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#### **Key Points:**

- The weakest slip system does not change with Fe content, such that CPO development is similar for Ferich and Fe-poor olivine
- Seismic anisotropy due to the formation of CPOs in natural olivine aggregates can be modeled using CPOs obtained from iron-rich olivine
- Recrystallized grain size at a given normalized stress increases with increasing Fe content

#### **Supporting Information:**

Supporting Information S1

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# **Evolution of Microstructural Properties in Sheared Iron-Rich Olivine**

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**Abstract** Iron-rich olivine is mechanically weaker than olivine of mantle composition, ca.  $F_{0_{90}}$ , and thus is more amenable to study under a wide range of laboratory conditions. To investigate the effects of iron content on deformation-produced crystallographic preferred orientation (CPO) and grain size, we analyzed the microstructures of olivine samples with compositions of Fo<sub>70</sub>, Fo<sub>50</sub>, and Fo<sub>0</sub> that were deformed in torsion under either anhydrous or hydrous conditions at 300 MPa. Electron backscatter diffraction (EBSD) observations reveal a transition in CPO from D-type fabric, induced by dislocation glide on both the (010)[100] and the (001)[100] slip systems, at low strains, to A-type fabric, caused by dislocation glide on the (010)[100] slip system, at high strains for all of our samples, independent of iron content and hydrous/anhydrous conditions. A similar evolution of fabric with increasing strain is also reported to occur for Fo<sub>90</sub>. Radial seismic anisotropy increases with increasing strain, reaching a maximum value of ~1.15 at a shear strain of ~3.5 for each sample, demonstrating that the seismic anisotropy of naturally deformed olivine-rich rocks can be well approximated by that of iron-rich olivine. Based on EBSD observations, we derived a piezometer for which recrystallized grain size decreases inversely with stress to the  $\sim 1.2$  power. Also, recrystallized grain size increases with increasing iron content. Our experimental results contribute to understanding the microstructural evolution in the mantle of not only Earth but also Mars, where the iron content in olivine is higher.

# 1. Introduction

The rheological properties of olivine influence many geological processes on Earth and other planets in our solar system. Experimental studies demonstrate that the rheological behavior of olivine not only depends on thermomechanical parameters, such as stress, temperature (e.g., Carter & Ave'Lallemant, 1970; Chopra & Paterson, 1981), confining pressure (e.g., Borch & Green, 1989; Karato & Jung, 2003; Karato & Rubie, 1997), oxygen fugacity (e.g., Bai et al., 1991; Ricoult & Kohlstedt, 1985), melt content (e.g., Cooper & Kohlstedt, 1986; Hirth & Kohlstedt, 1995a, 1995b; Mei et al., 2002; Scott & Kohlstedt, 2006; Zimmerman & Kohlstedt, 2004) and water content (e.g., Karato et al., 1986; Mackwell et al., 1985; Mei & Kohlstedt, 2000a, 2000b; Tielke et al., 2017), but also on microstructural properties, such as the crystallographic preferred orientation (CPO) (e.g., Hansen et al., 2012a, 2012b) and grain size (e.g., Hansen et al., 2011; Hirth & Kohlstedt, 1995b; Karato et al., 1986). As an olivine single crystal exhibits strong viscous anisotropy (Bai et al., 1991; Durham & Goetze, 1977), the formation of a CPO in an olivine aggregate enhances plastic deformation in one crystallographic direction relative to the others (Hansen et al., 2012b, 2016). Thus, understanding the development of CPOs at different stresses, pressures and chemical conditions helps constrain microstructurally induced weakening/strengthening during deformation and is essential for the application of laboratory-derived flow laws to natural environments (Kohlstedt & Hansen, 2015). During plastic deformation, changes in grain size occur due to dynamic recrystallization and grain growth (Poirier, 1985, pp. 170-190). Experimental studies have determined the relationship between grain size and stress, that is, the grain-size piezometer, which is frequently used for estimating paleostresses in naturally deformed rocks (e.g., Kang & Jung, 2019; Kohlstedt & Weathers, 1980; Mercier et al., 1977; Park & Jung, 2017; Platt & Behr, 2011; Stipp & Tullis, 2003; Tokle et al., 2019; Warren & Hirth, 2006). As grain size plays an important role in flow laws for grain-size sensitive deformation mechanisms, such as diffusion creep and grain boundary sliding accommodated by dislocation



creep (disGBS), understanding the evolution of grain size during deformation is critical for interpreting the deformation history of naturally deformed rocks (de Bresser et al., 2001).

Olivine exists as a solid solution, from pure Mg olivine, forsterite (Fo), to pure Fe olivine, fayalite (Fa). In the mantle of Earth, the composition of olivine ranges from  $Fo_{87}$  (100 times the molar Fo/(Fo + Fa) ratio) to  $Fo_{96}$  with an average of  $\sim Fo_{90}$  (Deer et al., 1966, pp. 12). In the mantle of Mars, olivine is richer in iron with an estimated composition between  $Fo_{75}$  and  $Fo_{77}$  (Bertka & Fei, 1997; Morgan & Anders, 1979). In gabbroic intrusions on Earth, olivine is significantly richer in iron with a Fo content of  $Fo_{69}$  or smaller (Morse, 1979). In previous studies, the rheological behavior of iron-rich olivine has been investigated systematically with laboratory experiments, yielding flow laws that incorporate the dependence of strain rate not only on temperature and stress but also on iron content (Tasaka et al., 2015; Zhao et al., 2009, 2018). Although  $Fo_{50}$  has sometimes been used to study the CPO development of Fe-Mg olivines (Hansen et al., 2012a, 2014, 2016; Tasaka et al., 2016), the influence of iron content on the evolution of microstructures is explored here over a wide range of iron contents in an attempt to provide a unified description that accounts for the influence of iron as well as temperature, stress, and strain.

In this contribution, we performed torsional deformation experiments on samples that cover a range of iron content under both anhydrous and hydrous conditions. By mapping a full radial section of each sample, CPOs and grain sizes at different radii (i.e., different strains and stresses) were determined. The objective of this paper is to investigate the effect of iron content on the evolution of CPO and grain size, in order to provide further insights into the coupling between deformation and microstructure.

# 2. Methods

#### 2.1. Sample Preparation and Deformation Assembly

Samples for torsion experiments were sliced from deformed samples reported in Zhao et al. (2009) and (2018). Here we briefly describe the preparation procedures. Fayalite, Fo<sub>0</sub> (Fa<sub>100</sub>), powders were synthesized in a one-atmosphere furnace from powders of Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> in a molar ratio of 1:1.002 at temperatures close to the melting point of fayalite under a controlled oxygen buffer (Zhao et al., 2009). Powders of Fo<sub>70</sub> and Fo<sub>50</sub> were fabricated from mixtures of natural San Carlos olivine, Fo<sub>90</sub>, and synthetic fayalite. The mixed powders were ground to a particle size of <10  $\mu$ m and annealed at 1670 K in a one-atmosphere furnace for 16 h under a controlled oxygen buffer. To keep the powders anhydrous, powders were stored in a vacuum oven after the anneal until being cold-pressed. Anhydrous samples of Fo<sub>70</sub>, Fo<sub>50</sub> and Fo<sub>0</sub> as well as hydrous samples of Fo<sub>70</sub> and Fo<sub>50</sub> were then fabricated by first cold-pressing powders into nickel (Fo<sub>70</sub> and Fo<sub>50</sub>) or iron (Fo<sub>0</sub>) capsules and then hot-pressing the assemblies. During the cold-press of hydrous samples, two drops of water (~0.03 mL per drop) were added in each capsule. Details of hot-pressing and the axial deformation experiments can be found in Zhao et al. (2009) for anhydrous conditions and Zhao et al. (2018) for hydrous conditions.

