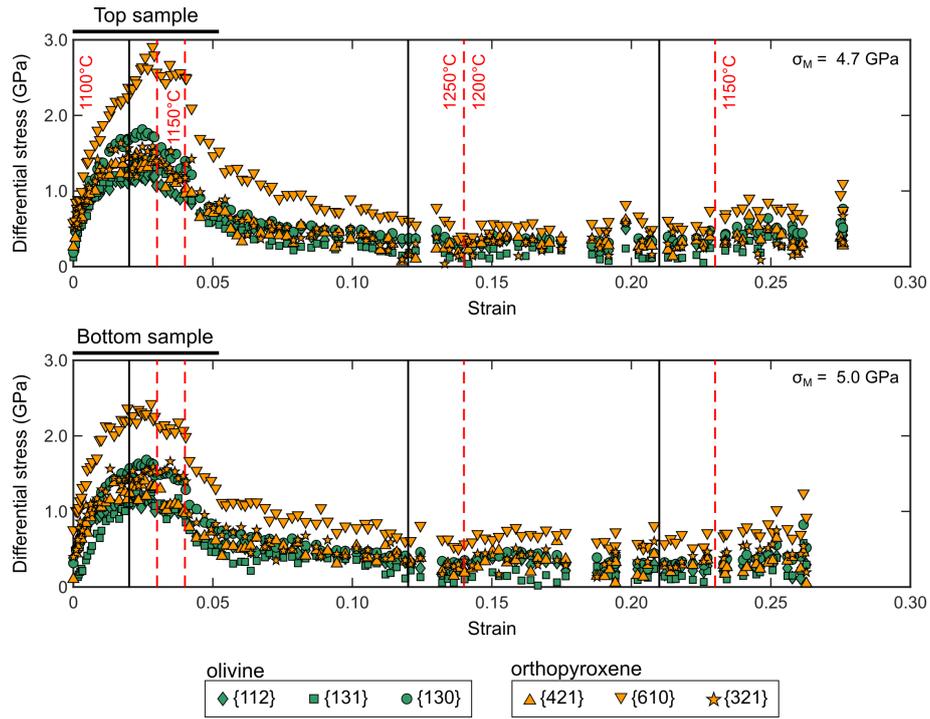
431 iment San396, the diffraction patterns in orthopyroxene in the harzburgite were too noisy  
432 to measure stress, and as such, it was not possible in this instance to compare how stress  
433 was partitioned between olivine and orthopyroxene.

434 In experiment San508, which was conducted at relatively low mean stresses of 1.6–2.5  
435 GPa and a temperature of 900°C, strain was greater in the mixture (0.22) than in the olivine  
436 sample (0.16). Within the mixture, the average stresses calculated from the peaks from  
437 orthopyroxene (170–280 MPa) lay within the range of stresses calculated from the peaks  
438 from olivine (-50–330 MPa).

### 439 *3.1.3 Harzburgite Stacked Against Harzburgite*

440 To attain greater strains within harzburgite, two samples of the same composition  
441 were deformed together in a single experiment (San409), reaching a total strain of 0.28  
442 (Figure 6). The samples initially deformed elastically, following the elastic modulus of  
443 harzburgite (Christensen, 1966), to strains of 0.005–0.01, after which the samples yielded  
444 and proceeded to strain harden. To take the samples through a variety of different stresses,  
445 the temperature and displacement rate of the experiment were modified at four and three  
446 points, respectively (see Table 2 and Figure 6). Temperature was first increased from 1100°C  
447 to 1150°C, and then subsequently increased further to 1250°C. After ~0.14 strain, the tem-  
448 perature was reduced to 1200°C and then further reduced to 1150°C at 0.23 strain to pre-  
449 vent the differential stress from relaxing below the noise level. Changes in the experimental  
450 conditions led to strain rates of  $1.7 \times 10^{-5} \text{ s}^{-1}$  to  $4.5 \times 10^{-5} \text{ s}^{-1}$ .

451 Average stresses calculated from the {421} and {321} peaks in orthopyroxene (290–410  
452 MPa) are consistent with the stresses from the olivine peaks (180–430 MPa), whereas stresses  
453 calculated from the {610} peaks were considerably greater (660 MPa and 690 MPa for the  
454 top and bottom samples, respectively).



**Figure 6.** Stress versus strain curves for harzburgites in experiment San409. The central Ni foil was not usable for strain measurements in this experiment. As a result, stresses for each sample are plotted instead against the total strain of the combined sample stack. The red dashed vertical lines represent changes in temperature, the black vertical lines represent changes in displacement rate, and  $\sigma_M$  refers to the mean stress during the final conditions of the experiment (see Table 2 for values).

Table 2: Experimental Conditions

Experiment	Position	Sample	Starting material	Temperature (°C)	Strain rate <sup>a</sup> ( $\times 10^{-5} \text{s}^{-1}$ )	Average mean stress <sup>b</sup> (GPa)	Final mean stress (GPa)	Total strain
San390	Top	Ol	33	1100	22.3	$5.0 \pm 0.5$	4.1	0.41
	Bottom	Hz(Px30)	PT-1299HiFe	1100	2.5	$4.9 \pm 0.5$	4.1	0.06
San391	Top	Hz(Px30)	Pt-1299HiFe	1100	0.7	$5.1 \pm 0.2$	5.5	0.04
				1150	0.2	$5.0 \pm 0.3$	4.5	0.08
	Bottom	Ol	33	1100	1.8	$5.2 \pm 0.2$	5.6	0.10
				1150	2.7	$5.1 \pm 0.3$	4.6	0.28
San396	Top	Hz(Px30)	PL-1299HiFe	1100	1.3	$9.1 \pm 0.1$	9.1	0.02
				1270	2.3	$8.2 \pm 0.3$	7.8	0.04
				1270	9.0	$6.3 \pm 0.9$	5.2	0.13
	Bottom	Ol	PT-1166	1100	2.3	$9.4 \pm 0.2$	9.7	0.03
				1270	7.9	$8.3 \pm 0.3$	7.9	0.10
				1270	22.0	$6.5 \pm 0.9$	5.2	0.35
San404	Top	Ol	LT-DrySC	1100	8.6	$7.2 \pm 0.1$	7.3	0.07
				1100	1.3	$7.0 \pm 0.1$	6.9	0.22
	Bottom	Hz(Px30)	PL-1299HiFe	1100	0.7	$7.0 \pm 0.1$	7.1	<0.01
San409 <sup>c</sup>	Top	Hz(Px30)	PL-1299HiFe	1100	0.6	$7.0 \pm 0.0$	6.9	0.07
				1100	1.7	$7.7 \pm 0.2$	8.0	0.02
				1100	1.8	$7.9 \pm 0.0$	8.0	0.03
				1150	1.8	$7.7 \pm 0.2$	7.6	0.04
				1250	3.9	$6.9 \pm 0.4$	6.3	0.12
				1250	4.5	$6.0 \pm 0.1$	6.0	0.14
				1200	4.5	$5.5 \pm 0.3$	4.9	0.21
				1200	4.2	$4.9 \pm 0.0$	4.9	0.23
				1150	4.2	$4.7 \pm 0.2$	5.0	0.28

Table 2 – continued from previous page

Experiment	Position	Sample	Starting material	Temperature (°C)	Strain rate <sup>a</sup> ( $\times 10^{-5} \text{s}^{-1}$ )	Average Mean stress <sup>b</sup> (GPa)	Final Mean stress (GPa)	Total strain
San409 <sup>c</sup>	Bottom	Hz(Px30)	PL-1299-HiFe	1100	1.7	$7.8 \pm 0.2$	8.1	0.02
				1100	1.8	$8.1 \pm 0.1$	8.2	0.03
				1150	1.8	$7.9 \pm 0.1$	7.8	0.04
				1250	3.9	$7.0 \pm 0.4$	6.3	0.12
				1250	4.5	$6.1 \pm 0.1$	6.0	0.14
				1200	4.5	$5.6 \pm 0.3$	5.2	0.21
				1200	4.2	$5.1 \pm 0.1$	5.1	0.23
				1150	4.2	$5.0 \pm 0.3$	5.7	0.28
San502	Top	OrthoPy(Px85)	PI-2056	800	2.5	N/A	N/A	0.01
				800	1.4	N/A	N/A	0.03
				900	2.4	N/A	N/A	0.11
	Bottom	Ol	PI-2056-ol	800	2.9	$2.5 \pm 0.5$	3.0	0.02
				800	1.9	$3.2 \pm 0.1$	3.4	0.03
				900	4.4	$2.3 \pm 0.2$	2.1	0.17
San508	Top	Hz(Px50)	PI-2056	900	3.3	$1.6 \pm 0.3$	1.9	0.04
				900	3.6	$2.1 \pm 0.1$	2.2	0.06
				900	5.6	$2.5 \pm 0.1$	2.3	0.22
	Bottom	Ol	PI-2056-ol	900	1.2	$2.0 \pm 0.1$	2.2	0.02
				900	2.3	$2.3 \pm 0.1$	2.2	0.03
				900	4.8	$2.5 \pm 0.1$	2.3	0.16

<sup>a</sup>Strain rate is taken as the gradient between strain and time fit through linear regression.

<sup>b</sup>Mean stress calculated using the {130} peak in the olivine in each sample.

<sup>c</sup>Due to a tilt in the middle foil, all reported strains and strain rates for this experiment are the average for the entire assembly.

**Table 3.** Final stresses measured by X-ray diffraction, rounded to the nearest 10 MPa.

Experiment	Sample <sup>a</sup>	Final stress: X-ray diffraction (MPa)					
		Olivine			Orthopyroxene		
		{112}	{131}	{130}	{421}	{610}	{321}
San390	OSS	320	310	340	-	-	-
	Mix	680	780	1330	680	1620	1130
San391	OSS	110	120	90	-	-	-
	Mix	350	140	390	380	590	500
San396	OSS	70	90	70	-	-	-
	Mix	330	310	390	-	-	-
San404	OSS	30	70	70	-	-	-
	Mix	260	130	280	170	310	220
San409	Mix:Top	420	320	510	310	740	320
	Mix:Bot	300	300	520	50	920	190
San502	OSS	620	630	650	-	-	-
	Mix	-	-	-	440	930	580
San508	OSS	230	220	180	-	-	-
	Mix	250	-60	260	90	240	30

