



Polycrystalline olivine rheology in dislocation creep: Revisiting experimental data to 8.1 GPa



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ABSTRACT

The rheology of polycrystalline San Carlos olivine is investigated on synchrotron beamline in the Deformation-DIA (D-DIA) at pressure (P) between 3.8 and 8.1 GPa, temperature (T) within 1373–1673 K, and at steady-state strain rates ranging from 1.1×10^{-5} to $5.8 \times 10^{-5} \text{ s}^{-1}$. Transmission electron microscopy (TEM) on run products reveals microstructures characteristic of the so-called “dislocation creep regime”. Fourier transform infrared (FTIR) measurements reveal hydroxyl concentrations within 153–1526 ppm H/Si (Paterson's calibration), indicating ‘wet’ conditions of deformation. Analysis of our data together with previously published ‘wet’ deformation data obtained at room and high P , assuming a stress exponent $n = 3.5$ in classical power law, results in a linear dependence of the activation enthalpy with P , *i.e.*, in an activation volume of $V^* = 12.8 \pm 5 \text{ cm}^3 \text{ mol}^{-1}$. This value of V^* is also consistent with a global dataset including ‘wet’ data and ‘dry’ published deformation data for olivine aggregates. We thus conclude that, up to 8 GPa, the effect of P on ‘dry’ and ‘wet’ olivine dislocation creep is consistent with $V^* = 12.8 \pm 5 \text{ cm}^3 \text{ mol}^{-1}$.

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1. Introduction

Lattice preferred orientations (LPO) produced in olivine aggregates deforming in dislocation creep are a key for the interpretation of upper mantle seismic velocity anisotropy in terms of mantle flows (*e.g.*, Gung *et al.*, 2003; Kneller *et al.*, 2005). Similarly, the rheological properties of olivine constrain geodynamic processes such as upper-mantle thermal convection and post-seismic relaxation (*e.g.*, Gerya *et al.*, 2002; Van Keken *et al.*, 2002; Freed and Burgmann, 2004; Kneller *et al.*, 2007). Olivine aggregate plasticity in dislocation creep is reasonably well documented at high temperatures (T) and low pressure ($P < 3 \text{ GPa}$) (*e.g.*, Carter and Ave'Lallemant, 1970; Durham and Goetze, 1977; Durham *et al.*, 1977; Karato *et al.*, 1986; Mei and Kohlstedt, 2000a,b; Hirth and Kohlstedt, 2003; Demouchy *et al.*, 2009; Faul *et al.*, 2011) and recent advances in high- P deformation devices (Yamazaki and Karato, 2001; Wang *et al.*, 2003; a review in Raterron and Merkel, 2009) now allow the investigation of olivine rheology over the whole P – T range of the upper mantle (Durham *et al.*, 2009; Ohuchi *et al.*, 2011; Hilaiet *et al.*, 2012; Dixon *et al.*, 2012). Despite those advances, olivine flow law parameters remain poorly constrained at high P . In classical power law creep, the effect of P on aggregate strain rate is quantified using an activation volume V^* . Reported

values, however, range from 0 to $27 \text{ cm}^3 \text{ mol}^{-1}$ (Table 1, *e.g.*, Ross *et al.*, 1979; Green and Borch, 1987; Borch and Green, 1989; Karato and Rubie, 1997; Kohlstedt and Wang, 2001; Wang, 2002; Karato and Jung, 2003; Li *et al.*, 2006; Durham *et al.*, 2009; Kawazoe *et al.*, 2009).