The recovered samples were cut into cylinders with a diameter of  $2R \approx 9.4$  mm and a thickness of 4.5– 5.5 mm. Anhydrous samples were taken from samples PI1050 (Fo<sub>70</sub>), PI1040 (Fo<sub>50</sub>), and PI1189 (Fo<sub>0</sub>) reported in Zhao et al. (2009). Hydrous samples were taken from samples PI1141 (Fo<sub>70</sub>) and PI1065 (Fo<sub>50</sub>) reported in Zhao et al. (2018). Grain sizes and CPOs of samples before torsion deformation are presented in Table 1 and Figure 2, respectively. Although all of the samples have experienced some axial shortening (4.9%–16.9%), the crystallographic axes are oriented roughly randomly, with very weak clustering of crystallographic axes in the Fo<sub>0</sub> sample. The axial strains accumulated in these samples before torsion deformation are presented in Table 1. Cylinders of Fo<sub>70</sub> and Fo<sub>50</sub> were then placed into nickel capsules, while cylinders of Fo<sub>0</sub> were inserted into iron capsules, all with porous alumina spacers as end caps, as illustrated in Figure 1a. The rest of the deformation assembly was prepared as detailed in Qi et al. (2015).

#### 2.2. Deformation Experiments

Torsion experiments were performed in a gas-medium apparatus fitted with a torsion actuator (Paterson & Olgaard, 2000). Samples of Fo<sub>70</sub> and Fo<sub>50</sub> were deformed at a temperature of T = 1473 K ( $0.79T_m$  for Fo<sub>70</sub> and  $0.86T_m$  for Fo<sub>50</sub>) and a confining pressure of P = 300 MPa. To ensure water saturation in the samples during



Table I
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# Summary of Experiments and Experimental Conditions

Summary of Experiments and Experimental Conditions											
Composition	Sample #	Hydrous/ Anhydrous	<i>d</i> <sub>0</sub> (μm)	$\epsilon_{ax}^{a}$	T (K)	$T/T_m^{b}$	P (MPa)	<i>R</i> (mm)	$\gamma(R)^c$	$ au(R)^d$ (MPa)	$\dot{\gamma}(R)(\mathrm{s}^{-1})$
Fo <sub>70</sub> /Fa <sub>30</sub>	PT263	Anhydrous	33	0.049	1473	0.79	300	4.6	4.4	91	$2.6 \times 10^{-4}$
	PT278	Hydrous	40	0.107	1473	0.79	300	4.7	4.0	83	$4.7 \times 10^{-4}$
Fo <sub>50</sub> /Fa <sub>50</sub>	PT248	Anhydrous	42	0.169	1473	0.86	300	4.7	4.0	88	$2.8 \times 10^{-4}$
	PT303	Hydrous	47	0.119	1473	0.86	300	4.6	3.9	65	$2.3 \times 10^{-4}$
Fo <sub>0</sub> /Fa <sub>100</sub>	PT345	Anhydrous	41	0.071	1373	0.93	300	4.6	3.7	43	$2.3 \times 10^{-4}$

<sup>*a*</sup>  $\varepsilon_{ax}$  is the axial strain accumulated in axial compression before torsion experiment.

 $^{b}$   $T/T_{m}$  is homologous temperature.  $T_{m}$  is the solidus temperature at 1 atmosphere estimated based on Bowen and Schairer (1935). For Fo<sub>70</sub>,  $T_{m} \approx 1863$  K. For Fo<sub>50</sub>,  $T_{m} \approx 1713$  K. For Fo<sub>0</sub>,  $T_{m} \approx 1478$  K. The effect of water content on solidus temperatures is not considered.

 $^{c} \gamma(R)$  is shear strain at the outer radius.

 $d \tau(R)$  and  $\dot{\gamma}(R)$  are the outer-radius shear stress and shear strain rate measured at the end of each experiment.

the experiments under hydrous conditions, 0.05 mL of distilled water was added to each of the porous alumina discs. For experiments carried out under water-saturated, hydrous conditions, the water fugacity  $f_{\rm H_2O} \approx 300$  MPa. The Fo<sub>0</sub> sample was deformed at T = 1373 K ( $0.93T_m$ ) and P = 300 MPa. All samples were deformed at one or more constant strain rates increments to an outer-radius shear strain of  $\gamma(R) = 4.1 \pm 0.4$ . Due to slip between sample and spacer or the condition of the apparatus, three samples (PT248, PT278, and PT303) were deformed in two or three sequential experimental runs to accumulate the target strain; samples were removed from the deformation apparatus and rejacketed between runs. Each of the three samples was identified by the number of the final run. For samples deformed at multiple strain rates (PT278 and PT303), the stress associated with the final microstructure was averaged from the last strain-rate increment. After achieving the target strain, each sample was cooled at ~2 K/s to 1300 K (1200 K for the Fo<sub>0</sub> sample) under the same torque as imposed at the end of the deformation experiment to preserve the deformation-produced microstructure and then cooled to room temperature with no torque applied.

#### 2.3. Analysis of Mechanical Data

Rotation angle and torque were recorded as a function of time during each experiment to determine shear stress,  $\tau$ , shear strain,  $\gamma$ , and shear strain rate,  $\dot{\gamma}$ . To determine  $\tau$ , corrections to the measured torque were made to account for the strength of the Fe jacket and Ni or Fe canister.  $\tau$  was then calculated from the corrected torque, *M*, using the relationship (Paterson & Olgaard, 2000)



**Figure 1.** (a) Schematic drawing of the sample assembly for torsion experiments. (b) Drawing of the radial section. The whole radial section (yellow) was mapped with electron-backscatter diffraction(EBSD) at coarse step sizes, while a stripe extending from the axial center to the outer edge (orange) was mapped with EBSD at a fine step size. (c) A photo of the cut and polished radial section of a sample (PT278).

$$\tau = \tau(R) = \frac{\left(3 + \frac{1}{n}\right)M}{2\pi R^3},\tag{1}$$

where *n* is the stress exponent in a power-law flow law such as those used later in this paper. The value of *n* is characteristic of the deformation mechanism. Values of n = 3.9 and n = 3.7 were used, respectively, for iron-rich olivine deformed by disGBS under anhydrous conditions (Zhao et al., 2009) and by dislocation creep under hydrous conditions (Zhao et al., 2018). The rotation angle  $\theta$  was converted to shear strain by taking into account the sample dimensions:

$$\gamma = \gamma(R) = \frac{\theta R}{h},\tag{2}$$

where *h* is the sample thickness. The rotation angle was also measured from the strain marker on the sample jacket. For all samples except PT248 (Fo<sub>50</sub> anhydrous), the recorded value of  $\theta$  corresponded well with the measured value. For PT248, in which slip between sample and spacer





**Figure 2.** Pole figure illustrating distributions of orientations of [100], [010], and [001] axes. All pole figures are lower-hemisphere projection based on all orientation data of the mapped area. The shortening direction is vertical in the previous triaxial compressive creep experiments reported in Zhao et al. (2009) and (2018). The contours on each stereonet are universally colored by their multiples of uniform distribution (MUD), which is illustrated in the color bar. J- and M-indexes are presented on the left of each group of pole figures.

occurred, the measured value of  $\theta$  was used to calculate the strain and strain rate. Summary data are presented in Table 1.

To compare with mechanical data from axial compression experiments, the von Mises equivalent stress,  $\sigma_{eq}$ , and the equivalent strain rate,  $\dot{\varepsilon}_{eq}$ , were calculated from  $\tau$  and  $\dot{\gamma}$  using the relationships

$$\sigma_{eq} = \sqrt{3}\tau \text{ and } \dot{\varepsilon}_{eq} = \frac{\dot{\gamma}}{\sqrt{3}}.$$
 (3)

Within a torsion sample, shear strain rate increases linearly with increasing radius, *r*, that is,

$$\dot{\gamma}(r) / \dot{\gamma}(R) = \frac{r}{R}.$$
(4)

We applied published flow laws to determine the stress at radius *r*. Under anhydrous (dry) conditions, our samples deformed by disGBS described by the following flow law (Zhao et al., 2009):

$$\dot{\varepsilon} = \dot{\varepsilon}_{gbs}^{0} \left(\frac{\sigma}{\mu}\right)^{3.9} \left(\frac{b}{d}\right)^{0.7} \left(\frac{p_{O_2}}{p_{O_2}^{ref}}\right)^{0.2} X_{Fa}^{1.1} \exp\left(-\frac{Q_d + \alpha_d X_{Fa}}{RT}\right).$$
(5)