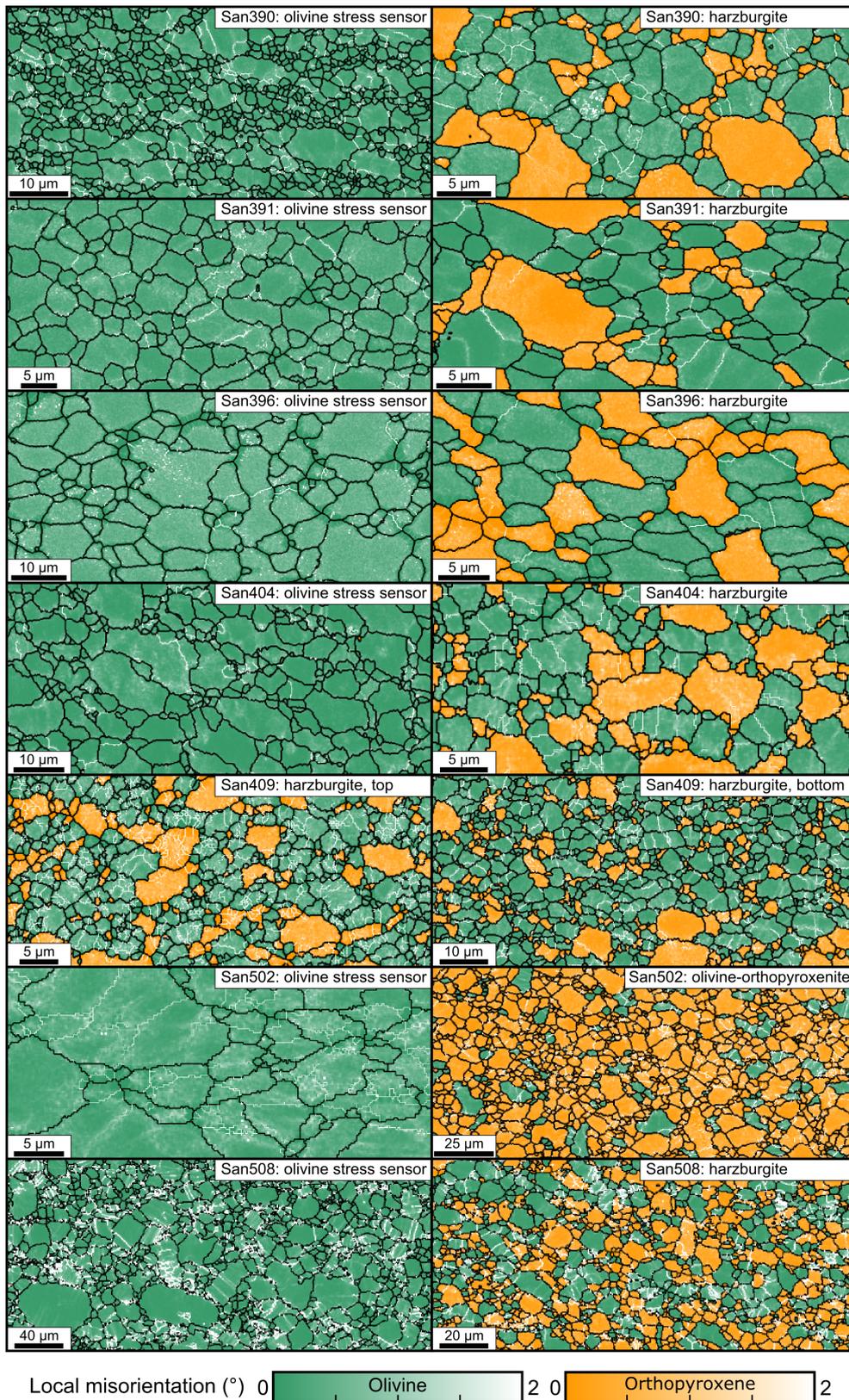
<sup>a</sup>OSS: Olivine stress sensor, Mix: Harzburgite or olivine orthopyroxenite

### 3.2 Microstructural Analysis

A map of kernel-average misorientation (KAM) for each sample is presented in Figure 7. Subgrain boundaries are common in olivine, both in the stress sensors and mixtures, and particularly in samples deformed at temperatures  $\geq 1100^\circ\text{C}$ . In orthopyroxene, subgrain boundaries are sparse (if present at all) in experiments conducted at high temperatures and mean stresses, with the exception of experiment San409, in which the stacked harzburgites reached a strain of 0.28 (Figure 6). However, subgrain boundaries are pervasive within orthopyroxene in the experiments conducted at lower temperatures and mean stresses (San502 and San508).

Average intercept lengths for boundaries with a misorientation angle of  $\geq 15^\circ$  (i.e., grain boundaries) in the olivine stress sensors range from 1.6–6.6  $\mu\text{m}$ . In the mixtures, the mean lengths are 1.9–3.7  $\mu\text{m}$  and 1.7–3.0  $\mu\text{m}$  for olivine and orthopyroxene, respectively. Where possible, comparisons to the initial starting microstructures show that grains within the olivine stress sensors are  $\sim 30\%$  smaller after deformation, whereas within the mixtures

469 the grain size remains roughly constant. Mean line-intercept lengths between boundaries  
470 with a misorientation angle of  $\geq 2^\circ$  (i.e., subgrain boundaries and grain boundaries) are  
471 1.3–4.8  $\mu\text{m}$  in the olivine stress sensors, corresponding to stresses of 190–630 MPa as esti-  
472 mated by the subgrain-size piezometer (Eq. 5). In the mixtures, mean line-intercept lengths  
473 for olivine (1.2–2.9  $\mu\text{m}$ ) and orthopyroxene (1.5–2.7  $\mu\text{m}$ ) correspond to estimated stresses  
474 of 300–680 MPa and 340–590 MPa, respectively. The average boundary spacing and the  
475 associated stresses are presented in Table 4.



**Figure 7.** Subsets of local misorientation maps from EBSD data. Phase maps of the full, unfilled datasets are presented in Figure S1 in the supplementary information. Grain and subgrain boundaries are displayed in black and white, respectively and are defined as boundaries with misorientation angles of  $\geq 15^\circ$  and  $\geq 2^\circ$ . The compressional axis is vertical.

**Table 4.** Grain-size measurement ( $\lambda_{gs}$ ) and subgrain boundary spacing, that is subgrain size, measurement ( $\lambda$ ) with associated piezometric stresses and errors. Both ( $\lambda_{gs}$ ) and ( $\lambda$ ) were measured using the mean-line intercept method.

Experiment	Position	Index rate (%)	Average MAD (°)	Grain-size measurement			Subgrain-size measurement			
				Phase	No. Intercepts	$\lambda_{gs}$ ( $\mu\text{m}$ )	No.intercepts	$\lambda$ ( $\mu\text{m}$ )	Stress (MPa)	Error (MPa)
San390	Top	72	0.40±0.33	Ol	60	1.6	60	1.3	630	+820/-360
	Bottom	68	0.51±0.38	Ol	60	1.9	60	1.5	550	+720/-310
San391	Top	87	0.37±0.26	Opx	50	1.7	60	1.5	590	+760/-340
	Bottom	86	0.45±0.30	Ol	70	2.2	70	1.9	450	+610/-260
San396	Top	62	0.41±0.36	Opx	90	2.1	90	2.0	460	+620/-260
	Bottom	69	0.51±0.38	Ol	70	2.6	70	2.3	380	+510/-220
San404	Top	91	0.33±0.30	Ol	40	2.3	70	2.2	400	+530/-230
	Bottom	83	0.44±0.36	Opx	80	2.8	70	2.5	370	+520/-210
San409	Top	65	0.63±0.53	Ol	70	3.7	70	3.2	290	+400/-170
	Bottom	82	0.50±0.38	Opx	40	3.0	60	2.8	330	+460/-190
San502	Top	79	0.45±0.35	Ol	80	2.0	70	1.6	550	+720/-300
	Bottom	80	0.51±0.36	Opx	70	2.0	70	1.8	520	+700/-300
San508	Top	87	0.39±0.32	Ol	80	1.9	80	1.2	680	+890/-380
	Bottom	98	0.29±0.21	Opx	60	2.3	80	1.5	600	+780/-340
San509	Top	79	0.45±0.35	Ol	60	1.9	60	1.5	570	+750/-320
	Bottom	80	0.51±0.36	Opx	60	1.8	80	1.6	580	+750/-330
San510	Top	87	0.39±0.32	Ol	80	3.0	80	2.3	370	+490/-210
	Bottom	98	0.29±0.21	Opx	60	3.0	70	2.7	340	+460/-200
San511	Top	87	0.39±0.32	Ol	80	4.0	70	2.1	390	+520/-230
	Bottom	98	0.29±0.21	Opx	60	2.7	80	2.4	480 <sup>a</sup>	+760/-300
San512	Top	87	0.39±0.32	Ol	80	3.7	80	2.9	300	+410/-180
	Bottom	98	0.29±0.21	Opx	60	2.7	60	2.4	380	+510/-220
San513	Top	87	0.39±0.32	Ol	80	6.6	80	4.8	190	+270/-110
	Bottom	98	0.29±0.21	Opx	60	6.6	80	4.8	190	+270/-110

<sup>a</sup>Calculated using the [001] Burger's vector for olivine, see Appendix B

## 4 Discussion

### 4.1 Comparing *In-situ* and *Ex-situ* Stress Measurements

A more complete understanding of the tectonic processes that occur on Earth requires constraints on the strength of the lower crust and mantle, both of which are generally made up of more than one mineral. The recent calibration of an EBSD-based subgrain-size piezometer (Goddard et al., 2020) offers new opportunities for estimating paleostress magnitudes within exhumed polymineralic rocks, while avoiding the potential effects of grain-boundary pinning by secondary phases. However, to date, no study has tested whether subgrain size accurately reflects stresses supported by individual phases within polymineralic rocks, nor what these piezometric stresses tell us about the overall stress experienced by an aggregate. In this study, we present the first direct comparison of stresses measured *in-situ* within individual phases to those recorded by the subgrain-size piezometer.

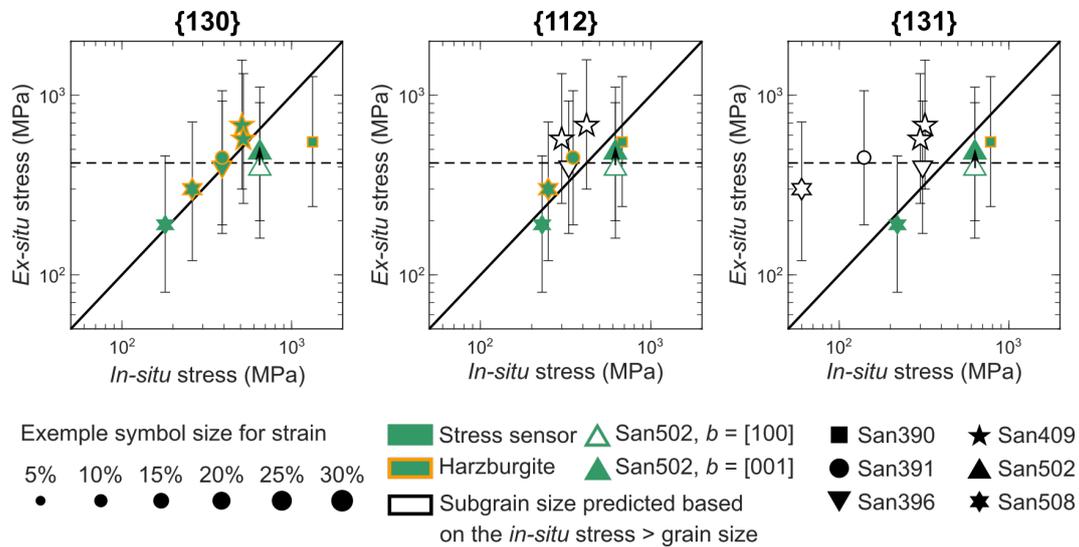
For consistency with the piezometer of Goddard et al. (2020), we begin by considering the final *in-situ* stress experienced by each sample. While we have a single stress to consider from subgrain-size piezometry, the different diffraction peaks in both olivine and orthopyroxene provide a range of stresses. This variation in stress is a result of the plastic anisotropy of olivine and orthopyroxene, as grains within plastically anisotropic materials subjected to a non-hydrostatic stress field will undergo different amounts of plastic strain depending on their orientation (e.g., Ashby, 1970). This heterogeneity in plastic strain is accompanied by heterogeneity in elastic strain. As such, grains in different orientations experience different stresses (Karato, 2021; Li et al., 2006; Wang et al., 2003; Weidner et al., 2010). Here, we find that the {130} peak from olivine resulted in stresses on average 1.5 times greater than those from the other olivine peaks, consistent with previous observations (e.g., Weidner et al., 2010). For orthopyroxene, the {610} peak resulted in the greatest stress, on average 1.6 times greater than the {421} and {321} peaks. Previous studies have taken the average of the stresses measured from individual diffraction peaks to represent the macroscopic differential stress experienced by the sample (e.g., Li et al., 2006). However, it is unclear whether or not the stresses measured from different diffraction peaks should be evenly weighted (c.f., Burnley & Kaboli, 2019). Instead, the comparison of piezometric stresses from a single phase to those estimated from different diffraction peaks may shed light on which peaks best represent the macroscopic stress in our experiments.

