

# Deformation of Diopside Single Crystal at Mantle Pressure, 1: Mechanical data

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#### **Revised Manuscript**

# 3 Deformation of Diopside Single Crystal at Mantle Pressure, 1: 4 Mechanical data

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#### 12 Abstract

13 Steady state deformation experiments were carried out in a Deformation-DIA 14 high-pressure apparatus (D-DIA) on oriented diopside single crystals, at pressure (P) ranging from 3.8 to 8.8 GPa, temperature (T) from 1100° to 1400°C, and differential 15 16 stress ( $\sigma$ ) between 0.2 and 1.7 GPa. Three compression directions were chosen in order 17 to test the activity of diopside dislocation slip systems, i.e.,  $\frac{1}{2} < 110 > \{1\overline{1}0\}$  systems activated together, both [100](010) and [010](100) systems together, or [001] 18 19 dislocation slip activated in (100), (010) and {110} planes. Constant applied stress and 20 specimen strain rates ( $\dot{\varepsilon}$ ) were monitored *in situ* using time-resolved synchrotron X-ray 21 diffraction and radiography, respectively. Transmission electron microscopy (TEM) 22 investigation of the run products revealed that dislocation creep was responsible for 23 sample deformation. Comparison of the present high-P data with those obtained at 24 room-P by Raterron and Jaoul (1991) - on similar crystals deformed at comparable T- $\sigma$ conditions - allows quantifying the effect of P on  $\frac{1}{2} <110 > \{1\overline{1}0\}$  activity. 25 This translates into the activation volume  $V^* = 17 \pm 6 \text{ cm}^3/\text{mol}$  in the corresponding creep 26 power law. Our data also show that both  $\frac{1}{2} <110$  dislocation slips and [001] have 27 28 comparable slip activities at mantle P and T, while [100](010) and [010](100) slip 29 systems remain marginal. These results show that P has a significant effect on high-T30 dislocation creep in diopside, the higher the pressure the harder the crystal, and that this 31 effect is stronger on  $\frac{1}{2} < 110$  slip than on [001] slip.

32

33 Keywords: upper mantle, clinopyroxenes, diopside, high pressure, dislocation creep,

- 34 slip systems, rheological law, activation volume
- 35

#### 36 **1. Introduction**

37 Clinopyroxenes (cpx) are major constituents of eclogites, and are present in 38 excess of 10 vol.% at most depths in the pyrolitic upper mantle (Green et Ringwood, 39 1963). Among mantle minerals, they exhibit the strongest anisotropy for seismic wave 40 propagation (see Mainprice et al., 2000). Cpx plastic properties may thus significantly 41 affect both mantle rheology and seismic anisotropy. Yet, no study of cpx rheology at 42 high-pressure (typically P > 3 GPa) has been reported so far - mostly because of 43 technical limitations - whereas P reaches 14 GPa in the deep upper mantle at the 44 transition zone boundary. A number of experimental studies of cpx crystal and/or cpxrich aggregate deformation, carried out at low  $P \leq 3$  GPa in dead load apparatuses, 45 46 Paterson machines, and Griggs apparati, have been reported since Griggs et al.'s (1960) 47 early work (Raleigh and Talbot, 1967; Avé Lallemant 1978; Kollé and Blacic, 1982, 1983; Kirby and Kronenberg, 1984; Boland and Tullis, 1986; Raterron et Jaoul, 1991; 48 Ingrin et al., 1991; Ingrin et al., 1992; Jaoul et Raterron, 1994; Raterron et al., 1994; 49 50 Mauler et al., 2000; Bystricky and Mackwell, 2001; Dimanov et al., 2003; Chen et al., 51 2006; Zhang et al., 2006; Zhang and Green, 2007). These studies address the 52 fundamentals of cpx plastic deformation mechanisms and their respective activities as a 53 function of temperature (T) and fluid (i.e., water, oxygen) fugacities, in a pressure range 54 representative of the Earth crust and uppermost mantle of the lithosphere. .