In Equation 5,  $\dot{\varepsilon}_{gbs}^0 = 5.7 \times 10^{24} \text{ s}^{-1}$  is a material dependent parameter,  $\mu$  is the shear modulus, *b* is the Burgers vector of length 4.75 × 10<sup>-4</sup> µm, *P*<sub>O2</sub> is oxygen partial pressure with  $p_{O2}^{ref} = 1$  Pa,  $Q_d = 435$  kJ/mol is the activation energy for creep,  $X_{Fa}$  is the mole fraction of fayalite,  $\alpha_d = -50$  kJ/mol reflects the dependence of activation energy on fayalite content, and R is the gas constant. In the original reporting of this flow law, a grain-size dependence of  $(1/d)^2$  was assumed; the dependence used here,  $(1/d)^{0.7}$ , was determined in subsequent studies (Hansen et al., 2011, 2012a). The dependence of strain rate on  $P_{O2}$  was obtained from the work of Keefner et al. (2011). Also, in the original reporting of this flow law,  $\mu$  and *b* were not included in the non-linear least-squares fit of the data even though

they were reported in the flow law to non-dimensionalize the stress and grain size terms. The values for the parameters reported here correct these errors. Under hydrous (wet) conditions, our samples deformed by dislocation creep described by the following flow law (Zhao et al., 2018):

$$\dot{\varepsilon} = \dot{\varepsilon}_{disl}^{0} \left(\frac{\sigma}{\mu}\right)^{3.7} \left(\frac{f_{\rm H_{2O}}}{f_{\rm H_{2O}}^{\rm ref}}\right)^{5/4} X_{\rm Fa}^{0.2} \exp\left(-\frac{Q_w + \alpha_w X_{\rm Fa}}{RT}\right). \tag{6}$$

In Equation 6,  $\dot{\varepsilon}_{disl}^0 = 1.5 \times 10^{20} \text{ s}^{-1}$ ,  $f_{\text{H}_{2}\text{O}}$  is water fugacity with  $f_{\text{H}_{2}\text{O}}^{\text{ref}} = 1$  MPa,  $Q_w = 525$  kJ/mol, and  $\alpha_w = -100$  kJ/mol. In the original reporting of this flow law,  $\mu$  was not included in the non-linear least squares fit of the data, even though it was included in the flow law to non-dimensionalize the stress term. The values for the parameters reported here correct this error.

Note that these two flow laws in Equations 5 and 6 are determined at strains <0.2, and therefore, cannot be directly applied to samples deformed to larger strains. Hansen et al. (2012a) reported that the stress and grain-size exponents in the flow law do not change with increasing strain, and strain weakening can be explained by the combination of grain-size reduction and CPO development. The weakening induced by CPO development can be quantified by a factor describing the degree of geometric softening, *F*, which is 1 for an isotropic polycrystalline materials and ranges between 1 and 0 depending on the strength of the dominant slip system relative to the isotropic case. Based on Equation 12 in Hansen et al. (2012a), *F* can be related to strain as



$$F = F_{ss} + (1 - F_{ss}) \exp\left(-\frac{\varepsilon}{\varepsilon_c}\right),\tag{7}$$

where the value of *F* at steady state,  $F_{ss}$ , is estimated to be 0.72 for (010)[100] slip system, and the critical strain for CPO development,  $\varepsilon_c$ , is estimated to be 1 for Fo<sub>50</sub>. Then the stress at a higher strain,  $\sigma_{hs}$ , can be calculated from the low-strain stress,  $\sigma_{ls}$ , derived from the flow law using the relationship

$$\sigma_{hs} = F \times \sigma_{ls}.$$
(8)

Thus, for the samples in our study, we can calculate the stress at a radius, r, from the measured values of strain rate and grain size (see next subsection), based on Equations 5–8.

#### 2.4. Analysis of Microstructure

After each deformation experiment, a radial section through the axial center of the sample was prepared for analyzing the crystallographic orientations; the process is illustrated in Figure 1. The radial section was polished down to a diamond grid size of  $0.5 \,\mu\text{m}$ , followed by a final step with 40-nm colloidal silica. Analyses of radial sections of samples deformed in torsion allow for obtaining microstructural data at different radii, that is, at different strains, strain rates and stresses. However, some microstructural features, such as tilt walls, cannot be observed, because most of them do not intersect the polished surface.

To obtain crystallographic data, orientation maps with different step sizes were collected for all sections using a scanning electron microscope equipped with an electron-backscatter diffraction (EBSD) system. For EBSD analyses, step sizes of  $4-7 \mu m$  were used to map the whole radial section, while a finer step size of  $1.5 \mu m$  was used to map smaller areas on the radial section. Information of the orientation maps are summarized in Table 2. Raw orientation data were processed using HKL CHANNEL5 software, including removal of single mis-indexed points, assigning the average orientation of neighboring pixels to unindexed points, and removal of systematic mis-indexed points, as outlined, for example, in Hansen et al. (2011). Grains were constructed from processed orientation data using the MTEX toolbox (Bachmann et al., 2011). Grain boundaries were drawn where the misorientation between neighboring pixels exceeded  $10^{\circ}$ . No extrapolation of orientation data was applied in MTEX since the data were already processed by the HKL CHANNEL5 software.

To distinguish recrystallized grains from relict grains, grains were separated based on their degrees of intracrystalline distortion. For each grain, grain orientation spread (GOS) was used as a measurement of intracrystalline distortion. The threshold value of GOS that separates recrystallized and relict grains was determined by the method described in Cross et al. (2017). The threshold value was generally between 2.0 and 2.5°. Grains with values of GOS below the threshold value were considered as recrystallized.

Grain size was determined by applying a scaling factor of  $4/\pi$  to the equivalent diameter of a circle with the area of each recrystallized grain in cross section. In the analysis of the average grain size for a map, grains containing less than 4 pixels or lying on the edge of the map were excluded. We calculated both the arithmetic mean and the geometric mean for the average grain size in selected areas.

For easy comparison with previous studies, orientation data were rotated into a kinematic reference frame, that is, the plane containing the shear direction and shear plane normal. Orientation distributions were generated from all data points from at least 500 individual grains with a half-width angle of 10° using the MTEX toolbox in MATLAB (Bachmann et al., 2010; Mainprice et al., 2015). To quantify the strength of the CPOs, both the J-index (Bunge, 1982) and the M-index (Skemer et al., 2005) were used.

To quantify the shape of the distribution of crystallographic axes in pole figures, an eigenvalue analysis following Woodcock (1977) was employed. Normalized eigenvalues,  $S_1$ ,  $S_2$ , and  $S_3$ , calculated from the orientation tensor, obey the relationships

$$S_1 + S_2 + S_3 = 1 \tag{9}$$



Table 2

Summary of Results From EBSD Analyses

Sample							
#	Data for C	РО		Data for microstructure			
	Step size (µm)	# Indexed	# Grains	Step size (µm)	# Indexed	# Grains	
PT248	7	629309	60316	1.5	617338	8191	
PT263	5	1226602	176934	1.5	597728	16366	
PT278	8	721695	92970	1.5	590263	8266	
PT303	8	524532	31925	1.5	5083240	18916	
PT345	4	1758097	40764	1.5	5273959	19026	

EBSD, electron-backscatter diffraction; CPO, crystallographic preferred orientation.

and

$$S_1 \ge S_2 \ge S_3. \tag{10}$$

The shape factor of the distribution of axes, *K*, is then defined by

$$K = \frac{\ln(S_1 / S_2)}{\ln(S_2 / S_3)}.$$
(11)

For the orientations of a specific group of crystallographic axes, K > 1 indicates that the distribution of axes forms a cluster, and K < 1 indicates that the distribution of axes forms a girdle.