507 Before comparing these *in-situ* stress measurements to those gathered *ex-situ*, it is first  
508 important to ensure that sufficient substructure developed for subgrain-size piezometry,  
509 a criterion not met by all the samples in this study (e.g., the olivine stress sensors of  
510 San396 and San404, Figure 7). One possible explanation for the lack of substructure is  
511 that the grains in these samples are too small to contain subgrains of the size predicted by  
512 the subgrain-size piezometer. For example, extrapolation of the grain- and subgrain-size  
513 piezometers of Twiss (1977) to greater stresses results in a theoretical subgrain size larger  
514 than the steady-state grain size. To test whether the grain size in our samples was large  
515 enough for subgrains to form, we calculated the predicted mean-line intercept length be-  
516 tween subgrain boundaries, herein referred to as subgrain size, using the final stress mea-  
517 sured from each X-ray peak and the pressure-corrected Burgers vectors and shear moduli.  
518 For each mineral, the predicted subgrain size was then compared to the measured grain  
519 size to assess whether subgrains would be expected to form at the final stresses experi-  
520 enced by that sample. The predicted subgrain size and its comparison to grain size for  
521 each phase in each sample can be found in Table S4 of the supplementary information.

522 For the olivine stress sensors in San390, San391, San396, and San404, all predicted  
523 subgrain sizes exceed the measured grain size. Similarly, the predicted subgrain sizes for  
524 both olivine and orthopyroxene in the harzburgite samples of San404 and in orthopyrox-  
525 ene in the harzburgite sample of San508 exceeded the measured grain sizes of the respec-  
526 tive phases. Thus, subgrains were likely not able to form in a manner consistent with the  
527 subgrain-size piezometer, and consequently, we do not include these samples in our initial  
528 analysis.

529 For some samples, the range in stresses calculated from X-ray diffraction resulted in  
530 predicted subgrain sizes that straddled the measured grain size. Specifically, the stress  
531 measured from the {130} peak from olivine in the harzburgites of experiments San391,  
532 San396, San409, and San508, as well as the stress measured from the {112} peak in San508,  
533 are the only ones predicted to be sufficient to produce subgrains within the constraints of  
534 the grain size. Similarly, for orthopyroxene only the {610} and {321} peaks in San391 and  
535 the {610} peaks in San409 give stresses great enough to predict subgrain sizes smaller than  
536 the grain sizes. In our analysis, we include these samples but identify those diffraction  
537 peaks that give stresses too low for subgrain boundaries to be extensively developed at the  
538 measured grain sizes.

539 For olivine, stresses from subgrain-size piezometry are plotted against a range of  
 540 stresses measured from X-ray diffraction in Figure 8. Open markers indicate individual  
 541 diffraction peaks for which the stress was sufficiently low that the subgrain size predicted  
 542 by the piezometer is larger than the grain size of the sample. Based on analysis of the ac-  
 543 tivity of different slip systems (see Appendix B), a piezometric stress was also calculated  
 544 for the olivine stress sensor of San502 by normalising the line-intercept length by the [001]  
 545 Burgers vector ( $5.98\text{\AA}$ , Deer et al., 1988, page 4, pressure correction from Abramson et al.,  
 546 1997) rather than the [100] Burgers vector used for the other olivine samples. The change in  
 547 Burgers vector increases the calculated stress from 390 to 480 MPa for San502. The stresses  
 548 calculated using the [100] and the [001] Burgers vectors are both plotted in Figure 8 for  
 549 comparison.



**Figure 8.** Comparison of *ex-situ* stresses from subgrain-size piezometry and final *in-situ* stresses from X-ray diffraction from olivine in both monomineralic and polymineralic samples. Error bars are calculated using a Monte-Carlo based approach, as described in Section 2.5 and Appendix A. The size of each data point is scaled by the total strain experienced by the sample. Stresses from individual X-ray peaks for which the predicted subgrain size is larger than the measured grain size of the sample are plotted as open symbols. The black solid line is 1:1 and the dashed horizontal line marks the upper calibration limit for olivine in the subgrain-size piezometer (Goddard et al., 2020). Two stresses from subgrain-size piezometry have been plotted for the stress sensor of San502, one where the mean-line intercept length was normalised by the length of the [100] Burgers vector and one where it was normalised by the length of the [001] Burgers vector.

550 A strong correlation exists between the *in-situ* stress measurements from the {130}  
551 diffraction peaks and the *ex-situ* stress measurements, in both the monomineralic and polymin-  
552 eral samples (Figure 8). A moderate correlation also exists for the {112} peak, as these  
553 stresses are often similar to those from the {130} peak. The stresses from the {131} peak  
554 tend to be significantly lower than those calculated from the other diffraction peaks, and  
555 in most cases are low enough that the subgrain size predicted by the piezometer is larger  
556 than the measured grain size (open symbols in Figure 8). For the few samples that can  
557 confidently be compared, X-ray stresses from the {131} peak lie within the uncertainty of  
558 the piezometric stresses. Recently, Girard and Karato (2022) demonstrated that the bulk  
559 stress experienced by a sample is most accurately represented by the diffraction peak pro-  
560 viding the greatest stress measurement, here the {130} olivine peak. Given the particularly  
561 close correspondence between the {130} stresses and the stresses measured via subgrain-  
562 size piezometry, we conclude that subgrain-size piezometry accurately reflects the stress  
563 within individual phases in a polymineralic rock.

564 Subgrain-size piezometry therefore provides a powerful tool for measuring stress in  
565 natural rocks, but care must be taken when applying it to extremely fine-grained samples.  
566 For example, olivine within polymineralic regions of the Erro-Tobbio peridotite in Italy,  
567 have average grain sizes as low as 5  $\mu\text{m}$  (Linckens & Tholen, 2021). Such small grain  
568 sizes would require differential stresses of  $> 175$  MPa to form subgrains, an unrealistic  
569 condition considering shear zones are regions of weakening and localisation (e.g., Skemer  
570 et al., 2010; Stenvall et al., 2019; Warren & Hirth, 2006). As many other natural shear zones,  
571 such as the Josephine peridotite ( $\sim 250$   $\mu\text{m}$ , Hansen & Warren, 2015) and the Oman-United  
572 Arab Emirates ophiolite ( $\geq 100$   $\mu\text{m}$  Ambrose et al., 2018), are relatively coarse grained, the  
573 application of subgrain-size piezometry remains broad.

#### 574 **4.2 Subgrain-size Piezometry as a Tool For Unpicking Complex Stress Histories**

575 A key challenge in paleopiezometry lies in accounting for the potentially complex  
576 stress histories experienced by natural samples, compared to samples deformed in the  
577 laboratory that often experience steady-state deformation (Kidder et al., 2016; Speciale et  
578 al., 2022). It is currently not clear how much strain is required to set or reset the stress  
579 recorded by the subgrain size. Previous work on olivine by Ross et al. (1980) compared  
580 constant strain rate and constant stress experiments to those in which the stress, strain  
581 rate, or temperature was varied incrementally. In those experiments, subgrain sizes were

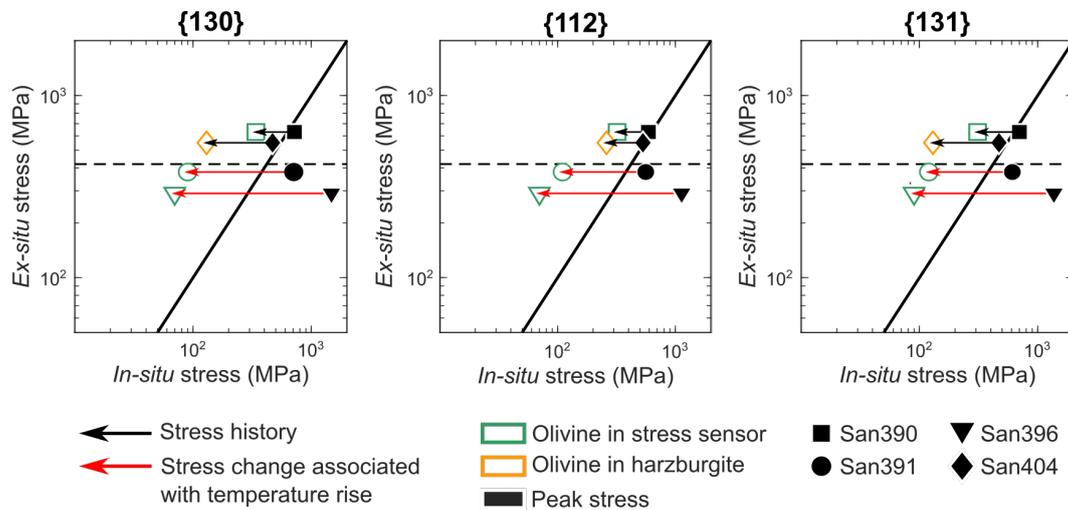
582 found to decrease with increasing stress but did not coarsen with decreasing stress, sug-  
583 gesting that the subgrain size is set by the maximum stress applied to a sample. However,  
584 experiments on steel by Qin et al. (2003) found that subgrains were able to coarsen if con-  
585 tinuously strained under stresses of a reduced magnitude, suggesting that strain, rather  
586 than time, is important for subgrain coarsening. Goddard et al. (2020) also demonstrated a  
587 correlation between subgrain-boundary spacing and the final stress experienced by olivine  
588 and quartz deformed in the laboratory, despite some samples having experienced greater  
589 (peak) stresses earlier in their deformation history.

590 To test how subgrain sizes respond to complex stress histories, we subjected the sam-  
591 ples in all but one of our experiments to either temperature or strain-rate steps, which  
592 imparted strains between 0.01 and 0.25. Based on the initial calibration by Goddard et al.  
593 (2020), we would expect stresses estimated using the subgrain-size piezometer to match  
594 the final stresses measured through X-ray diffraction. Indeed, as seen in the previous sec-  
595 tion, such a correlation is apparent in Figure 8. However, some of the samples with grain  
596 sizes predicted to be too small to contain subgrains still contain numerous subgrain bound-  
597 aries (e.g., the olivine stress sensor in San390 and the harzburgite in San404 in Figure 7). It  
598 is therefore possible that the subgrain boundaries within these samples formed during an  
599 earlier portion of the deformation history under a differential stress greater than the final  
600 stress.