We report here the first investigation of cpx rheology at asthenosphere pressure. We investigate the high-temperature ( $T \ge 1100$ °C) plasticity of diopside single crystals because i) diopside is a good representative of mantle cpx, and ii) the rheology of diopside single crystals was thoroughly investigated at room *P* in the early 1990's, which gives 1-atm reference data for comparison with our high-pressure data. The present steady-state deformation experiments were carried out in a Deformation-DIA

apparatus (D-DIA) coupled with X-ray synchrotron radiation. This high-pressure
equipment (see Durham et al., 2002, and Wang et al., 2003) allows measurement *in situ*of the applied stress (see Li et al., 2004; Burnley and Zhang, 2008) and resulting
specimen strain rates (Raterron et al., 2007, and 2009).

#### 65 **2. Samples and deformation experiments**

#### 66 2.1 Starting material

Eleven cylindrical specimens - about 1.4 mm in diameter and up to ~2 mm in length - were cored in natural centimeter-sized diopside crystals, and polished on both ends. The starting crystals were of gem-quality, dark green and transparent, and contained no detectable defect at the binocular microscope scale. Their formula was determined either by electron microprobe or by energy dispersive spectroscopy (EDS) in the transmission electron microscope. It varies slightly (fractions of at.%) from one crystal to the next, with an average formula:

74

 $Ca_{0.960}Na_{0.024}Mg_{0.947}Fe_{0.043}Cr_{0.015}Ti_{0.002}Al_{0.008}Mn_{0.004}Ni_{0.007}Si_{1.994}O_{6}$ 

75

#### 2.2 Crystal orientations and active slip systems

76 In the pyroxene structure (see Cameron and Papike, 1981), dislocation lines 77 cannot cut through the  $(SiO_3)_{\infty}$  chains because of the strong Si-O-Si bound. In the 78 diopside monoclinic structure (C2/c space group), this allows four possible glide planes: 79 (100) and (010) planes, and two duplex planes {110}. According to previous 80 experimental studies, twinning in (100) and (001) is in diopside a major deformation 81 process at low temperature - typically lower than 800°C, e.g., Avé Lallemant (1978). At 82 temperature higher than 1000°C, diopside plastic deformation is dominated by the activation of duplex  $\frac{1}{2} <110 > \{1\overline{1}0\}$  dislocation slip systems (e.g., Raterron et al., 83 84 1994), with Burgers' vector  $(b_r)$  0.66 nm in length. These systems are followed in

activity by [001]{110} then by [001](100), both with  $\|\boldsymbol{b}_{\boldsymbol{r}}\| = 0.52$  nm, while [100](010) and [010](100) slip systems remain marginal because of their large Burgers vectors (0.97 and 0.89 nm, respectively).

88 In order to test the activities of diopside dislocation slip systems, specimens were 89 cut in three crystallographic orientations, which were verified by transmission electron 90 microscopy (TEM). Cylinders were cut with their long axes within 5 degrees of the 91 chosen orientations parallel to [010], [110] and [225] directions, corresponding to the 92 orientations respectively labelled [2], [3], and [4] by previous authors (Raterron and 93 Jaoul, 1991; Jaoul and Raterron, 1994; Raterron et al., 1994). Five specimens were cut 94 in orientation [2], which allows the duplex  $\frac{1}{2} < 110 > \{1\overline{1}0\}$  systems with a maximum Schmid factor  $S \sim 0.5$  (Table 1). Three other specimens were cut in orientation [3] 95 96 which allows mostly both [100](010) and [010](100) systems (with  $S \sim 0.5$ ). Finally 97 three specimens were cut in orientation [4] in order to activate [001] glide in (100), 98 (010) and {110} planes, with  $0.3 \le S \le 0.5$  in all glide planes.

99

#### 2.3 Deformation apparatus and deformation protocols

100 Specimens were deformed in between two alumina pistons, in compression along 101 their axis - at P ranging from 3.8 to 8.8 GPa, T within 1100°-1400°C, and at  $\sigma$  ranging 102 from 210 to 1660 MPa (details below) - in the D-DIA on the X17-B2 beam line of the 103 National Synchrotron Light Source (NSLS, Upton, NY, USA). We used boron-epoxy 104 cubic pressure media and, to compress the cells, tungsten carbide anvils opaque to the X 105 rays together with 1 or 2 transparent sintered-diamond anvils to allow back lateral 106 (horizontal) diffraction. For the deformation cell, we used a similar design as that 107 reported in Raterron et al. (2009); within the 6.15-mm D-DIA cell, temperature was 108 generated using a vertical graphite furnace separated from the boron epoxy cube by an