#### 2.5. Calculation of Seismic Properties

To calculate the seismic anisotropy of an aggregate, stiffness tensors for individual grains obtained at room pressure and temperature for olivine (Abramson et al., 1997) were averaged based on the measured distribution of crystallographic orientations using the MTEX toolbox for MAT-

LAB (Mainprice et al., 2011). The Hill average was used, as it yields the best approximation of the magnitudes of individual elastic constants (Zhong et al., 2014). Magnitudes of seismic anisotropy were then calculated using the average stiffness tensor, following the procedures described by Hansen et al. (2014) and Skemer and Hansen (2016). In Voigt notation, the stiffness tensor is described by the 36 components in  $C_{ij}$ , where *i* and *j* range from 1 to 6 (e.g., Karato, 2008, pp. 53). In our reference frame, subscripts 1, 2, and 3 correspond to the shear direction, the normal to the shear plane, and the vorticity direction, respectively. We calculated the radial anisotropy,  $(V_{SH}/V_{SV})^2$ , assuming the shear plane is horizontal.  $V_{SH}$  and  $V_{SV}$  are the velocities of *S*-waves polarized parallel to the shear plane and the normal to the shear plane, respectively. The radial anisotropy can be calculated using components of the average stiffness tensor through the relationship (Montagner & Nataf, 1986)

$$\left(\frac{V_{SH}}{V_{SV}}\right)^2 = \frac{\frac{1}{8}(C_{11} + C_{22}) - \frac{1}{4}C_{12} + \frac{1}{2}C_{66}}{\frac{1}{2}(C_{44} + C_{55})}.$$
(12)

# 2.6. FTIR Analysis

Hydroxyl contents of the olivine aggregates were determined from Fourier transform infrared (FTIR) analyses of doubly polished slices  $\sim$ 0.3 mm in thickness and  $\sim$ 5 mm in length and width. FTIR spectra were collected on two deformed hydrous samples. Details of the analysis can be found in Zhao et al. (2018).

# 3. Results

#### 3.1. Mechanical Data

The environmental conditions along with the outer radius shear strain, shear stress, and shear strain rate at the end of each experiment are summarized in Table 1. Graphs of outer-radius shear stress plotted against outer-radius shear strain (hereafter "stress" and "strain" for brevity in this subsection) are presented in Figure 3. Stresses were calculated using the reported flow laws, as described in Section 2.3. All curves show a rapid rise in stress to a peak stress at a relatively small strain of ~0.3. In experiments PT263 (Fo<sub>70</sub>, anhydrous,  $T = 0.79T_m$ ), PT278 (Fo<sub>70</sub>, hydrous,  $T = 0.79T_m$ ) and PT248 (Fo<sub>50</sub>, anhydrous,  $T = 0.86T_m$ ), the peak stress was followed by a steep drop in stress with increasing strain. In PT263, stress increased rapidly to a second peak after the steep drop, due to an imposed increase in strain rate; after this second peak, stress gradually evolved to a plateau at  $\gamma > 3$ . In PT278, after the steep drop, stress increased and then decreased with increasing strain due to slip between the sample and a spacer, which led to the end of the first part of this experiment. The sample was then taken out of the deformation apparatus and re-jacketed with new spacers and pistons. In the second part, stress increased in steps that correlate with stepped increases in strain rate. In PT248, the steep drop in stress was due to slip between the sample and one of the spacers,



which led to the end of the first part of the experiment. The second part also ended with a rapid decrease in stress. In the third part, stress slowly evolved within a range of  $\pm 10$  MPa with increasing strain. In PT303 (Fo<sub>50</sub>, hydrous,  $T = 0.86T_m$ ), stress decreased gradually with increasing strain and plateaued at  $\gamma > 2.5$ . In PT345 (Fo<sub>0</sub>, anhydrous,  $T = 0.93T_m$ ), stress remained roughly constant after the peak stress at  $\gamma < 1.5$ , gradually dropped at  $1.5 < \gamma < 2.5$ , and slightly increased at  $\gamma > 2.5$ . Although strain-rate steps were made in three of the experiments, the mechanical data were not further investigated, as the mechanical behaviors of these samples have been investigated and reported in Zhao et al. (2009),and (2018). Flow laws of iron-rich olivine under anhydrous and hydrous conditions have been determined in previous studies (Tasaka et al., 2015; Zhao et al., 2009, 2018). The mechanical data are compared with the flow laws in Figure S1. Note that the data represent the strain rate at the outer radius and the stress calculated based on the torque over the full radius, such that, the data points should only be compared qualitatively to the flow laws.

# 3.2. FTIR Results

The FTIR spectra in Figure 4 obtained from two hydrous samples,  $Fo_{70}$  (PT278) and  $Fo_{50}$  (PT303), after deformation demonstrate that these samples were indeed sheared under hydrous conditions. The broadband extending from 3,000 to 3,700 cm<sup>-1</sup> arises from water trapped in pores. Note that this approach yields the "free water" not the structurally bound water/hydroxyl (OH). This source of water insures that deformation of these two samples occurred under water-saturated conditions (Karato et al., 1986). The observations for hydrous samples are in good agreement with those reported by Zhao et al. (2018) for samples of Fe-rich olivine deformed in triaxial compression and with those reported by Tasaka et al. (2016) for samples of Fe-rich olivine deformed in torsion. Likewise, the OH concentration in samples deformed under anhydrous conditions was below the FTIR detection limit (Zhao et al., 2018).

#### 3.3. Crystallographic Preferred Orientations

In this subsection, CPOs of samples with different iron contents deformed under either anhydrous or hydrous conditions are described. The results are presented as orientation maps for radial sections, plots of CPO strength versus radius, and distributions of orientations at different radii in Figures 5–7.

#### 3.3.1. Strain series

In all samples, near the axial center, r = 0, the crystallographic axes are randomly distributed. With increasing radius, that is, with increasing strain, the crystallographic axes gradually concentrate into clusters. The strength of the CPO, characterized by the J- and M-indexes, increases roughly linearly with increasing radius. Converting radius to shear strain and averaging the indexes from regions at the same strain (symmetric with respect to the torsional axis), plots of fabric strength versus local shear strain, as illustrated in Figure 8a, reveal that M-index increases roughly linearly with increasing strain for  $\gamma < 3.5$  and then remains constant or decreases slightly for  $\gamma > 3.5$ .

For regions at  $r \ge 1$  mm in all samples, a weak secondary cluster of [100] axes occurs 60–70° to the primary [100]-axes cluster, synthetic to the shear direction; the only exception is the region at r = 4.2 mm in the hydrous Fo<sub>50</sub> sample (PT303). With increasing radius (strain), the primary [100]-axis cluster strengthens and the secondary [100]-axis cluster weakens, as characterized by their values of multiples of uniform distribution (MUD). At small radii, [010] and [001] axes form girdles subnormal to the shear direction. With increasing radius, [010] and [001] axes become less girdled and gradually concentrate into elongated clusters. The strengths of the [010]- and [001]-axis clusters also increase with increasing radius (strain), as characterized by the values of MUD. Near the outer radius of all samples, CPOs are characterized by a strong primary cluster of [100] axes aligning parallel to the shear direction and a cluster of [010] axes aligning subnormal to the shear plane, that is, the A-type fabric (Jung et al., 2006; Jung & Karato, 2001b). In all samples except for the anhydrous Fo<sub>50</sub> sample (PT248), a very weak cluster of [001] axes occurs, aligned 30°–60° from shear plane antithetic to shear direction. This cluster becomes weaker with increasing radius.

Converting radius to shear strain and averaging the shape factor, *K*, for the same strain, plots of fabric shape versus local shear strain, as illustrated in Figure 9, provide a more quantitative measurement of the evolution of fabric shape. The distributions of [100] axes form clusters at very small strains and become more





Figure 3. Stress-strain curves for all experimental runs. (a) PT263. (b) PT278. (c) PT248. (d) PT303. (e) PT345.

clustered as strain increases. The distributions of [010] axes form girdles at small strains, become more girdled as strain increases to ~1, and then become less girdled as strain increases further. The distributions of [001] axes evolve differently in different samples. In the anhydrous Fo<sub>70</sub> sample, the value of *K* starts above 1, decreases with increasing strain for  $\gamma < 2.5$ , and increases slightly with increasing strain for  $\gamma > 2.5$ , but remains below 1. In the hydrous Fo<sub>70</sub> sample, the value of *K* starts above 1 and decreases with increasing

 $\begin{array}{c} 2.5 \\ \hline \\ 2.0 \\ 1.5 \\ 0.0 \\ \hline \\ 0.5 \\ 4000 \\ 3800 \\ 3800 \\ 3600 \\ 3600 \\ 3400 \\ 3200 \\ 3200 \\ 3000 \\ \end{array}$ 

**Figure 4.** Absorption coefficient versus wavenumber from FTIR analyses of two hydrous samples. Orange is  $Fo_{70}$  (PT278). Green is  $Fo_{50}$  (PT303).

strain. In  $Fo_{50}$  samples, the value of *K* starts below 1 and slightly increases with increasing strain. In the  $Fo_0$  sample, the value of *K* varies between 0.9 and 3.