601 To explore whether these ‘unexpected’ subgrain boundaries correspond to an earlier  
602 part of the deformation history, we plot in Figure 9 both the peak stresses and the final  
603 stresses measured by X-ray diffraction for samples not presented in Figure 8. To be clear,  
604 in all these remaining experiments the grain sizes were too small to accommodate sub-  
605 grains of the size predicted by the piezometer based on the final stress measured by X-ray  
606 diffraction. We exclude data from the olivine stress sensor for experiment San404, as the  
607 predicted subgrain size from the peak stress was also too large to be accommodated within  
608 the grains.

609 The subgrain size in the samples with the most prolific intragranular boundaries give  
610 piezometric stresses that match the peak stress experienced during each experiment. The  
611 remaining two samples—the olivine stress sensors in experiments San391 and San396—give  
612 piezometric stresses that are midway through the peak stress and the final stress implying  
613 that their subgrains underwent some coarsening. Notably, during experiments San391 and

San396 the stress reductions were the result of increases in temperature from 1100°C to 1150°C and 1100°C to 1270°C, respectively. The two experiments that instead record the peak stresses were held at 1100°C. We therefore conclude that, at higher temperatures, subgrain boundaries are more easily removed. Ross et al. (1980) conducted experiments at temperatures of 1000–1300°C, however the majority of experiments used for piezometry were conducted at 1100°C. Our experiments at the equivalent temperature that exhibit little subgrain evolution are roughly consistent with the observations of Ross et al. (1980). Therefore, if the grain size is too fine for the subgrains to coarsen to the size determined by a new lower stress, and temperatures are relatively low, subgrain structure within a rock could relate to a previous, even transient, high stress.

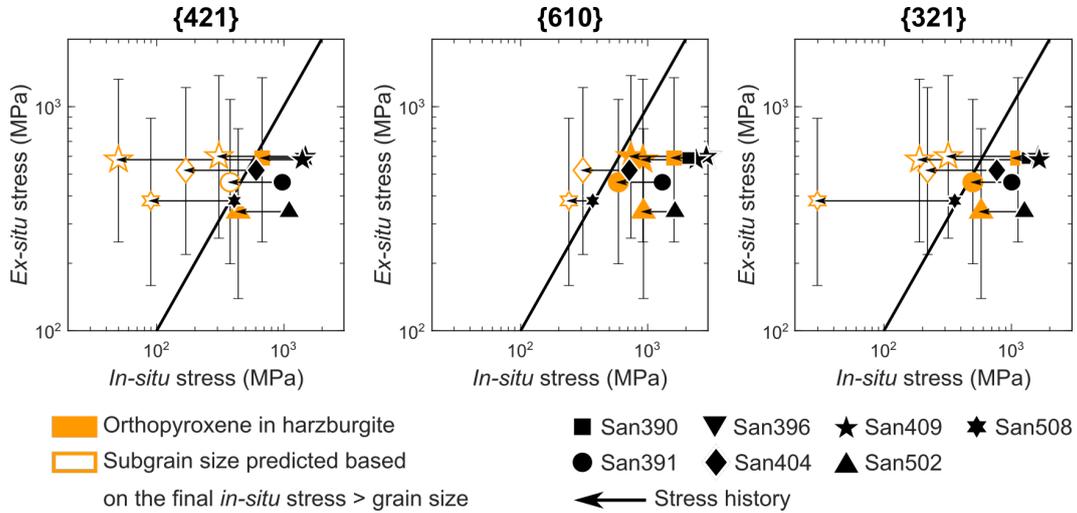


**Figure 9.** Comparison of *ex-situ* stress, from subgrain-size piezometry, and *in-situ* stress, from X-ray diffraction, for olivine in all samples for which the subgrain size predicted by the final stress was larger than the measured grain size. Black symbols represent the peak stress measured by X-ray diffraction. Horizontal arrows link the peak stress to the final stress experienced by the sample. The black solid line is 1:1 and the dashed horizontal line represents the calibration limit for olivine in the subgrain-size piezometer (Goddard et al., 2020). For clarity we did not include error bars, though errors on the piezometric stress estimates can be found in Table 4.

### 4.3 Subgrain-size Piezometry in Orthopyroxene

The experiments presented here provide an opportunity to explore whether the subgrain-size piezometer published by Goddard et al. (2020) can be reliably applied to orthopyroxene, a mineral not included in the original calibration. Such analysis would greatly expand the use of this piezometer and negate the common requirement to calibrate a subgrain-size piezometer for each mineral of interest. Stresses from subgrain-size piezometry of orthopy-

630 roxene are plotted against those from X-ray diffraction in Figure 10. Similar to Figures 8  
 631 and 9, we identify, with open symbols, stresses from X-ray diffraction that are too low for  
 632 subgrains to form at the grain sizes of these samples.



**Figure 10.** Comparison of *ex-situ* stress, from subgrain-size piezometry, and *in-situ* stress, from X-ray diffraction, for orthopyroxene in all samples. Black symbols represent the peak stress measured by X-ray diffraction and black horizontal arrows link the peak stress to the final stress experienced by the sample. Error bars are calculated using the Monte-Carlo based approach, as described in Section 2.5 and Appendix A. Stresses from individual peaks for which the subgrain size predicted by the piezometer was larger than the measured grain size are plotted as open symbols. The black solid line is 1:1.

633 There is no consistent relationship between the stresses preserved by the subgrain-  
 634 boundary spacing in orthopyroxene and either the peak stresses or the final stresses expe-  
 635 rienced by the sample. However, this lack of correlation is expected given the limited intra-  
 636 granular substructure within the orthopyroxene in most samples. In addition, the low total  
 637 strains in most of the mixtures, alongside the greater strength of orthopyroxene compared  
 638 to olivine at high confining pressures (e.g., Raterron et al., 2016), indicates that the former  
 639 may account for very little of the total strain of the aggregate. We therefore limit further  
 640 discussion to experiments San409 and San508, in which the total strain of the harzburgite  
 641 exceeds 0.20, and experiment San502, in which the sample is mostly orthopyroxene (85%)  
 642 and a total strain of 0.11 was reached.

643 For experiment San409, the final stresses in the top and bottom samples measured  
 644 through X-ray diffraction (310–740 MPa and 50–920 MPa, respectively) straddle those mea-  
 645 sured by subgrain-size piezometry (600 MPa and 580 MPa, respectively), though the large

646 range in *in-situ* stresses limits the significance of this comparison. For experiment San502,  
647 the stress of 340 MPa from subgrain-size piezometry is close to the final stress of 440 MPa  
648 measured from the {421} peak, but is less than the stresses of 930 MPa and 580 MPa from  
649 the {610} and {321} diffraction peaks, respectively. For San508, the grain size is too small  
650 to form subgrains of the size predicted by the piezometer based on the final stress. How-  
651 ever, the stresses from X-ray diffraction only began to drop during the final 0.02 of strain.  
652 The previous 0.12 of strain occurred under a near constant stress of 360–410 MPa, which  
653 is consistent with the stress of 380 MPa measured in the orthopyroxene by subgrain-size  
654 piezometry.

655 These results broadly support the use of subgrain-size piezometry on orthopyroxene  
656 as long as sufficient substructure exists. These experiments also highlight the effects of  
657 large strength contrasts when applying the subgrain-size piezometer, as the strain experi-  
658 enced by an individual mineral is an important factor in determining whether subgrains  
659 in that mineral evolve to the size predicted by the piezometric relationship.

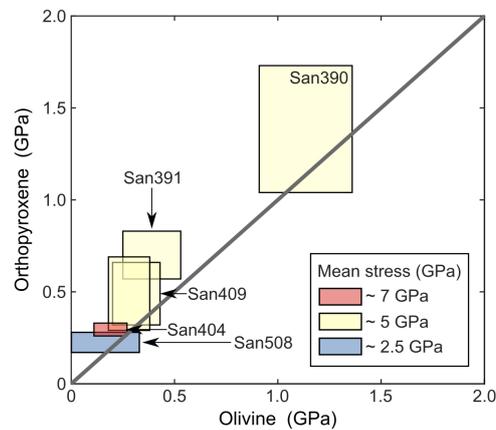
#### 660 **4.4 Stress Partitioning from X-ray Diffraction**

661 The results of this study are, to the best of our knowledge, the first to provide direct  
662 evidence that subgrain sizes reflect the stress supported by an individual phase deforming  
663 in an aggregate. A relevant question that still remains, however, is how these measured  
664 stresses relate to the macroscopic stress applied to the rock. In the work presented here,  
665 the general lack of subgrains in orthopyroxene within experiments deformed at a high  
666 mean stress limits the use of subgrain-size piezometry as a tool for exploring stress parti-  
667 tioning. However, stress partitioning can still be explored via X-ray diffraction. Relatively  
668 few studies to date have used X-ray diffraction in this way (Li et al., 2007; Wang et al.,  
669 2013) and no studies, to our knowledge, have used this method in olivine-orthopyroxene  
670 systems.

671 When considering stress partitioning, the relevant question is how the stress within an  
672 individual phase relates to the bulk stress experienced by a rock. Stacked samples have pre-  
673 viously been assumed to nominally experience the same stress (e.g., Hansen et al., 2019).  
674 This assumption holds true for our experiment San409, in which the same material was  
675 used for both the top and bottom samples (Figure 6). If the same were true of our other  
676 experiments, then the stress supported by the sample of monomineralic olivine in each ex-

677 periment should represent the bulk stress of the adjacent polyminerale sample. However,  
 678 in all experiments conducted at high mean stresses ( $> 5$  GPa), differential stresses mea-  
 679 sured within the mixture were significantly greater than those in the olivine stress sensor  
 680 (Figure 5). Silber et al. (2022) suggested that such differences in stress within an assembly  
 681 can occur if the samples are weak relative to the confining medium. Our stacked sam-  
 682 ples cannot therefore be used to explore how stresses within each phase relate to the bulk  
 683 stress, but we can see how different conditions affect the relative stresses within olivine  
 684 and orthopyroxene.

685 We calculated the average stress from each peak over the final controlled conditions  
 686 of the experiment, that is, after the final temperature or displacement-rate step (see sup-  
 687 plementary Table S3). For experiment San409, we instead averaged stress over the final  
 688 0.2 strain, due to the small amount of strain under each set of conditions. Of the stresses  
 689 calculated from the three peaks, we consider the least and greatest values to represent the  
 690 ‘stress range’ of each phase in the mixture, and compare these in Figure 11. In experiments  
 691 performed at high mean stresses ( $> 5$  GPa) and temperatures (1100–1270°C), the mean of  
 692 three X-ray diffraction stresses is 1.2–1.7 times greater in orthopyroxene than olivine. How-  
 693 ever, in experiment San508, which was deformed at a lower mean stress of  $\leq 4$  GPa, the  
 694 stresses supported by olivine and orthopyroxene are the same within error.