109 alumina sleeve, and from the sample by a BN sleeve. Temperature was measured with 110 two horizontal W3%Re-W25%Re thermocouples which junctions placed next to the 111 vertical compression column in the middle of the cell. The uncertainty on temperature, 112 mostly due to T gradients within the cell (no pressure correction), has been previously 113 measured in the center of similar cells to less than 20 K/mm. Given the size of our specimens and other possible sources of errors, such as the effect of P on thermocouple 114 115 emf and/or the distance between thermocouple and sample, we estimate run T116 uncertainty to  $\pm 100^{\circ}$ C in a deformed cell; this large uncertainty also results from a 117 power vs. temperature relationship which evolves with the cell strain, promoting 118 significant deviations from calibration curves at large strain.

119 Two types of experiments were conducted: single-crystal experiments, where one 120 single crystal is loaded (Figure 1.a), and bi-crystal experiments (Figure 1.b) where two 121 crystals of different orientations are loaded in a given cell. This latter geometry, 122 introduced by Raterron et al. (2007), ensures that both crystals experience the same 123 stress field during deformation. It allows direct comparison of crystal deformations (i.e., of dislocation slip system activities) at identical T, P, and  $\sigma$  conditions, with no 124 125 need of precise knowledge of the applied stress. Along the compression column, we 126 used porous-alumina pistons in order to accommodate cold compression deformation 127 (piston cataclastic compaction) with as little damage as possible on specimen(s) and 128 calibrants (details below). At pressure and temperature, porous alumina becomes dense, 129 hard sintered, which ensures the piston strength during deformation. Re foils were used 130 to mark samples ends on the radiographs (Figure 1) and allow *in situ* measurement of 131 sample strain and strain rate (details below).

Experimental run conditions and resulting specimen strains and strain rates are reported in Table 2, where they are regrouped by crystal orientations for a total of 33

134 steady state conditions. Three of the 8 conducted experiments, runs Dio18, Dio21 and 135 Dio24, were bi-crystal experiments with 2 diopside crystals of different orientations in 136 each cell. The other 5 experiments (Dio06, Dio11, Dio12, Dio17, and Dio19) were 137 single-crystal experiments. During all experiments, specimens were cold compressed to 138 the desired pressure in typically 3 to 4 hours. T was then increased in 30 min to 1 hour 139 to 1000°C or more. Stress free and constant P and T conditions were maintained until 140 diffraction patterns arising from the polycristalline stress calibrants within the cell 141 (olivine, forsterite, spinel and/or alumina, see below) showed no evidence of stress 142 remaining from cold compression, i.e., no peak broadening and similar spectra on all 143 EDS detectors. Both inner (vertical) rams of the D-DIA were then moved forward at 144 constant rates to deform the cell assembly, which usually translated into specimen 145 steady state deformation. All of the reported data in Table 2 correspond to steady state 146 deformation condition, i.e., constant stress and specimen strain rate. Each steady state 147 condition corresponds, for all but 5 of the 33 experimental points, to a specimen strain 148 of about 3 % (and up to 10 %). Strains < 2 % are only obtained when one crystal in the 149 hard orientation [3] is deformed together with a soft orientation-[2] crystal (bi-crystal 150 experiments). This was the case during runs Dio21 and Dio24 (Table 2), where crystals 151 in orientation-[2] and the cells achieved large strains before the experiments were 152 stopped. During all experiments, the applied stress  $\sigma$  and resulting specimen strain ( $\varepsilon$ ) 153 and strain rate ( $\dot{\varepsilon}$ ), as well as pressure, were measured in situ by X-ray radiography and 154 diffraction (see next section). Specimens were quenched (temperature dropped quickly) 155 at the end of the runs, by cutting off the furnace power. Pressure was then decreased to 156 room P within 2 to 3 hours. To preserve specimen deformation microstructures during 157 decompression, specimen length was monitored by radiography and maintained 158 constant by adjusting D-DIA inner-ram decompression rate manually. This latter

adjustment was, however, not very accurate and specimens often achieved some strain(typically 0.5 to 1 % strain) during cold decompression.