At the crystal scale, experimental data and calculations suggest a pressure-induced transition in the dominant dislocation slip systems, translating into changes in the produced LPO in the deformed polycrystals (*e.g.*, Couvy *et al.*, 2004; Durinck *et al.*, 2005, 2007; Raterron *et al.*, 2007, 2009, 2012; Jung *et al.*, 2009). For single crystals, this transition occurs between 6 and 9 GPa at 1673 K (Raterron *et al.*, 2009), and its effect on aggregate plasticity remains to be constrained (*e.g.*, Mainprice *et al.*, 2005; Ohuchi *et al.*, 2011; Raterron *et al.*, 2011). At low P , changes in dominant dislocation slip systems have been attributed to the effect of increasing hydroxyl content ([OH]) dissolved in olivine crystals (Karato and Jung, 2003; Jung *et al.*, 2006). It has also been reported that a small hydroxyl concentrations soften significantly olivine low- P plasticity, by about one order of magnitude on the strain rate (Poumellec and Jaoul, 1984; Mackwell *et al.*, 1985; Hirth and Kohlstedt, 2003; Demouchy *et al.*, 2012). In high- P experiments, however, controlling and/or maintaining crystal [OH] is challenging (Durham *et al.*, 2009; Otsuka and Karato, 2011) and actual high- P rheological measurements on olivine containing high [OH] have only been performed on single crystals (Girard *et al.*, 2013). Overall, more work is necessary in order to understand the respective effects of P and [OH] on olivine flow mechanisms.

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Table 1
Conditions of deformation and reported activation volume for olivine aggregate in dislocation creep regime.

References	Sample		Conditions				V^* (cm ³ mor ⁻¹)	Apparatus
			P (GPa)	T (K)	$\dot{\epsilon}$ (s ⁻¹)	n		
Ross et al. (1979)	Aggregates (dunite)	Dry	0.2–0.4	1373–1623			13.4 (2.0)	Griggs
Green and Borch (1987)	Aggregates		1.2–2.5	1373–1573	10^{-4} – 10^{-5}		27.0	Piston-cylinder
Borch and Green (1989)	Aggregates (peridotites)	Wet ^a	0.6–2.0	1250–1700	10^{-6}		27.0	Griggs
Karato and Rubie (1997)	Aggregates		0.3–15.0	300–1900	10^{-4} – 10^{-5}	3.0	14.0 (1.0)	Multi-anvil
Kohlstedt and Wang (2001) ^b	Aggregates		0.2–0.4				23.0	
Wang (2002) ^c	Aggregates	Dry	0.0–0.5	1473–1523	10^{-4} – 10^{-6}	3.3	23.0	Gas-medium
Karato and Jung (2003)	Aggregates	Wet	1.0–2.0	1473–1573	10^{-4}	3.0 ^d	24.0 (3.0)	Griggs
		Dry	1.0–2.0	1473–1573	10^{-4}	3.0 ^d	14.0 (2.0)	Griggs
Li et al. (2006)	Aggregates	Wet (?)	9.6	1473	10^{-5}	3.0 ^d	0.0 (5.0)	D-DIA
Durham et al. (2009)	Aggregates	Dry	2.7–4.9	1473	10^{-6} – 10^{-5}	4.0 ^d	9.5 (7.0)	D-DIA
Kawazoe et al. (2009)	Aggregates	Dry	4.9–9.6	1300–1870	10^{-5}	3.5	17.5 (2.5)	RDA
This study	Aggregates	Wet	3.8–8.1	1373–1673	10^5	3.5	13.0 (5.0)	D-DIA

^a After Hirth and Kohlstedt (1996).

^b After Hirth and Kohlstedt (2003).

^c After Durham et al. (2009).

^d After Mei and Kohlstedt (2000).

Here we report new results from high P – T deformation experiments performed on San Carlos olivine aggregates up to 8.1 GPa and 1673 K. Together with previous data, these results are interpreted with the aim of better understanding the respective effects of P and [OH] on olivine plasticity and lead to an activation volume of $V^* = 12.8 \pm 5 \text{ cm}^3 \text{ mol}^{-1}$.

2. Experimental details

Deformation experiments were performed in the Deformation-DIA apparatus (D-DIA, Durham et al., 2002; Wang et al., 2003) at the X17B2 beamline of the National Synchrotron Light Source (Upton, NY, USA), at P ranging from 3.8 to 8.1 GPa, T in the range 1373–1673 K and at controlled strain rates $\dot{\epsilon}$ ranging from 1.1×10^{-5} to $5.8 \times 10^{-5} \text{ s}^{-1}$ (Table 2).