**Figure 11.** Comparison of the average differential stresses measured in olivine and orthopyroxene by X-ray diffraction. The boxes, colour coded for mean stress, cover the full range of stresses calculated from the different diffraction peaks of each phase.

695 This observation of a greater stress supported by the orthopyroxene when at high  
 696 mean stress can be considered in relation to Handy’s (1994) rheological mixing model.

697 As the harzburgites in this study consist mainly of olivine (~70%), and as orthopyroxene  
698 is stronger than olivine at these experimental conditions (e.g., Raterron et al., 2016), the  
699 phase distribution within these samples can be categorised as interconnected weak layers  
700 (Handy, 1990). Stresses within the individual phases of samples with interconnected weak  
701 layers have been proposed to lie close to the isostress limit (Handy, 1994), also known as  
702 the Sachs model, in which it is assumed that each grain within an aggregate experiences the  
703 same stress tensor (Sachs, 1928). Handy's (1994) model predicts a slight variation from the  
704 Sachs model, with marginally greater stress within the 'strong' phase in mixtures when the  
705 viscosity contrast between phases is less than one order of magnitude. The greater stresses  
706 within the strong orthopyroxene in this study are therefore consistent with Handy's (1994)  
707 model. Similarly, work by Li et al. (2007) using X-ray diffraction to explore stresses within  
708 composites of varying proportions of MgO and spinel measured greater stresses within  
709 the strong phase when it comprised just 25% of the mixture. Clearly, *in-situ* stress mea-  
710 surements make a good dataset to examine polymineralic mixing models, which can then  
711 enable more accurate interpretations of stress estimates from natural samples.

## 712 5 Conclusions

713 We deformed olivine and olivine-orthopyroxene mixtures in a D-DIA apparatus at  
714 temperatures and mean stresses similar to mantle conditions. *In-situ* stress measurements  
715 from X-ray diffraction are compared to *ex-situ* measurements from subgrain-size piezome-  
716 try. A good correlation between the *in-situ* and *ex-situ* stresses in olivine in both monomin-  
717 eral and polymineralic aggregates demonstrates that subgrain size is unaffected by grain-  
718 boundary pinning by secondary minerals. Subgrain size also displays a good correlation  
719 with the final stress experienced by the sample, even in experiments with complex stress  
720 histories. However, this study identifies a number of important factors to consider when  
721 applying subgrain-size piezometry to natural rocks. First, in polymineralic rocks, caution  
722 must be taken in applying the subgrain-size piezometer to strong minerals that undergo  
723 little strain, such as those behaving as passive clasts within a deforming matrix. Second,  
724 while some initial data for orthopyroxene looks promising, further experiments are still  
725 required to confirm the applicability of the Goddard et al. (2020) piezometer to minerals  
726 other than quartz and olivine, with which it was calibrated. Third, very fine-grained rocks  
727 may not have grain sizes sufficiently large to host subgrains that have formed under low  
728 stresses. Therefore, in particularly fine-grained samples, the stresses measured from ex-

729 isting subgrains may not necessarily record the most recent stress state if the associated  
730 subgrain size was larger than the grain size, but may instead record earlier deformation as-  
731 sociated with greater stresses and finer subgrain sizes. Finally, for the compositional ratios  
732 of samples deformed at high mean stresses of 4.7–9.4 GPa in this study, the stresses sup-  
733 ported by orthopyroxene and olivine in harzburgites are not equal. As such, care must be  
734 taken when extrapolating the stress measured from an individual phase to the bulk stress  
735 experienced by the sample.

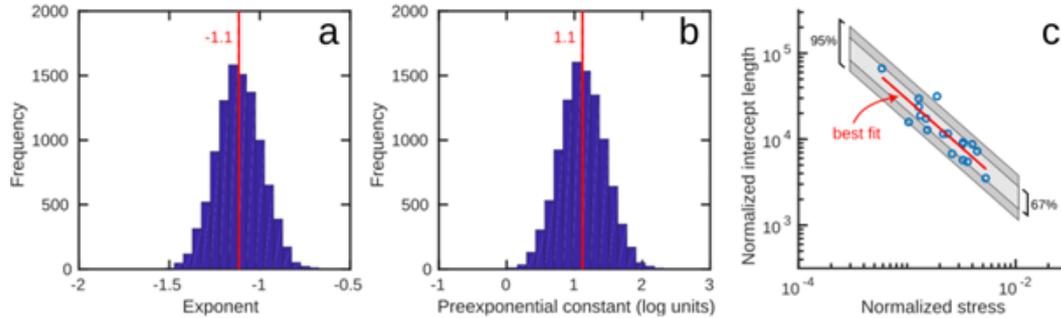
## 736 **6 Data Availability Statement**

737 Mechanical data from the Deformation-DIA experiments alongside EBSD data of re-  
738 covered samples are available from OSF via <https://doi.org/10.17605/OSF.IO/UATJG> (Goddard  
739 et al., 2023). Codes for processing EBSD data can be found at [https://github.com/](https://github.com/RellieGoddard/SGPiezometry.git)  
740 [RellieGoddard/SGPiezometry.git](https://github.com/RellieGoddard/SGPiezometry.git).

## 742 **Appendix A Error Estimation on the Subgrain-size Piezometer**

743 We use a Monte-Carlo-based approach to estimate the errors in our subgrain-size mea-  
744 surements, the errors in parameters in the subgrain-size piezometer, and errors in stress es-  
745 timates. The procedures described here are carried out in log units under the assumption,  
746 which is validated below, that errors in measurements of subgrain size are lognormally dis-  
747 tributed. As an initial step, we refit the piezometer to the data determined using a threshold  
748 misorientation angle of  $2^\circ$ , above which boundaries are measured. For each measurement  
749 of subgrain size used in this refitting, we assume that the probability of measuring the true  
750 subgrain size is represented by a normal distribution (in log units) with standard devia-  
751 tion equal to the error in subgrain-size measurement. This error is justified below to be  
752 approximately 0.13 log units. We then randomly select a value from that distribution and  
753 perturb the measured subgrain size by that amount. This new data set of stresses and per-  
754 turbed subgrain sizes is then used to determine the parameter values for the subgrain-size  
755 piezometer using a linear least-squares approach. This process is repeated 10,000 times,  
756 randomly generating new perturbed measurements on each iteration. The distributions of  
757 best-fit parameter values determined over all of these iterations are illustrated in Figures  
758 A1a and b. The means of these distributions are, within three significant digits, equivalent  
759 to the parameter values determined by a least-squares fit to the unperturbed data set. We

760 take the standard deviation of these distributions to reflect the error in estimation of the  
 761 piezometer parameters, resulting in a pre-exponential constant of  $1.1 \pm 0.3$  and an exponent  
 762 of  $-1.1 \pm 0.1$ .



**Figure A1.** Estimation of error on best-fit parameters for the subgrain-size piezometer. Distributions of parameter estimates from 10,000 individual fitting exercises for (a) the exponent and (b) the preexponential constant in the piezometer. The means of the distributions are indicated in red. The standard deviations of the distributions are taken to represent the error in the estimate. (c) Best-fit piezometer compared to data used for calibration and 67% and 95% confidence intervals. Confidence intervals are estimated assuming that the error in subgrain-size measurement is 0.13 log units.

763 Figure A1 compares the best-fit piezometer to the measured data. This figure also  
 764 includes 67% and 95% confidence intervals determined using one or two times the error in  
 765 the subgrain-size measurement, respectively. We compare these confidence intervals to the  
 766 distribution of data as a check on the validity of our error estimation. We find that setting  
 767 the error on subgrain-size measurement to 0.13 log units, as presented in Figure A1, results  
 768 in 67% and 95% confidence intervals that contain 67% and 94% of the data, respectively.  
 769 This exercise not only demonstrates that an error of 0.13 log units in our subgrain-size  
 770 measurements is consistent with the data, but it also provides confidence that the error in  
 771 those measurements is lognormally distributed.

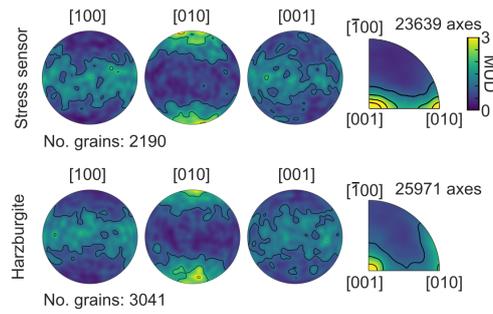
772 A similar procedure can be used to estimate the errors in stresses predicted using the  
 773 piezometer. Here we again assume that the probability distributions for the subgrain size,  
 774 pre-exponential constant, and exponent follow lognormal distributions with standard deviations  
 775 (in log units) given by 0.13, 0.3, and 0.1, respectively, and with means given by the  
 776 measured or best-fit values. We randomly draw values from these distributions and use  
 777 them in conjunction with appropriate values of the Burgers vector and shear modulus to  
 778 make a prediction of the applied differential stress. This process is repeated 10,000 times to  
 779 generate a distribution of predicted stresses. The mean of this distribution (in log units) is

780 equivalent to the predicted stress used simply with the measured subgrain size and best-fit  
 781 piezometer parameters. We take the standard deviation of this distribution to represent  
 782 the error in the predicted stress. As an illustrative example, for a measured subgrain size  
 783 of 1.5  $\mu\text{m}$  with Burgers vector of 0.475 nm and shear modulus of 77.8 GPa, we estimate the  
 784 normalised stress to be  $10^{-2.2\pm 0.4}$  and the absolute stress to be  $510_{-290}^{+680}$  MPa.

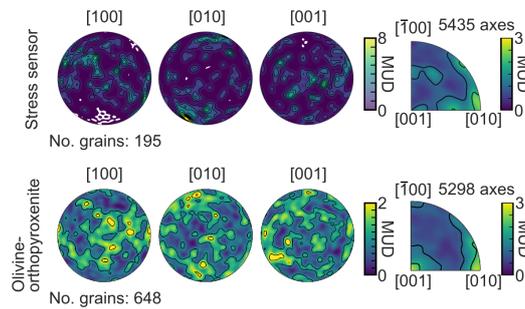
## 785 **Appendix B Choice of Burgers vector for Experiments San502 and San508**

786 The subgrain-size piezometer presented by Goddard et al. (2020), was calibrated us-  
 787 ing high-temperature experiments in which dislocation creep of olivine occurs primarily  
 788 by the slip system (010)[100] (e.g., Bai et al., 1991; Hansen et al., 2014; Wallis et al., 2019).  
 789 However, two experiments from this study, San502 and San508, were carried out at lower  
 790 temperatures ( $\leq 900^\circ\text{C}$ ) where the (100)[001] slip system, and particularly [001] screw dislo-  
 791 cations, can instead dominate deformation (e.g., Carter & Ave'Lallemant, 1970; Druiventak  
 792 et al., 2011; Gaboriaud et al., 1981; Idrissi et al., 2016; Wallis et al., 2020). Here, we interpret  
 793 the slip systems active in these two samples based on the pole figures and misorientation  
 794 inverse pole figures. The pole figures are lower-hemisphere projections, are constructed  
 795 using one point per grain and a kernel half-width of  $5^\circ$ , and are oriented such that the  
 796 maximum principal stress axis ( $\sigma_1$ ) is vertical. For all inverse pole figures, misorientation  
 797 axes were plotted for neighbouring pixels with misorientation angles between  $2^\circ$  and  $15^\circ$ .  
 798 Colour scales in Figures B1–B3 represent multiples of uniform distribution (MUD).