161

#### 2.4 In situ stress and strain rate measurements

162 P,  $\sigma$ , and  $\dot{\varepsilon}$  were monitored by time-resolved x-ray diffraction and radiography. 163 These techniques allow in situ measurement of P and stress applied on specimens, and 164 resulting specimen strain rates. Pressure and applied stress were measured from the 165 quantification of x-ray peak shifts in diffraction patterns arising from stress calibrants: 166 polycrystalline olivine (San Carlos olivine or pure forsterite Fo100) and spinel pellets 167 placed in the compression column, and/or the piston polycrystalline alumina. Stress 168 calibrants were separated from the sample(s) by Re foils (Figure 1). Except for the 169 alumina pistons and spinel pellets, calibrants were loose powders at the beginning of the 170 runs and were sintered at pressure and temperature. Pressure was deduced from 171 calibrant unit-cell volumes using the corresponding equations of state (for San Carlos olivine and pure forsterite: Isaak (1992), Zha et al. (1996); for spinel: Askarpour et al. 172 173 (1993), Anderson and Isaak (1995), Kruger et al.(1997); and for alumina: Anderson et 174 al.(1992)), while stress  $\sigma = \sigma_1 - \sigma_3$  was deduced from differences in the *d*-spacing 175 characterizing lattice planes in different orientations with respect to the principal stress 176  $\sigma_1$  (Li et al., 2004; see also Singh et al., 1998). Stress uncertainty using these techniques 177 is fairly large, since it depends on the mechanical response of individual grains to the 178 applied stress (Burnley et Zhang, 2008; Merkel et al., 2009), as well as the accuracy of -179 d-spacing measurement - i.e., on the position of X-ray diffraction peak maxima and 180 shifting toward higher energy on compression (smaller *d*-spacing) - which in turn 181 depends on several experimental factors (see Raterron et al., 2007). For this calculation, 182 we assumed that  $\sigma_1$  was homogeneous along the vertical compression column, and that 183 the (horizontal)  $\sigma_3$  was constant throughout the BN sleeve surrounding the samples.

184 This was often verified by the consistency, within uncertainties, in stress values 185 measured using different calibrants. In some cases however, mostly at temperature of 186 1100°C, stress values obtained from different materials gave inconsistent results. This 187 discrepancy may be due to several factors: i) at 1100° C, parts of the assembly such as 188 the alumina pistons may not fully relax, leading to pressure and stress gradients within 189 the cell; ii) stress-calibrant diameter depends on material responses to cold compression 190 - the softer, the larger diameter, i.e. the more materials out of the compression column -191 which may result in underestimating the applied stress. Other sources of discrepancy 192 includes: iii) different uncertainties on different equation-of-state parameters and 193 elastic-constants used to process the data; iv) stress-calibrant grain growth, leading to 194 offset diffraction peak positions; v) diffraction peaks from cell materials (BN, Re foils, 195 etc.) superimposed on stress calibrant peaks; iv) Re strain markers partly shield the 196 vertical EDS detectors from diffracted x-rays, etc.

197 Specimen plastic strain was measured on time-resolved X-ray radiographs 198 collected on an x-ray fluorescent YAG crystal. Radiographs were recorded after 199 magnification on a CCD camera. Sample and calibrant end positions appear as black 200 lines on the radiographs (e.g., Raterron et al., 2007). These lines result from X-ray 201 absorption by 25-um thick metal-foils (strain markers: here Re foils). They allow 202 measurement of specimen length l(t) as a function of time, from which specimen strain 203 and strain rate can be deduced. Specimen strain as a function of time  $\varepsilon$  (t) was deduced 204 from l(t) using the relationship:  $\varepsilon(t) = \ln l_o/l(t)$  (here in compression  $\varepsilon(t) \ge 0$ ), where  $l_o$ 205 is the initial length of the specimen at given conditions. Steady-state strain rates ( $\dot{\varepsilon}$ ) 206 and their uncertainties were then deduced from straight slopes observed over more than 207 typically 1% strain on  $\varepsilon$  (t) versus time plots, all other parameters (P, T, and  $\sigma$ ) being 208 constant.