# 3.3.2. Iron-Content Series

In samples deformed under anhydrous conditions, at a given strain, the fabric in the  $Fo_{70}$  sample is the weakest and the fabrics in the  $Fo_{50}$  and  $Fo_0$  samples have similar strengths. In samples deformed under hydrous conditions, at similar strains, the fabric in the  $Fo_{70}$  sample is weaker than the fabric in the  $Fo_{50}$  sample. At similar strains, the [010]-axis distribution is more clustered in samples with higher iron content, as illustrated in Figure 9b. Note that samples with a higher iron content are also deformed at a higher homologous temperature.

To compare fabrics amongst samples, consider the fourth group of pole figures ( $\gamma = 2.9 - 3.5$ ) for each anhydrous sample. For the Fo<sub>70</sub> sample



(PT263, Figure 5a), [010] axes form a girdle subnormal to the shear direction. For the Fo<sub>50</sub> sample (PT248, Figure 6a), [010] axes form a diffuse cluster elongated subnormal to the shear direction. For the Fo<sub>0</sub> sample (PT345, Figure 7), [010] axes form a strong, round cluster normal to the shear plane, with a weak concentration of [010] axes lying in the shear plane subnormal to the shear direction. However, this correlation between the distribution of [010] axes and iron content is not observed in the distributions of [100] and [001] axes. At similar strains, the distribution of [100] axes in Fo<sub>50</sub> samples is the most clustered, and that in Fo<sub>0</sub> sample is least clustered (Figure 9a). At similar strains, the distribution of [001] axes in Fo<sub>50</sub> samples is the most girdled; while for  $\gamma > 2.5$ , those in Fo<sub>50</sub> and Fo<sub>70</sub> samples are similar in shape.

# 3.3.3. Water-Content Series

The two samples deformed under hydrous conditions exhibit the same CPO pattern as the samples deformed under anhydrous conditions. At the same strain, fabric strengths are similar in anhydrous and hydrous samples (Figure 8a). At the same strain, fabric shapes for all axes are also similar in anhydrous and hydrous samples (Figure 9).

# 3.4. Seismic Anisotropy

The evolution of radial seismic anisotropy with shear strain is presented in Figure 8b. In all samples, the value of radial anisotropy increases from ~1 to ~1.15 with increasing strain. In each case, the slope of the curve decreases with increasing strain with the curve becoming roughly flat as strain approaches its maximum value. For the same strain in the range of  $0.5 < \gamma < 3$ , the value of radial anisotropy in the Fo<sub>0</sub> sample is the largest, and values of radial anisotropy in Fo<sub>70</sub> samples are smallest. For the same iron content, the radial anisotropy of anhydrous and hydrous samples exhibits similar values and trends.

#### 3.5. Recrystallized Grain Sizes

Orientation maps and dynamically recrystallized grain-size distributions for representative areas near the axial center and close to the outer edge are presented in Figure 10, and grain sizes of dynamically recrystallized grains at different radii are presented in Figures 11a and 11b. In all samples, grain sizes are larger near the axial center than close to the outer edge, that is, grain size decreases with increasing radius. At the same radius, the grain size in samples with higher iron content is larger than that in samples with lower iron content, and the grain size in hydrous samples is larger than that in anhydrous samples with the same iron content. Subgrain boundaries and intragranular distortions are observed across the whole radius. In all selected regions, the distributions of grain size are skewed, with a peak at finer grain sizes and a tail extending to coarser grain sizes. The grain-size distributions at regions close to the outer edge are approximately log-normal, as demonstrated by the fitted curves in Figure 10. Comparatively, the grain-size distributions near the axial center are not as well fit by log-normal distributions (see supporting information Figure S2 for grain-size distributions on a logarithmic scale). The geometric mean is always smaller than the arithmetic mean and is closer to the peak of the grain-size distribution. The difference between the arithmetic mean and the geometric mean is smaller when the distribution of grain sizes is tighter (smaller standard deviation). To make a statistically reliable analysis, a minimum number of 433 grains should be measured (Lopez-Sanchez & Llana-Fúnez, 2015). Thus, regions with fewer than 433 grains, for example, Figures 10c and 10e, were not used for further analyses in the Discussion section.

# 4. Discussion

# 4.1. Slip Systems

For all samples, CPOs are characterized by a cluster of [100] axes parallel to the shear direction and girdles of [010] and [001] axes subnormal to the shear direction, that is, D-type fabrics, at lower strains, and a cluster of [100] axes parallel to the shear direction and a cluster of [010] axes subnormal to the shear plane, that is, A-type fabrics, at higher strains. Moreover, during our experiments, stresses were always between 50 and 100 MPa, well below the 350–400 MPa reported for the stress transition between the A- and D-type fabrics



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**Figure 5.** CPOs of  $Fo_{70}$  samples deformed under (a) anhydrous (PT263) and (b) hydrous (PT278) conditions. In each panel, an orientation map for the radial section of the sample is presented in the top left, a plot of CPO strength versus radius is presented in the bottom left, and distributions of orientations at different radii are presented in the right. Each orientation map is colored by the crystallographic orientation normal to the radial section, that is, parallel to the shear direction, according to the color map. Shear strain and shear strain rate across the radius are plotted on the map using green and red dashed lines, respectively. Below the map, CPO strength characterized by J- (blue squares) and M-indexes (orange circles) is plotted with radius, with horizontal error bars illustrating the range of radius represented by each data point. Radius r = 0 corresponds to the location of the torsional axis, which is the center of the map. The *x*-axis of the plot shares the same scale as the map. For each selected radius, a pole figure representing distributions of [100], [010], and [001] axes is contoured based on all orientation data in a 0.4-mm wide area around this radius, as illustrated by the shaded stripe on the map. Pole figures are lower-hemisphere projections. The orientation data for the pole figures are rotated 90°, so that the sense of shear for the pole figures is top to the right. The contours on each stereonet are universally colored by their multiples of uniform distribution (MUD), which is illustrated in the color bar. The maximum values of MUD are listed for each stereonet. CPO, crystallographic preferred orientation.





**Figure 6.** CPOs of  $Fo_{50}$  samples deformed under (a) anhydrous (PT248) and (b) hydrous (PT303) conditions. The organization of this figure is the same as Figure 5. Each orientation map is colored by the crystallographic orientation normal to the radial section, that is, parallel to the shear direction, according to the color map. Shear strain and shear strain rate across the radius are plotted on the map using green and red dashed lines, respectively. Below the map, CPO strength characterized by J- (blue squares) and M-indexes (orange circles) is plotted with radius, with horizontal error bars illustrating the range of radius represented by each data point. Radius r = 0 corresponds to the location of the torsional axis, which is the center of the map. The *x*-axis of the plot shares the same scale as the map. For each selected radius, a pole figure representing distributions of orientations of [100], [010], and [001] axes is contoured based on all orientation data in a 0.4-mm wide area around this radius, as illustrated by the shaded stripe on the map. Pole figures are lower-hemisphere projections. The orientation data for the pole figures are rotated 90°, so that the sense of shear for the pole figures is top to the right. The contours on each stereonet are universally colored by their MUD, which is illustrated in the color bar. The maximum values of MUD are listed for each stereonet. CPO, crystallographic preferred orientation.