799 Pole figures and inverse pole figures for olivine in both the olivine stress sensor and the  
 800 harzburgite sample of experiment San508 are plotted in Figure B1. Olivine exhibits a crystal  
 801 preferred orientation (CPO) with [100] and [001] axes in girdles perpendicular to  $\sigma_1$  and  
 802 [010] axes in a cluster parallel to  $\sigma_1$ . Taken in isolation, this CPO indicates that (010) is the  
 803 slip plane of the dominant slip system and that [100] and/or [001] is the dominant Burgers  
 804 vector. The misorientation axes of olivine form a dominant cluster parallel to [001] and a  
 805 secondary cluster parallel to [010]. Misorientation axes parallel to [001] are characteristic of  
 806 tilt boundaries composed of edge dislocations on the (010)[100] slip system. Misorientation  
 807 axes parallel to [010] can result from three possible types of subgrain boundary, specifically  
 808 tilt boundaries composed of edge dislocations on the (001)[100] or (100)[001] slip systems  
 809 and twist boundaries composed of [100] and [001] screw dislocations. Taken together, these  
 810 observations indicate that (010)[100] was the dominant slip system, whilst (001)[100] and  
 811 or (100)[001] were also active but to a lesser degree.



**Figure B1.** Pole and inverse pole figures for olivine in the stress sensor and harzburgite samples of San508. Colour represents multiples of uniform distribution (MUD).

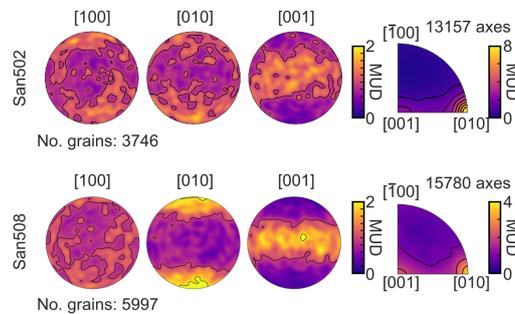


**Figure B2.** Pole and inverse pole figures for olivine in the stress sensor and olivine-orthopyroxenite samples of San502. Colour represents multiples of uniform distribution (MUD).

812 Figure B2 contains the equivalent pole figures and inverse pole figures for olivine in  
 813 both the stress sensor and the olivine-orthopyroxenite sample in experiment San502. In  
 814 all pole figures olivine displays no discernible CPO. For the stress sensor, this lack of CPO  
 815 may in part be due to the low number of grains available for analysis (195). However, this  
 816 reasoning does not apply to the olivine in the olivine orthopyroxenite, for which 648 grains  
 817 were measured. Despite the lack of CPOs, the presence of subgrain boundaries within  
 818 olivine in both the stress sensor and the mixture implies that dislocations were active dur-  
 819 ing deformation. Similar to San508, olivine in both samples exhibits subgrain boundaries  
 820 with misorientation axes clustered approximately parallel to  $[001]$  and  $[010]$ . Unlike San508,  
 821 the olivine in the olivine orthopyroxenite also contains subgrain boundaries with misori-  
 822 entation axes clustered parallel to  $[100]$ . These latter subgrain boundaries can represent tilt  
 823 boundaries composed of  $(010)[001]$  edge dislocations. In both cases the cluster parallel to  
 824  $[001]$  is secondary in strength. Without crystal preferred orientations it is not possible to  
 825 identify the exact slip system, however the misorientation axes indicate that  $(010)[100]$  is

826 not likely to be the dominant slip system and therefore the dominant Burgers vector was  
 827 likely [001].

828 Figure B3 presents pole and inverse pole figures for orthopyroxene in San502 and  
 829 San508. In both cases, misorientation axes of subgrain boundaries are predominantly clus-  
 830 tered around [010], indicating that the subgrain boundaries are formed by dislocations on  
 831 the (001)[100] and/or (100)[001] slip systems. Both samples also exhibit a submaximum  
 832 of misorientation axes parallel to [001], indicating a subpopulation of subgrain bound-  
 833 aries composed of dislocations on the (010)[100] and/or (100)[010] slip systems. In sample  
 834 San502, both [100] and [010] axes are clustered parallel to  $\sigma_1$  with [001] axes in a girdle nor-  
 835 mal to  $\sigma_1$ . These grain orientations indicate that [001] was the dominant slip direction in  
 836 this sample and, in combination with the interpretations from misorientation axes of sub-  
 837 grain boundaries, suggest that (100)[001] is the dominant slip system. In sample San508,  
 838 [100] axes are randomly oriented, [010] axes cluster parallel to  $\sigma_1$ , and [001] axes form a  
 839 girdle normal to  $\sigma_1$ . These grain orientations suggest that (010)[001] was the slip system  
 840 providing the greatest contribution to grain rotations. Overall, these results indicate that  
 841 [001] was the dominant Burgers vector in orthopyroxene in these experiments, which is  
 842 consistent with previous observations of experimental and natural samples (Jung et al.,  
 843 2010).



**Figure B3.** Pole figures and inverse pole figures for orthopyroxene in the harzburgite sample of San508 and the olivine-orthopyroxenite sample of San502. Colour represents multiples of uniform distribution (MUD).

#### 844 **Acknowledgments**

845 We are grateful for the expertise and technical assistance of Haiyan Chen at beamline  
 846 6-B,M-B at the Advanced Photon Source. Use of the Advanced Photon Source, an Of-  
 847 fice of Science User Facility operated for the U.S. Department of Energy (DOE) by Ar-

gonne National Laboratory, was supported by U.S. DOE under Contract No. DE-AC02-06CH11357. Experiments were run at the Argonne National Laboratory under general user proposals (GUP) 55176 and 68700. We are grateful for the assistance of Jamie Long and James King with sample preparation and Jon Wells for thin-section preparation. Leif Tolke and Amanda Dillman generously provided sample materials. This work was supported by NERC Environmental Research DTP grant NE/L002612/1, University of Oxford, to R.M.G., National Science Foundation grants EAR-1806791 to K.M.K. and EAR-2022433 to L.N.H., a UK Research and Innovation Future Leaders Fellowship [grant number MR/V021788/1] to D.W., and a grant from the Netherlands Organisation for Scientific Research, User Support Programme Space Research [grant number ALWGO.2018.038] to D.W. Support for EBSD data collection at the Marine Biological Laboratory was provided by National Science Foundation grant EAR-2003389 to A.J.C. Portions of this work were performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. LLNL-JRNL-851361.

## References

- Abramson, E., Brown, J. M., Slutsky, L. J., & Zaug, J. (1997). The elastic constants of san carlos olivine to 17 gpa. *Journal of Geophysical Research: Solid Earth*, *102*, pp. 12253-12263. doi: <https://doi.org/10.1029/97JB00682>
- Ambrose, T. K., Wallis, D., Hansen, L. N., Waters, D. J., & Searle, M. P. (2018). Controls on the rheological properties of peridotite at a palaeosubduction interface: A transect across the base of the oman–uae ophiolite. *Earth and Planetary Science Letters*, *491*, pp. 193-206. doi: <https://doi.org/10.1016/j.epsl.2018.03.027>
- Angel, R. J., & Hugh-Jones, D. A. (1994). Equations of state and thermodynamic properties of enstatite pyroxenes. *Journal of Geophysical Research: Solid Earth*, *99*, pp.19777–19783. doi: <https://doi.org/10.1029/94JB01750>
- Ashby, M. F. (1970). The deformation of plastically non-homogeneous materials. *Philosophical Magazine*, *21*, pp. 399-424. doi: <https://doi.org/10.1080/14786437008238426>
- Bachmann, F., Hielscher, R., & Schaeben, H. (2010). Texture analysis with mtex – free and open source software toolbox. *Solid State Phenomena*, *160*, pp. 63-68. doi: <https://doi.org/10.4028/www.scientific.net/SSP.160.63>
- Bai, Q., Mackwell, S. J., & Kohlstedt, D. L. (1991). High-temperature creep of olivine single crystals 1. mechanical results for buffered samples. *Journal of Geophysical Research:*

- 880 *Solid Earth*, 96, pp. 2441-2463. doi: <https://doi.org/10.1029/90JB01723>
- 881 Behr, W. M., & Platt, J. P. (2011). A naturally constrained stress profile through the middle  
882 crust in an extensional terrane. *Earth and Planetary Science Letters*, 303, pp. 181-192.  
883 doi: <https://doi.org/10.1016/j.epsl.2010.11.044>
- 884 Boutonnet, E., Leloup, P. H., Sassier, C., Gardien, V., & Ricard, Y. (2013). Ductile strain  
885 rate measurements document long-term strain localization in the continental crust.  
886 *Geology*, 41, pp. 819–822. doi: <https://doi.org/10.1130/G33723.1>
- 887 Burnley, P. C., & Kaboli, S. (2019). Elastic plastic self-consistent (epsc) modeling of san  
888 carlos olivine deformed in a d-dia apparatus. *American Mineralogist*, 104, pp. 276-281.  
889 doi: <https://doi.org/10.2138/am-2019-6666>
- 890 Bystricky, M., Heidelbach, F., & Mackwell, S. (2006).  
891 *Large-strain deformation and strain partitioning in polyphase rocks: Dislocation creep of*  
892 *olivine–magnesiowüstite aggregates*, 427, pp. 115-132. doi: [https://doi.org/10.1016/](https://doi.org/10.1016/j.tecto.2006.05.025)  
893 [j.tecto.2006.05.025](https://doi.org/10.1016/j.tecto.2006.05.025)
- 894 Campbell, L. R., & Menegon, L. (2019). Transient high strain rate during localized viscous  
895 creep in the dry lower continental crust (lofoten, norway). *Journal of Geophysical Re-*  
896 *search: Solid Earth*, 124, pp. 10240-10260. doi: <https://doi.org/10.1029/2019JB018052>
- 897 Carter, N. L., & Ave'Lallemant, H. G. (1970). High temperature flow of dunite and peri-  
898 dotite. *Geological Society of America Bulletin*, 81, pp. 2181–2202. doi: [https://doi.org/](https://doi.org/10.1130/0016-7606(1970)81[2181:HTFODA]2.0.CO;2)  
899 [10.1130/0016-7606\(1970\)81\[2181:HTFODA\]2.0.CO;2](https://doi.org/10.1130/0016-7606(1970)81[2181:HTFODA]2.0.CO;2)
- 900 Chai, M., Brown, J. M., & Slutsky, L. J. (1997). The elastic constants of an aluminous  
901 orthopyroxene to 12.5 gpa. *Journal of Geophysical Research: Solid Earth*, 102, pp. 14779-  
902 14785. doi: <https://doi.org/10.1029/97JB00893>
- 903 Christensen, N. I. (1966). Elasticity of ultrabasic rocks. *Journal of Geophysical Research (1896-*  
904 *1977)*, 71, pp. 5921-5931. doi: <https://doi.org/10.1029/JZ071i024p05921>
- 905 Cross, A. J., & Skemer, P. (2017). Ultramylonite generation via phase mixing in high-strain  
906 experiments. *Journal of Geophysical Research: Solid Earth*, 112, pp. 1744-1759. doi:  
907 <https://doi.org/10.1002/2016JB013801>
- 908 Deer, W., Howie, R. A., & Zussman, J. (1988). *An introduction to the rock-forming minerals*.  
909 London, UK: Longman.
- 910 Dixon, N. A., & Durham, W. B. (2018). Measurement of activation volume for creep of dry  
911 olivine at upper-mantle conditions. *Journal of Geophysical Research: Solid Earth*, 123,  
912 pp. 8459-8473. doi: <https://doi.org/10.1029/2018JB015853>