209

#### 2.5 Run oxygen fugacity (fO<sub>2</sub>) and silica activity $(a_{SiO_2})$

210 Diopside plastic properties depend slightly on oxygen fugacity, with an  $fO_2$ 211 exponent  $m \sim -0.2$  in single-crystal rheological laws (Jaoul and Raterron, 1994).  $fO_2$ 212 was not controlled during our runs, although the Re-foils used as strain markers stayed 213 in contact with diopside specimens during the runs. Re metal was still present in the run 214 products, and no obvious sign of diopside reduction or oxidation (change of crystal 215 color, intracrystalline precipitates) was detected by optical microscopy or TEM. This 216 suggests that specimens stayed in their  $fO_2$  stability field during the run. The pressure 217 of either olivine or forsterite (used as stress calibrant in every cell) has implications for 218 silica activity. Contact with the diopside specimens was maintained on the sides of the 219 Re foils. Silica-rich precipitates were not observed within run products which shows that diopside early partial melting (EPM, e.g., Doukhan et al., 1993; Raterron et al., 220 221 1995) did not occur during our experiments.

222

#### 2.6 Sample water contents

223 Diopside crystals can host significant amounts of water (e.g. Bell and Rossman, 224 1992). One can distinguish, however, between the hydroxyl (OH) groups part of 225 amphibole lamellae present as inclusions within diopside bulk, from the OH<sup>-</sup> present in 226 diopside solid solution as protons (H<sup>+</sup>) linked to structural oxygen. These two types of 227 hydroxyls do not have the same effects on crystal rheology, since amphibole (not structural) hydroxyls do not affect diopside dislocation activities while  $H^+$  defects 228 229 within diopside may significantly affect diopside rheology (see Chen et al., 2006). It is, 230 however, not trivial to separate the contribution of each type of hydroxyl group in 231 unpolarized Fourier transformed infrared (FTIR) spectra. In the following, we assume 232 that all hydroxyls detected by FTIR in the diopside crystals we investigated are related

10

to structural H<sup>+</sup> defect concentration, since no amphibole inclusions were observed in
the crystals, either optically or by TEM.

235 I.R. spectra were obtained using a PerkinElmer 2000 spectrometer fitted with a 236 MCT/B nitrogen-cooled detector attached to a microscope. An unpolarized beam was focused on several regions of each  $\sim 0.3$  to 0.5-mm thick double-polished section cut 237 from the starting crystals or the retrieved deformed specimens. Residues of the epoxy 238 239 used to preserve the run products during section preparation resulted in a wide band in 240 the middle of the spectra, which was subtracted in order to reveal the OH-bands 241 observable in the range 3200 and 3800 cm<sup>-1</sup>. The absorbance normalized to 1-cm 242 sample thickness was obtained by integrating four OH-bands centered at 3647, 3530, 3435 and 3355 cm<sup>-1</sup>, using Bell et al.'s (1995) calibration. We are aware that Bell et al 243 244 (1995) calibration is given for polarized IR measurements, while we used unpolarized 245 IR. Unfortunately there is no theoretical basis for a relationship between polarized and 246 unpolarized measurements. In the case of olivine, Bell et al. (2003) showed that 247 unpolarized IR measurements may underestimate hydroxyl contents by a factor ranging from 1.5 to 3.5, depending on the assumed orientation factor. On this basis, we 248 249 systematically multiply by 3 the absorbance we measured in our samples before 250 calculating the resulting water contents. Because of this rough correction, the water 251 contents reported here may be overestimated and the relative uncertainty is large, on the 252 order of 50%. The water contents measured in starting material and run products using 253 the method described above are reported in Table 3.3. Results

#### 255 **3.2** Run product microstructures

Quenched run products were investigated by optical microscopy and TEM. The corresponding observations will be reported in details in a companion paper ("Deformation of diopside single crystals at mantle pressure 2: TEM characterization of dislocation microstructures"); we report here a short summary of the observed microstructures.

261 Petrographic sections  $\sim$  30-um thick were prepared from the retrieved specimens. 262 Sections for TEM were ion thinned to electron transparency with a 5-kV argon beam, 263 then carbon coated and investigated with a Philips CM30 electron microscope operating at 300 kV. TEM investigation revealed the dislocation slip systems expected to be 264 265 active in each crystal orientation – i.e., in orientation [2] the duplex  $\frac{1}{2} < 110 > \{1\overline{1}0\}$ 266 systems; in orientation [3] both [100](010) and [010](100) systems; in orientation [4] 267 glide of [001] dislocation in {110}, (100), and (010) planes. TEM observations also 268 show that, although  $\frac{1}{2} < 110$  and [001] were as expected responsible for most specimen 269 strain in orientation [2] and [4], respectively, these systems contribute to some extent to 270 the deformation of all specimens. In order to quantify this effect, their contributions to 271 specimen strain were calculated from geometrical considerations and the observed 272 dislocation densities. We estimate that  $\frac{1}{2} < 110$  dislocation glide contributes to less 273 than 7% and 14% of the strain of specimen in orientation [4] and [3], respectively, while 274 glide of [001] dislocation in (100), (010) and {110} planes contributes to 20% and 8% 275 of the strain of specimens oriented along [2] and [3], respectively.