**Figure 7.** CPOs of the  $Fo_0$  sample (PT345) deformed under anhydrous condition. In the top left, an orientation map is colored by the crystallographic orientation normal to the radial section, that is, parallel to the shear direction, according to the color map. Shear strain and shear strain rate across the radius are plotted on the map using green and red dashed lines, respectively. Below the map, CPO strength characterized by J- (blue squares) and M-indexes (orange circles) is plotted with radius, with horizontal error bars illustrating the range of radius represented by each data point. Radius r = 0 corresponds to the location of the torsional axis, which is the center of the map. The *x*-axis of the plot shares the same scale as the map. In the right, for each selected radius, a pole figure representing distributions of orientations of [100], [010] and [001] axes is contoured based on all orientation data in a 0.4-mm wide area around this radius, as illustrated by the shaded stripe on the map. Pole figures are lower-hemisphere projections. The orientation data for the pole figures are rotated 90°, so that the sense of shear for the pole figures is top to the right. The contours on each stereonet are universally colored by their MUD, which is illustrated in the color bar. The maximum values of MUD are listed for each stereonet. CPO, crystallographic preferred orientation.

at our experimental temperature and pressure (Karato et al., 2008); thus, the change from D-type to A-type fabric is associated with increasing strain. These observations are consistent with those previously reported for anhydrous  $Fo_{90}$  and  $Fo_{50}$  samples (Hansen et al., 2014). The D-type fabric at low strains is caused by dislocation glide on the {0kl}[100] slip systems (Carter & Ave'Lallemant, 1970), but more specifically, on the (010)[100] and the (001)[100] slip systems (Hansen et al., 2014), as the two slip systems are similar in strength (Bai et al., 1991; Durham et al., 1977; Tielke et al., 2016) and the major dislocations in the {0kl}



**Figure 8.** Evolution of fabric strength and radial anisotropy with local shear strain. Orange is  $Fo_{70}$ . Green is  $Fo_{50}$ . Blue is  $Fo_0$ . Solid circles and lines are results for anhydrous samples. Open circles and dashed lines are results for hydrous samples. Horizontal error bars are the range of strain represented by each data point. (a) Plot of M-index versus shear strain. (b) Plot of magnitude of radial anisotropy versus shear strain. Radial anisotropy is calculated assuming the shear plane is horizontal.





**Figure 9.** Evolution of fabric shape with local shear strain. Orange is Fo<sub>70</sub>. Green is Fo<sub>50</sub>. Blue is Fo<sub>0</sub>. Solid lines are results for anhydrous samples. Dashed line are results for hydrous samples. Horizontal error bars indicate the range of strain represented by each data point. (a) Plot of shape factor for the distribution of [100] axes versus strain. (b) Plot of shape factor for the distribution of [010] axes versus strain. The gray horizontal line marks K = 1. (c) Plot of shape factor for the distribution of [001] axes versus strain. In panels (b) and (c), the data point at very small strains was removed, as the distributions of axes are quite random such that values of *K* are also random.

[100] family (Hansen et al., 2014; Wallis et al., 2019). As strain increases, the A-type fabric dominates, indicating that (010)[100] is the weakest slip system. The observed fabric evolution with increasing strain, therefore, is primarily caused by the transition from a combination of the (010)[100] and (001)[100] slip systems to dominantly the (010)[100] slip system.

In addition to the primary cluster of [100] axes aligning parallel to the shear direction, a weak secondary cluster of [100] axes occurs at small radii/low strains. The occurrence of this secondary [100]-axis cluster has been reported for Fo<sub>90</sub> samples deformed under anhydrous conditions that were characterized by an A-type fabric (Zhang et al., 2000) and accepted as a minor feature of the A-type fabric (Karato et al., 2008). This secondary [100]-axis cluster is sub-perpendicular to the maximum principal compressive stress. Zhang et al. (2000) argued that this secondary cluster is stress controlled. New grains in low-Schmid-factor orientations will have the lowest dislocation densities (Karato, 1987). Because the active systems are mainly (010)[100] and (001)[100], grains with [100] orienting at 90° to the maximum principal stress represent the lowest-Schmid-factor orientations. Thus, grains with this crystallographic orientation grow and consumes other grains via grain boundary migration (Zhang et al., 2000). Because grain boundary migration does not contribute to strain, as strain increases, grain boundary migration contributes less to CPO formation, subgrain rotation contributes more, and the secondary [100]-axis cluster disappears.

In the review of the CPOs of olivine samples naturally and experimentally deformed under anhydrous conditions, Skemer and Hansen (2016) reported that the secondary cluster of [100] axes moves toward the primary cluster of [100] axes with increasing strain. For example, in samples experimentally deformed in direct shear, the angle between secondary and primary clusters of [100] axes decreases from ~45° at  $\gamma < 0.2$  to 0° at  $\gamma > 1.5$  (Zhang & Karato, 1995). However, such a decrease was not observed in our samples. Rather, in our samples, with increasing strain, the weak secondary cluster of [100] axes roughly remains at 60-70° to the primary cluster of [100] axes through  $\gamma = 1-4$ . Similarly, in samples experimentally deformed in torsion by Skemer et al. (2011), the angle between the two clusters of [100] axes only decreases slightly from mid-50° to mid-40° with strain increasing from  $\gamma \approx 1.2$  to 3.5. The difference in the deformation kinematics, direct shear versus torsion, is likely the cause for the difference in the change in orientation of the secondary cluster, as direct shear is a combination of compression and simple shear.

The observation that the A-type fabric dominates at high strains in all samples suggests that (010)[100] is the weakest slip system over a wide range of iron content at our experimental conditions, from San Carlos olivine, Fo<sub>90</sub>, to fayalite, Fo<sub>0</sub>. No correlation was found between the evolution of fabric shape (Figure 9) and iron content, suggesting that varia-

tions in iron content do not change the relative strengths of primary slip systems in olivine, consistent with results from deformation experiments on single crystals as reported by Kohlstedt and Ricoult (1984).

Before looking into the CPOs of samples deformed under hydrous conditions, we estimate the OH concentration in these samples. Under water-saturated conditions, as in our experiments, at 300 MPa and 1200°C the OH concentration in single crystals of San Carlos olivine (ca.  $Fo_{90}$ ) is ~300 ppm H/Si (Bai & Kohlstedt, 1992; Tasaka et al., 2016; Zhao et al., 2004) using the Paterson (1982) OH-infrared calibration. Based on the results of a study by Withers et al. (2011), water solubility in  $Fo_{70}$  and  $Fo_{50}$  is ~1.6 times and ~2.2 times that in  $Fo_{90}$ , respectively. That is, under water-saturated conditions, the OH concentration in our samples





**Figure 10.** Orientation maps and grain-size distribution histograms for selected regions in all samples. Regions near the axial center are in the left column, and regions close to the outer edge are in the right column. In each panel, an orientation map colored by the crystallographic orientation parallel to the shear direction is on the left, and a histogram of the distribution of grain size is on the right. In the map, grain boundaries, characterized by a misorientation of  $<10^{\circ}$  and  $\geq 2^{\circ}$ , are gray. Unindexed spots are white. Color maps are the same as in Figures 5–7. Maps are subareas of the whole mapped area (except for PT263). The whole mapped area was used to calculate grain size. In the histogram, the red line marks the arithmetic mean and the orange line marks the geometric mean. The blue curve is a log-normal fit of the grain-size distribution. The number of grains used to calculate the means is also provided in each histogram. Grain size distributions are plotted in logarithm scale in Figure S2.





**Figure 11.** Evolution of grain size with (a) and (b) radius and (c) and (d) normalized stress. In the left column, average grain sizes are calculated as arithmetic means; in the right column, average grain sizes are calculated as geometric means. Orange is Fo<sub>70</sub>. Green is Fo<sub>50</sub>. Blue is Fo<sub>0</sub>. Solid circles and lines are results for anhydrous samples. Open circles and dashed lines are results for hydrous samples. In (c) and (d), normalized equivalent stresses are calculated using the flow laws in Equations 5 and 6.  $p_{O_2} = 10^{-3}$  Pa is used in Equation 5.  $f_{H_2O} = 300$  MPa is used in Equation 6. Solid and dashed lines are the linear fits for data points that are (1) taken at r > 1 mm, and (2) averaged based on more than 400 grains. Slopes are presented in Table 3.

are  $\sim$ 500 ppm H/Si for Fo<sub>70</sub> and  $\sim$ 700 ppm H/Si for Fo<sub>50</sub>. We note that OH solubilities cannot be calculated from our FTIR spectra, because the broad band associated with molecular water swamps the signal from OH groups.