- 913 Druiventak, A., Trepmann, C. A., Renner, J., & Hanke, K. (2011). Low-temperature plastic-  
914 ity of olivine during high stress deformation of peridotite at lithospheric conditions  
915 — an experimental study. *Earth and Planetary Science Letters*, 311, pp. 199-211. doi:  
916 <https://doi.org/10.1016/j.epsl.2011.09.022>
- 917 Durham, W. B., Goetze, C., & Blake, B. (1977). Plastic flow of oriented single crystals  
918 of olivine: 2. observations and interpretations of the dislocation structures.  
919 *Journal of Geophysical Research*, 82, pp. 5755-5770. doi: [https://doi.org/10.1029/  
920 JB082i036p05755](https://doi.org/10.1029/JB082i036p05755)
- 921 Durham, W. B., Mei, S., Kohlstedt, D., Wang, L., & Dixon, N. (2009). New measurements  
922 of activation volume in olivine under anhydrous conditions. *Physics of the Earth and  
923 Planetary Interiors*, 172, pp. 67-73. doi: <https://doi.org/10.1016/j.pepi.2008.07.045>
- 924 Durham, W. B., Weidner, D. J., Karato, S.-I., & Wang, Y. (2002). New developments in  
925 deformation experiments at high pressure. *Reviews in Mineralogy Geochemistry*, 51,  
926 pp. 21–49. doi: <https://doi.org/10.2138/gsrmg.51.1.21>
- 927 Evans, B., Renner, J., & Hirth, G. (2001). A few remarks on the kinetics of static grain  
928 growth in rocks. *International Journal of Earth Sciences*, 90, pp. 88-103. doi: [https://  
929 doi.org/10.1007/s005310000150](https://doi.org/10.1007/s005310000150)
- 930 Gaboriaud, R. J., Darot, M., Gueguen, Y., & Woignard, J. (1981). Dislocations in olivine  
931 indented at low temperatures. *Physics and Chemistry of Minerals*, 7, pp. 100-104. doi:  
932 <https://doi.org/10.1007/BF00309460>
- 933 Girard, J., & ichiro Karato, S. (2022). An experimental study of evolution of strength of  
934 olivine aggregates under upper mantle conditions using in-situ synchrotron x-ray..
- 935 Goddard, R. M., Hansen, L. N., Wallis, D., Stipp, M., III, C. W. H., Kumamoto, K. M., &  
936 Kohlstedt, D. L. (2020). A subgrain-size piezometer calibrated for ebsd. *Geophysical  
937 Research Letters*, 47. doi: <https://doi.org/10.1029/2020GL090056>
- 938 Goddard, R. M., Kumamoto, K. M., Hansen, L. N., Wallis, D., Cross, A. J., & Thom, C. A.  
939 (2023). *D-dia: Subgrain-size piezometry* [dataset]. OSF. doi: [https://doi.org/10.17605/  
940 OSF.IO/UATJG](https://doi.org/10.17605/OSF.IO/UATJG)
- 941 Handy, M. R. (1990). The solid-state flow of polymineralic rocks. *Journal of Geophysical Re-  
942 search: Solid Earth*, 95, pp. 8647-8661. doi: <https://doi.org/10.1029/JB095iB06p08647>
- 943 Handy, M. R. (1994). Flow laws for rocks containing two non-linear viscous phases:  
944 A phenomenological approach. *Journal of Structural Geology*, 16, pp. 287-301. doi:  
945 [https://doi.org/10.1016/0191-8141\(94\)90035-3](https://doi.org/10.1016/0191-8141(94)90035-3)

- 946 Hansen, L. N., Kumamoto, K. M., Thom, C. A., Wallis, D., Durham, W. B., Goldsby, D. L., . . .  
 947 Kohlstedt, C. D. M. . D. L. (2019). Low-temperature plasticity in olivine: Grain size,  
 948 strain hardening, and the strength of the lithosphere. *Journal of Geophysical Research:*  
 949 *Solid Earth*, 124, pp. 5427–5449. doi: <https://doi.org/10.1029/2018JB016736>
- 950 Hansen, L. N., & Warren, J. M. (2015). Quantifying the effect of pyroxene on deformation  
 951 of peridotite in a natural shear zone. *Journal of Geophysical Research: Solid Earth*, 120,  
 952 pp. 717-2738. doi: <https://doi.org/10.1002/2014JB011584>
- 953 Hansen, L. N., Zhao, Y.-H., Zimmerman, M. E., & Kohlstedt, D. L. (2014). Protracted fabric  
 954 evolution in olivine: Implications for the relationship among strain, crystallographic  
 955 fabric, and seismic anisotropy. *Earth and Planetary Science Letters*, 387, pp. 157-168.  
 956 doi: <https://doi.org/10.1016/j.epsl.2013.11.009>
- 957 Hiraga, T., Tachibana, C., Ohashi, N., & Sano, S. (2010). Grain growth systematics for  
 958 forsterite ± enstatite aggregates: Effect of lithology on grain size in the upper mantle.  
 959 *Earth and Planetary Science Letters*, 291, pp. 10-20. doi: [https://doi.org/10.1016/j.epsl](https://doi.org/10.1016/j.epsl.2009.12.026)  
 960 .2009.12.026
- 961 Huang, Y., Humphreys, F., & Ferry, M. (2000). Hot deformation and annealing of cube  
 962 oriented aluminium single crystals. *Materials Science and Technology*, 16, pp. 1367-  
 963 1371. doi: <https://doi.org/10.1179/026708300101507271>
- 964 Idrissi, H., Bollinger, C., Boioli, F., Schryvers, D., & Cordier, P. (2016). Low-temperature  
 965 plasticity of olivine revisited with in situ tem nanomechanical testing. *Science Ad-*  
 966 *vances*, 2, e1501671. doi: <https://doi.org/10.1126/sciadv.1501671>
- 967 III, C. W. H., & Kronenberg, A. K. (2010). Accurate differential stress measurement using  
 968 the molten salt cell and solid salt assemblies in the griggs apparatus with applications  
 969 to strength, piezometers and rheology. *Tectonophysics*, 494, pp.17-31. doi: [https://](https://doi.org/10.1016/j.tecto.2010.08.001)  
 970 [doi.org/10.1016/j.tecto.2010.08.001](https://doi.org/10.1016/j.tecto.2010.08.001)
- 971 Isaak, D. G. (1992). High-temperature elasticity of iron-bearing olivines. *Journal of Geophys-*  
 972 *ical Research: Solid Earth*, 97, pp. 1871-1885. doi: <https://doi.org/10.1029/91JB02675>
- 973 Jung, H., Park, M., Jung, S., & Lee, J. (2010). Lattice preferred orientation, water content,  
 974 and seismic anisotropy of orthopyroxene. *Journal of Earth Science*, 21, pp. 555–568.  
 975 doi: <https://doi.org/10.1007/s12583-010-0118-9>
- 976 Karato, S. (2012). *Deformation of earth materials: An introduction to the rheology of solid earth*.  
 977 Cambridge University Press.

- 978 Karato, S. (2021). A theory of inter-granular transient dislocation creep: Implications for the  
979 geophysical studies on mantle rheology. *Journal of Geophysical Research: Solid Earth*,  
980 126. doi: <https://doi.org/10.1029/2021JB022763>
- 981 Karato, S., Toriumi, M., & Fujii, T. (1980). Dynamic recrystallization of olivine single crystals  
982 during high temperature creep. *Geophysical Research Letters*, 7, pp. 649-652. doi:  
983 <https://doi.org/10.1029/GL007i009p00649>
- 984 Kidder, S., Hirth, G., Avouac, J. P., & Behr, W. (2016). The influence of stress history on  
985 the grain size and microstructure of experimentally deformed quartzite. *Journal of*  
986 *Structural Geology*, 83, pp.194-206. doi: <https://doi.org/10.1016/j.jsg.2015.12.004>
- 987 Kohlstedt, D. L., & Weathers, M. S. (1980). Deformation-induced microstructures, paleo-  
988 piezometers, and differential stresses in deeply eroded fault zones. *Journal of*  
989 *Geophysical Research: Solid Earth*, 85, pp. 6269-6285. doi: [https://doi.org/10.1029/](https://doi.org/10.1029/JB085iB11p06269)  
990 [JB085iB11p06269](https://doi.org/10.1029/JB085iB11p06269)
- 991 Kumazawa, M., & Anderson, O. L. (1969). Elastic moduli, pressure derivatives,  
992 and temperature derivatives of single-crystal olivine and single-crystal forsterite.  
993 *Journal of Geophysical Research*, 74, pp. 5961-5972. doi: [https://doi.org/10.1029/](https://doi.org/10.1029/JB074i025p05961)  
994 [JB074i025p05961](https://doi.org/10.1029/JB074i025p05961)
- 995 Li, L., Addad, A., Weidner, D., Long, H., & Chen, J. (2007). High pressure deformation  
996 in two-phase aggregates. *Tectonophysics*, 439, pp. 107-117. doi: [https://doi.org/10](https://doi.org/10.1016/j.tecto.2007.04.004)  
997 [.1016/j.tecto.2007.04.004](https://doi.org/10.1016/j.tecto.2007.04.004)
- 998 Li, L., Weidner, D., Raterron, P., Chen, J., Vaughan, M., Mei, S., & Durham, B. (2006). Deformation  
999 of olivine at mantle pressure using the d-dia. *European Journal of Mineralogy*,  
1000 18, pp. 7-19. doi: <https://doi.org/10.1127/0935-1221/2006/0018-0007>
- 1001 Linckens, J., & Tholen, S. (2021). Formation of ultramylonites in an upper mantle shear  
1002 zone, erro-tobbio, italy. *Minerals*, 11. doi: <https://doi.org/10.3390/min11101036>
- 1003 Luton, M., & Sellars, C. (1969). Dynamic recrystallization in nickel and nickel-iron alloys  
1004 during high temperature deformation. *Acta Metallurgica*, 17, pp. 1033-1043. doi:  
1005 [https://doi.org/10.1016/0001-6160\(69\)90049-2](https://doi.org/10.1016/0001-6160(69)90049-2)
- 1006 Maitland, T., & Sitzman, S. (2007). Scanning microscopy for nanotechnology: Techniques  
1007 and applications. In (p. pp. 41-75). Springer.
- 1008 Mao, Z., Fan, D., Lin, J.-F., Yang, J., Tkachev, S. N., Zhuravlev, K., & Prakapenka, V. B.  
1009 (2015). Elasticity of single-crystal olivine at high pressures and temperatures. *Earth*  
1010 *and Planetary Science Letters*, 426, pp. 204-215. doi: <https://doi.org/10.1016/j.epsl>