2.1. Starting material, a_{OPx} and f_{O_2} conditions

The starting material was prepared by crushing one gem quality single crystal of San Carlos Olivine ($\text{Mg}_{0.9}\text{Fe}_{0.1}\text{SiO}_4$) in an alumina-mortar, grinding it to a fine-grained powder at the optical scale (average grain size $\sim 10 \mu\text{m}$). The starting olivine crystal was dry, as verified by Fourier transform infrared spectroscopy (FTIR, details below). The dry powder was mixed with a small fraction (5 wt.%) of natural Fe-bearing enstatite powder ($\text{En}_{84}\text{Fs}_{13}\text{Wo}_3$) in order to buffer the orthopyroxenes (OPx) activity ($a_{\text{OPx}} = 1$) and limit grain growth during deformation at high temperature. Each olivine specimen was simultaneously deformed with a forsterite specimen (pure Mg_2SiO_4 + 5 wt.% enstatite) – both materials separated by metal foils and alumina powder to avoid contact. Forsterite rheological data, however, goes beyond the topic of this study and is not reported here. Oxygen fugacity (f_{O_2}) was not controlled during the runs, to avoid encapsulating the specimens into metal jackets which diffraction lines would reduce the accuracy on differential stress (σ) evaluation by X-ray diffraction. However, recovered specimens were transparent with no color alteration related to oxidation or reduction, and no silica-rich or metallic micro-precipitates resulting from crystal destabilization (e.g., Raterron et al., 2000; Leroux et al., 2003) were observed at the TEM scale. This attests that olivine remained within its Redox stability field during the runs.

2.2. Experimental set up

Specimens were loaded into the D-DIA deformation cell (Fig. 1) in between two machinable alumina end plugs; once sintered at

high- P , the end plugs become hard pistons. Taking advantage of the ten-element energy-dispersive multi-detector (Weidner et al., 2010), P and σ were measured *in situ* following the protocol described by Bollinger et al. (2012). Specimen strain (ϵ) and strain rate ($\dot{\epsilon}$) were measured by time-resolved X-ray radiography (Vaughan et al., 2000) from the positions on radiographs of the metal foils placed at sample ends.

In the deformation cell, T is generated with a sleeve of graphite (the heater) and measured using two horizontal W3%Re–W25%Re thermocouples (no pressure correction) whose junctions are placed at the centre of the cell, next to the alumina powder. The uncertainty on T , mostly due to vertical gradients within the cell, has been calibrated at the centre of this assembly (Raterron et al., 2013) and can be in excess of $\sim 150 \text{ K/mm}$ at 1673 K. Given the nominal temperature of the present runs and the size and vertical position of our specimens, we estimate the sample T uncertainty to $\pm 100 \text{ K}$ during the run. Temperature gradients in the cell do not affect significantly X-ray diffraction stress measurement from the alumina pistons but they cannot be omitted if measuring the cell pressure from the equation of state alumina (Raterron et al., 2013). Here, T gradients have been accounted for in the whole analysis by assuming that temperature in the pistons was 200 K lower than that measured in the thermocouple.

2.3. Deformation protocol

A total of 8 D-DIA experiments were performed, resulting in 20 steady-state deformation conditions in axial compression (Table 2). Most experiments were performed following the same protocol. The cell is first pressurized at room T and then brought to high T by supplying power to the graphite heater (Fig. 1). Hydrostatic conditions are maintained at high T for about 1 h at the onset of the run. This ensures the sintering of pistons and specimens, the recovery of specimen damage, and the relaxation of internal stress resulting from cold compression. Axisymmetric compression of the cell assembly by displacement of D-DIA vertical anvils is initiated after the hydrostatic annealing, and maintained at a fixed rate until the desired sample strain (typically several %) at constant strain rate is achieved. Several temperature and/or sample strain-rate conditions are usually tested during each run (Table 2; Fig. 2), while specimen images and diffraction spectra are recorded by X-ray radiography and diffraction, respectively. At the end of the run, T is quenched by turning off the power supply and both D-DIA vertical anvils are stopped. P is then decreased gradually to room- P for 1–2 h. To preserve specimen deformation microstructures during

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