The CPOs of our samples deformed under hydrous conditions exhibit both similarities to and differences from the CPOs of hydrous Fo<sub>50</sub> samples reported by Tasaka et al. (2016). CPOs at  $\gamma \leq 2$  in our hydrous Fo<sub>70</sub> (PT278) and Fo<sub>50</sub> (PT303) samples are very similar to the CPOs in samples deformed to  $\gamma = 2.4$  (PT954) and  $\gamma = 3.0$  (PT939) in Tasaka et al. (2016). Note that the difference in the orientations of the [001]-axis girdles between this study and Tasaka et al. (2016) is due to different projections of crystallographic orientations (see Wallis et al., 2019, for correction of the projection). All of our pole figures are plotted using lower-hemisphere projection, as was done as in many previous studies (e.g., Hansen et al., 2014). Because the two slip systems (001)[100] and (100)[001] are similar in strength at T = 1423-1473 K for Fo<sub>90</sub> deformed under anhydrous (Durham & Goetze, 1977; Tielke et al., 2016) and hydrous conditions (Wallis et al., 2019) and because iron content does not affect the relative strengths of the primary slip systems (an assumption in Tasaka et al. (2016), but an interpretation in this study), Tasaka et al. (2016) concluded that these CPOs resulted from slip on a combination of the (010)[100], (001)[100], and (100)[001] slip systems, in which the operation of the (010)[100] and (001)[100] slip systems leads to the D-type fabric and operation of the (100)[001] slip system leads to the weak [001]-axes cluster sub-parallel to shear direction. This explanation applies to our hydrous samples at lower strains ( $\gamma \leq 2$ ). With increasing strain, in our samples, the (010)[100] slip system contributed more than the (001)[100] slip system to the CPO, leading to an A-type fabric at higher strains  $(\gamma \gtrsim 3)$ ; however, in samples of Tasaka et al. (2016), the (001)[100] and (100)[001] slip systems dominated, leading to bimodal distributions of [100] and [001] axes. In our samples, although the (010)[100], (001) [100], and (100)[001] slip systems were activated, the (010)[100] slip system remained the weakest, thus dominating CPO development at high strains. Considering the estimated water contents in our samples, these results suggest the A-type fabric dominates over a broader range of water content in iron-rich olivines than in  $Fo_{90}$  (Jung et al., 2006).







**Figure 12.** Comparison with previously reported piezometric relationships for anhydrous olivines. The red shaded region represents the data for anhydrous  $Fo_{90}$  deformed in torsion at 1523 K (Meyers, 2020). The light green line marks the piezometer for anhydrous  $Fo_{50}$  reported by Hansen et al. (2012a) with a slope of -1.2. The solid red line marks the piezometer for anhydrous  $Fo_{90}$  ( $\mu = 63.5$  GPa) reported by Van der Wal et al. (1993) and Jung and Karato (2001a) with a slope of -1.33 (data of Karato et al. (1980) are included in Van der Wal et al. (1993)).

The CPOs of our samples deformed under hydrous conditions differ from the CPOs of Fo<sub>90</sub> samples deformed under hydrous condition in previous studies that were carried out at higher confining pressures,  $P \ge 500$  MPa, and thus at higher water fugacity,  $f_{\rm H_2O} > 600$  MPa, and higher OH concentrations,  $C_{\rm OH} > 1000$  ppm H/Si (Jung et al., 2006; Jung & Karato, 2001b; Katayama et al., 2004). Jung et al. (2006) reported that, at low differential stresses (similar to those used in our study), the easiest slip system changes from (010)[100] at low OH content, to (001)[100] at moderate OH content, and then to (100)[001] at high OH content. The water content in our samples, independent of iron content, must lie below that required to cause a change in the dominant slip system from (010)[100] to (001)[100].

# 4.2. Seismic Anisotropy

The value of radial anisotropy increases from  $\sim 1$  at  $\gamma = 0$  to  $\sim 1.15$  at higher strains in all samples. Although the values of radial anisotropy in samples with different iron contents increase at different rates with increasing strain, all of the values converge to the same maximum at  $\gamma > 3.5$ . Similarly, previous data for radial anisotropy of Fo<sub>90</sub> and Fo<sub>50</sub> also reached a maximum of  $\sim 1.15$  at large strains,  $\gamma \approx 5$  (Hansen et al., 2014). Therefore, olivines with different iron contents exhibit the same radial anisotropy at a shear strain as low as 3.5, demonstrating that seismic anisotropy in natural olivine aggregates can be well-modeled using CPO data obtained from laboratory experiments on iron-rich olivine.

#### 4.3. Recrystallized Grain Size and Stress

The relationship between dynamically recrystallized grain size,  $d_{rex}$ , and stress provides an important tool for estimating the stress experienced by a naturally deformed rock. This relationship can be analyzed in terms of either the piezometer (Twiss, 1977) or the wattmeter (Austin & Evans, 2007) formalism. Here, because of the lack of information on grain growth kinetics as a function of iron content in olivine, information that is critical for the wattmeter, we focus on the piezometer in the form (Hirth & Kohlstedt, 2015)

$$d_{\rm rex} = \frac{2\gamma_{\rm gb}}{\alpha\mu A} \left(\frac{\sigma}{\mu}\right)^{-q},\tag{13}$$

where  $\gamma_{gb}$  is the average grain boundary energy and  $\alpha$  is a geometrical constant associated with the interactions between dislocations. The material-dependent parameters *A* and *q* enter through the empirical relationship between dislocation density,  $\rho$ , and stress (Twiss, 1977)



$$\rho b^2 = A \left(\frac{\sigma}{\mu}\right)^q. \tag{14}$$

For our samples, recrystallized grain sizes at different radii were measured from the orientation maps in Figures 5–7. Stresses at different radii were calculated using Equations 5–8. Based on Figure 8, M-index, an indicator for CPO development, increases faster with increasing strain in Fo<sub>50</sub> than in Fo<sub>70</sub> and Fo<sub>0</sub>, such that, the critical strain for CPO development, estimated to be 1 for Fo<sub>50</sub> (Hansen et al., 2012a), should be larger for Fo<sub>70</sub> and Fo<sub>0</sub>. Consider the M-index at  $\varepsilon_c = 1$  for Fo<sub>50</sub>, and to achieve a similar M-index,  $\varepsilon_c$  is estimated to be 1.5 for Fo<sub>70</sub> and 1.1 for Fo<sub>0</sub>. Therefore, a piezometric relationship can be determined for each sample.

It is worth noting that this analysis is based on two assumptions. First, for each sample, the radial stress distribution calculated from the published flow law determined for that sample (Zhao et al., 2009, 2018) is accurate. Based on microstructural observations, dynamic recrystallization by subgrain rotation widely occurred, demonstrating that dislocations played a major role in deformation of our samples. This observation is in good agreement with our interpretation based on the mechanical data that samples were deformed in disGBS for anhydrous samples and dislocation creep for hydrous samples. Moreover, since the samples used to determine the flow laws were the same as those deformed in our torsion experiments, the flow laws should provide a good description of the rheological properties of our samples. Second, the measured grain size represents the recrystallized grain size. As described in the Methods section, recrystallized grains were distinguished using the GOS-thresholding method developed for quartz by Cross et al. (2017). However, if grains in certain orientations have lower GOS values than those in other orientations, and thus, are preferentially picked as "recrystallized," this GOS-thresholding method cannot stand. In the application of this method, it is important to ensure that grain orientation does not influence the GOS. A plot of GOS versus Schmid factor of the (010)[100] slip system reveals that the value of GOS slightly increases by 0.4° as the Schmid factor increases from 0 to 0.5 (see Figure S3). This observation suggests that there is at most a weak effect of grain orientation on the residue intragranular misorientations. Therefore, the GOS-thresholding method could be used without causing significant bias. Another concern is whether the grain size observed in a radial section is the same as that observed in the corresponding tangential section of a torsion sample. A comparison between the microstructure from a radial section and a tangential section from similar radii reveals very similar average grain sizes (see Figure S4). Thus, we conclude that the grain sizes observed on radial sections are representative.

Plots of dynamically recrystallized grain size versus normalized stress for all of our samples are presented in Figures 11c and 11d using the arithmetic and geometric mean values of grain size, respectively. Three general observation warrant comment.

First, except for hydrous Fo<sub>50</sub>, values of *q* determined from fits of the results using the arithmetic mean for each sample in Figures 11c to Equation 13 lie in the range  $1.5 \le q \le 1.9$ , as reported in Table 3. This result is slightly higher than the values for *q* previously reported for Fo<sub>90</sub> and Fo<sub>50</sub> (e.g., Karato et al., 1980; Hansen et al., 2012a; Hirth & Kohlstedt, 2015). Except for hydrous Fo<sub>50</sub>, values of *q* determined from fits of the results using the geometric mean for each sample in Figures 11d to Equation 13 lie in the range  $1.3 \le q \le 1.4$ . This result is in good agreement with values for *q* obtained under hydrous and anhydrous conditions for samples of Fo<sub>90</sub> (Hirth & Kohlstedt, 2015; Jung & Karato, 2001a; Karato et al., 1980; Van der Wal et al., 1993) and under anhydrous conditions for samples of Fo<sub>50</sub> (Hansen et al., 2012a).