- 1011 .2015.06.045
- 1012 Mei, S., Suzuki, A. M., Kohlstedt, D. L., Dixon, N. A., & Durham, W. B. (2010). Experi-  
 1013 mental constraints on the strength of the lithospheric mantle. *Journal of Geophysical*  
 1014 *Research: Solid Earth*, 115, Q08012. doi: <https://doi.org/10.1029/2009JB006873>
- 1015 Menegon, L., Campbell, L., Mancktelow, N., Camacho, A., Wex, S., Papa, S., ... Pen-  
 1016 nacchioni, G. (2021). The earthquake cycle in the dry lower continental crust: in-  
 1017 sights from two deeply exhumed terranes (musgrave ranges, australia and lofoten,  
 1018 norway). *Philosophical transactions of the Royal Society A*, 379, p. 20190416. doi:  
 1019 <https://doi.org/10.1098/rsta.2019.0416>
- 1020 Meyers, C., Kohlstedt, D., & Zimmerman, M. (2017, December). Densification and grain  
 1021 growth in polycrystalline olivine rocks synthesized by evacuated hot-pressing. In  
 1022 *Agu fall meeting abstracts*.
- 1023 Nicolas, A. (1978). Stress estimates from structural studies in some mantle peridotites.  
 1024 *Philosophical Transactions of the Royal Society of London. Series A, Mathematical and Phys-*  
 1025 *ical Sciences*, 288, pp. 49–57. doi: <https://doi.org/10.1098/rsta.1978.0005>
- 1026 O'Haver, T. (2018). Pragmatic introduction to signal processing 2019: Applications in  
 1027 scientific measurement. *Independently Published*.
- 1028 Qin, Y., Götz, G., & Blum, W. (2003). Subgrain structure during annealing and creep of the  
 1029 cast martensitic cr-steel g-x12crmowvnb n 10-1-1. *Materials Science and Engineering: A*,  
 1030 341, pp. 211-215. doi: [https://doi.org/10.1016/S0921-5093\(02\)00215-0](https://doi.org/10.1016/S0921-5093(02)00215-0)
- 1031 Raleigh, C. B., & Kirby, S. H. (1970). Creep in the upper mantle. *Mineralogical Society of*  
 1032 *America Special Paper*, 180, pp. 113-121.
- 1033 Raterron, P., Fraysse, G., Girard, J., & III, C. W. H. (2016). Strength of orthoenstatite single  
 1034 crystals at mantle pressure and temperature and comparison with olivine. *Earth and*  
 1035 *Planetary Science Letters*, 450, pp. 326-336. doi: <https://doi.org/10.1016/j.epsl.2016.06>  
 1036 .025
- 1037 Ross, J. V., Lallemand, H. G. A., & Carter, N. L. (1980). Stress dependence of recrystallized-  
 1038 grain and subgrain size in olivine. *Tectonophysics*, 70, pp. 39-61. doi: [https://doi.org/10.1016/0040-1951\(80\)90020-7](https://doi.org/10.1016/0040-1951(80)90020-7)
- 1039
- 1040 Sachs, G. Z. (1928). The plastic deformation mode of polycrystals. *Zeitschrift des Vereines*  
 1041 *Deutscher Ingenieure*.
- 1042 Servi, I. S., Norton, J. T., & Grant, N. J. (1952). Some observations of subgrain formation  
 1043 during creep in high purity aluminum. *The Journal of The Minerals, Metals Materials*

- 1044 *Society*, 4, pp. 965–971. doi: <https://doi.org/10.1007/BF03397754>
- 1045 Shimizu, I. (2008). Theories and applicability of grain size piezometers: The role of dy-  
1046 namic recrystallization mechanisms. *Journal of Structural Geology*, 30, pp. 899-917.  
1047 doi: <https://doi.org/10.1016/j.jsg.2008.03.004>
- 1048 Silber, R. E., Girard, J., & ichiro Karato, S. (2022). Effects of pressure on diffusion creep  
1049 in wet olivine aggregates. *Physics of the Earth and Planetary Interiors*, 324, pp. 106840.  
1050 doi: <https://doi.org/10.1016/j.pepi.2022.106840>
- 1051 Singh, A. K., Balasingh, C., kwang Mao, H., Hemley, R. J., & Shu, J. (1998). Analysis of  
1052 lattice strains measured under nonhydrostatic pressure. *Journal of Applied Physics*, 83,  
1053 pp. 7567–7575. doi: <https://doi.org/10.1063/1.367872>
- 1054 Skemer, P., Warren, J. M., Kelemen, P. B., & hirth, G. (2010). Microstructural and rheological  
1055 evolution of a mantle shear zone. *Journal of Petrology*, 51, pp. 43-53. doi: [https://doi.org/10.1093/](https://doi.org/10.1093/petrology/egp057)  
1056 [petrology/egp057](https://doi.org/10.1093/petrology/egp057)
- 1057 Smith, C. (1948). Zener pinning. *Transactions of the Metallurgical Society of AIME*, 175, pp.  
1058 15–51.
- 1059 Soleymani, H., Kidder, S., Hirth, G., & Garapić, G. (2020). The effect of cooling during  
1060 deformation on recrystallized grain-size piezometry. *Geology*, 46, pp. 531–535. doi:  
1061 <https://doi.org/10.1130/G46972.1>
- 1062 Speciale, P., Tokle, L., & Behr, W. (2022). Feldspar and orthopyroxene piezometers con-  
1063 strained using quartz–feldspar and olivine–orthopyroxene mineral pairs from nat-  
1064 ural mylonites. *Journal of Structural Geology*, 154, 104495. doi: [https://doi.org/](https://doi.org/10.1016/j.jsg.2021.104495)  
1065 [10.1016/j.jsg.2021.104495](https://doi.org/10.1016/j.jsg.2021.104495)
- 1066 Stenvall, C. A., Åke Fagereng, & Diener, J. F. A. (2019). Weaker than weakest: On the  
1067 strength of shear zones. *Geophysical Research Letters*, 46, 7404-7413.
- 1068 Stipp, M., & Tullis, J. (2003). The recrystallized grain size piezometer for quartz. *Geophysical*  
1069 *Research Letters*, 30. doi: <https://doi.org/10.1029/2003GL018444>
- 1070 Tasaka, M., Zimmerman, M. E., Kohlstedt, D. L., Stünitz, H., & Heilbronner, R. (2017).  
1071 Rheological weakening of olivine + orthopyroxene aggregates due to phase mixing:  
1072 Part 2. microstructural development. *Journal of Geophysical Research: Solid Earth*, 122,  
1073 pp. 7597-7612. doi: <https://doi.org/10.1002/2017JB014311>
- 1074 Toriumi, M. (1979). Relation between dislocation density and subgrain size of naturally  
1075 deformed olivine in peridotites. *Contributions to Mineralogy and Petrology*, 68, pp.  
1076 181–186. doi: <https://doi.org/10.1007/BF00371899>

- 1077 Trepmann, C. A., & Seybold, L. (2019). Deformation at low and high stress-loading rates.  
1078 *Geoscience Frontiers*, *10*, pp. 43-54. doi: <https://doi.org/10.1016/j.gsf.2018.05.002>
- 1079 Tullis, J. A. (1979). High temperature deformation of rocks and minerals. *Tectonophysics*,  
1080 *17*, pp. 1137-1154. doi: <https://doi.org/10.1029/RG017i006p01137>
- 1081 Twiss, R. (1977). Stress in the earth. In (p. pp. 227-244). Birkhäuser, Basel. doi: [https://doi.org/10.1007/978-3-0348-5745-1\\_13](https://doi.org/10.1007/978-3-0348-5745-1_13)
- 1082  
1083 Twiss, R. (1986). Variable sensitivity piezometric equations for dislocation density and sub-  
1084 grain diameter and their relevance to olivine and quartz. In B. E. Hobbs & H. C. Heard  
1085 (Eds.), *Mineral and rock deformation: laboratory studies* (p. pp. 247–263). AGU Geophysical  
1086 Monographs.
- 1087 van der Wal, D., Chopra, P., Drury, M., & Gerald, J. (1993). Relationships between dy-  
1088 namically recrystallized grain size and deformation conditions in experimentally de-  
1089 formed olivine rocks. *Geophysical Research Letters*, *20*, pp. 1479–1482. doi: <https://doi.org/10.1029/93GL01382>
- 1090  
1091 Wallis, D., Hansen, L., Tasaka, M., Kumamoto, K., Parsons, A., Lloyd, G., ... Wilkinson,  
1092 A. (2019). The impact of water on slip system activity in olivine and the formation  
1093 of bimodal crystallographic preferred orientations. *Earth and Planetary Science Letters*,  
1094 *508*, pp. 51-61. doi: <https://doi.org/10.1016/j.epsl.2018.12.007>
- 1095 Wallis, D., Hansen, L. N., Kumamoto, K. M., Thom, C. A., Plümper, O., Ohl, M., ... Wilkin-  
1096 son, A. J. (2020). Dislocation interactions during low-temperature plasticity of olivine  
1097 and their impact on the evolution of lithospheric strength and planetary science  
1098 letters. *Earth and Planetary Science Letters*, *543*, 116349. doi: <https://doi.org/10.1016/j.epsl.2020.116349>
- 1099  
1100 Wang, Y., Durham, W. B., Getting, I. C., & Weidner, D. J. (2003). The deformation-dia: A  
1101 new apparatus for high temperature triaxial deformation to pressures up to 15 gpa.  
1102 *Review of Scientific Instruments*, *74*, pp. 3002–3011. doi: <https://doi.org/10.1063/1.1570948>
- 1103  
1104 Wang, Y., Hilaiet, N., Nishiyama, N., Yahata, N., Tsuchiya, T., Morard, G., & Fiquet, G.  
1105 (2013). High-pressure, high-temperature deformation of cago3 (perovskite)±mgo  
1106 aggregates: Implications for multiphase rheology of the lower mantle. *Geochemistry*,  
1107 *Geophysics, Geosystems*, *14*, pp. 3389-3408. doi: <https://doi.org/10.1002/ggge.20200>
- 1108 Warren, J. M., & Hirth, G. (2006). Grain size sensitive deformation mechanisms in naturally  
1109 deformed peridotites. *Earth and Planetary Science Letters*, *248*, 438-450. doi: <https://doi.org/10.1016/j.epsl.2006.05.011>

1110 doi.org/10.1016/j.epsl.2006.06.006

1111 Weidner, D. J., Vaughan, M. T., Wang, L., Long, H., Li, L., Dixon, N. A., & Durham, W. B.

1112 (2010). Precise stress measurements with white synchrotron x rays. *Review of Scientific*

1113 *Instruments*, 81. doi: <https://doi.org/10.1063/1.3263760>

1114 White, S. (1979). Contributions to mineralogy and petrology. *Contributions to Mineralogy*

1115 *and Petrology*, 70, pp. 193-202. doi: <https://doi.org/10.1007/BF00374448>