Second, at a given stress, the grain size of a sample deformed under anhydrous conditions is approximately equal to that of a sample of the same composition deformed under hydrous conditions (Figures 11c and 11d). A similar result was reported for samples of Fo<sub>90</sub> deformed at  $f_{\rm H_{2O}} \lesssim 300$  MPa (Jung & Karato, 2001a; Van der Wal et al., 1993). At  $f_{\rm H_{2O}} \gtrsim 500$  MPa, the dynamically recrystallized grain size exceeds that for samples deformed under anhydrous conditions with grain size increasing with increasing water content (Jung & Karato, 2001a).

Third, at a given stress under anhydrous conditions, the grain size is larger for the sample of  $Fo_0$  than for samples of  $Fo_{70}$  and  $Fo_{50}$  (Figures 11c and 11d). To extend this comparison over a wider range of iron con-



tent, the piezometer determined from radial sections of  $Fo_{90}$  samples deformed in torsion in our lab under anhydrous conditions (Meyers, 2020) is plotted with our results in Figure 12. Inclusion of the piezometer for  $Fo_{90}$  reveals a clear trend of increasing recrystallized grain size with increasing iron content. The fact that olivine samples with higher iron contents are deformed at higher homologous temperatures is unlikely the cause for this trend. Previous studies demonstrate for  $Fo_{90}$  olivine that the material parameters in Equation 13 do not vary significantly over a wide range of temperature (Karato et al., 1980; Van der Wal et al., 1993). Thus, we attribute this trend to the effect of iron.

Some insight into the reason that iron influences the relationship between dynamically recrystallized grain size and normalized stress comes from considering the terms that make up Equation 13. In this piezometric relation, iron potentially affects  $\gamma_{\rm gb}$ ,  $\mu$ , A, and/or q. Grain boundary energy for olivine is largely insensitive to iron content (Hiraga et al., 2010). In contrast, the shear modulus decreases with increasing iron content (Bass, 1995, Table 7), such that grain size would increase with increasing content. The modulus for Fo<sub>90</sub> is a factor of ~1.7 larger than the modulus of Fo<sub>0</sub> at our experimental temperatures (1200°C and 1100°C, respectively). However, at a given stress, the grain size of the Fo<sub>0</sub> sample is ~2.5 times larger than the grain size of Fo<sub>90</sub> samples, suggesting that the dislocation density—stress relationship in Equation 14, which is used in deriving Equation 13, accounts for at least part of the dependence of grain size on iron content.

A comparison of our results obtained on samples of  $Fo_{50}$  and  $Fo_{90}$  deformed under anhydrous conditions with published piezometers is also presented in Figure 12. For  $Fo_{50}$ , the piezometer determined in this study is in very good agreement with that obtained by Hansen et al. (2012a), which was based on analyses of tangential sections of a series of samples deformed to high strain in torsion. In contrast, at a given normalized stress, recrystallized grain sizes for anhydrous  $Fo_{90}$  calculated from the piezometers determined by Van der Wal et al. (1993) and Jung and Karato (2001a) are significantly larger than those calculated using the piezometer of Meyers (2020). This discrepancy is likely due to differences in the methods used for measuring grain size. Piezometers reported for  $Fo_{90}$  prior to our study used grain sizes measured by optical microscopy. In the present paper, grain size was determined by EBSD analyses. As demonstrated by Hansen et al. (2011), average grain sizes determined optically are often significantly larger than those determined by EBSD, likely accounting for the offset between the two piezometers for  $Fo_{90}$  olivine.

In addition, the value of q for hydrous Fo<sub>50</sub> sample is larger than the values for the other samples. Additional experimental work is needed to determine if this observation is robust or if it simple reflects sample-to-sample variations.

# 4.4. Arithmetic Mean and Geometric Mean

Values of grain size calculated from the arithmetic mean and the geometric mean were compared in several regions of each sample as illustrated in Figure 10. The geometric mean is always closer to the frequency peak of the grain-size distribution than the arithmetic mean, and the difference between the two means varies with the grain-size distribution. Several previous studies have proposed that the geometric mean provides a more representative estimate of the average grain size (Lopez-Sanchez, 2020; Lopez-Sanchez & Llana-Fúnez, 2015; Ranalli, 1984; Warren et al., 2008). However, the geometric mean is seldom used in the literature, because one key in processing a paleopiezometer is the reproducibility across studies (e.g., Stipp et al., 2010), and many studies prior to 2000 used the arithmetic mean.

Here we compare the grain size-stress relationships derived using both methods in Figures 11c and 11d. For the same sample, the magnitude of the exponent q in Equation 13 is smaller using the geometric mean than using the arithmetic mean. It seems that the deviation between the arithmetic mean and the geometric mean is larger if the grain-size distribution does not fit well to a log-normal distribution. However, the values of q obtained using the geometric means have a smaller deviation than those obtained using the arithmetic means. This result suggests that geometric mean may provide more consistency in the grain size analyses.



Parameters in the Piezometric Relationship for Each Sample							
Composition	Hydrous/anhydrous	μ(GPa)	Arithmetic mean	Geometric mean			
			q	q			
Fo <sub>70</sub> /Fa <sub>30</sub>	Anhydrous	56.7	$1.6 \pm 0.5$	$1.4 \pm 0.4$			
	Hydrous		$1.8 \pm 0.2$	$1.4 \pm 0.2$			
Fo <sub>50</sub> /Fa <sub>50</sub>	Anhydrous	51.4	$1.5 \pm 0.5$	$1.3 \pm 0.4$			
	Hydrous		$2.3 \pm 0.5$	$2.1 \pm 0.4$			
Fo <sub>0</sub> /Fa <sub>100</sub>	Anhydrous	37.3	$1.9 \pm 0.5$	$1.4 \pm 0.4$			

Table 3

# 4.5. Geological Applications

Although CPOs and the associated seismic anisotropy of sheared iron-rich olivine, typically Fo<sub>50</sub>, have been used to simulate those in natural olivine, Fo<sub>90</sub> (Hansen et al., 2012a, 2016, 2014; Tasaka et al., 2016; Wallis et al., 2019), whether or not the seismic anisotropy is the same in deformed  $Fo_{50}$  as in deformed  $Fo_{90}$  has not been tested. Our experiments reveal that, in olivines with different iron contents, deformation-induced CPOs are the same and the strength of seismic anisotropy reaches the same maximum after a shear strain of ~3.5. In addition, the evolution of radial seismic anisotropy for deformed olivine is not highly sensitive to iron content such that the experimental results in Figure 8b should provide a good approximation for naturally deformed, olivine-rich rocks as proposed by Hansen et al. (2014). Moreover, our results also suggest that seismic anisotropy in the mantle of Mars, if caused by the CPO of olivine, can be interpreted based on our understanding of the mantle of Earth, even though the iron content in olivine is different on Mars than on Earth.

# 5. Conclusions

- · In olivines with different iron contents deformed at a confining pressure of 300 MPa, CPOs transition from D-type fabrics, induced by dislocation glide on both the (010)[100] and (001)[100] slip systems, at low strains, to A-type fabrics, induced by dislocation glide on the (010)[100] slip system, at high strains.
- Variations in the iron content do not change the relative strengths of the primary slip systems in olivine, which are the (010)[100], (001)[100], and (100)[001]. CPOs in iron-rich olivines can be used to simulate CPOs in natural olivine, Fo<sub>90</sub>.
- Olivines with different iron contents exhibit the same strength of seismic anisotropy at a shear strain as low as 3.5, demonstrating that seismic anisotropy in natural olivine aggregates can be well-modeled using CPO data obtained from laboratory experiments on iron-rich olivine.
- The value of the stress exponent, q, in the piezometeric relationship lies between 1.3 and 1.4 over the range of iron contents investigated, similar to the previously reported values of 1.2-1.4.

# **Data Availability Statement**

Data can be found in this repository: https://doi.org/10.6084/m9.figshare.11838936.v1.

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