TEM observations also reveal twin lamellae which in most sample are likely the result of cold decompression, although high-temperature twinning may have also

occurred in sample subjected to high stress levels (typically > 1 GPa) at high
temperature.

280

#### 3.3 High-P rheological data analysis and results

281 *Effect of P on \frac{1}{2} < 110> dislocation glide* 

In order to quantify the effect of *P* on the activity of  $\frac{1}{2} <110 > \{1 \overline{1} 0\}$  slip systems, orientation-[2] specimen rheological data were fitted to a rheological law. A general creep power law for an Fe-bearing silicate reads:

285 
$$\dot{\varepsilon} = A\sigma^n f O_2^m \exp{-\frac{E^* + PV^*}{RT}}$$
(1)

where *A*, *n*, *m* are constants, *R* is the gas constant,  $E^*$  is the activation energy, and  $V^*$  is the activation volume which quantifies the effect of *P* on crystal high-*T* rheology. Since diopside rheology does not strongly depend on  $fO_2$  and specimens stayed in their  $fO_2$ stability field during our experiments (see section 2), we omit the  $fO_2$  term in Equation (1) which leads to the expression:

291 
$$\dot{\varepsilon} = A\sigma^n \exp{-\frac{E^* + PV^*}{RT}}$$
(2)

292 During our experiments, P and T were known to within  $\pm 0.5$  GPa and  $\pm 100^{\circ}$ C, 293 respectively. Yet, the large uncertainty affecting  $\sigma$  values (typically > 30 MPa) prevents 294 the accurate determination of the stress exponent n in Equation (2) from the high-P data 295 alone, i.e. the full determination of specimen rheological laws. The large stress 296 uncertainty which affects only high-P data, addressed in details by Burnley and Zhang 297 (2008), lead Raterron et al. (2009) to combine room-P and high-P data to constrained 298 olivine rheological laws. In the following, we combine also our high-P data with the 299 room-P deformation data reported by Raterron and Jaoul (1991). These authors carried 300 out high-temperature creep experiments on diopside single crystals cut in orientation [2] 301 and of comparable composition to that of our specimens. Raterron and Jaoul's

304 by diopside EPM (see section 2) and are thus representative of equilibrated orientation-

305 [2] crystal deformed at room *P*. At room pressure (*P*=0) Equation (2) becomes:

306 
$$\dot{\varepsilon} = A\sigma^n \exp{-\frac{E^*}{RT}}$$
(3).

302

303

307 Hence, high-*P* strain rates  $\dot{\varepsilon}(P,T,\sigma)$  can be deduced from room-*P* strain rates 308  $\dot{\varepsilon}(0,T,\sigma)$  by combining Equations (2) and (3):

309 
$$\dot{\varepsilon}(P,T,\sigma) = \dot{\varepsilon}(0,T,\sigma) \times \exp{-\frac{PV^*}{RT}}$$
(4).

At given *T*- $\sigma$  conditions, values of *V*\* can be obtained from the slopes on ln( $\dot{\varepsilon}$ ) versus (-*P/RT*) plots. Complete rheological laws taking into account the effect of *P* can thus be written using Equation (4), by coupling high-*P* data with well established room-*P* rheological laws as expressed in Equation (3).

314 Figure 2.a shows  $\log_{10}(\dot{\varepsilon})$  versus (-*P/RT*) plots for crystals in orientation [2], 315 as obtained from Table 2 data points coupled with room-P data points which were 316 calculated using Raterron et al.'s (1991) rheological law. Error rectangles around high-317 P experimental points were calculated taking into account experimental uncertainties on P, T, and  $\dot{\varepsilon}$ , as reported in Table 2. Stress uncertainties were taken into account when 318 319 calculating room-P strain rates: each high-P point, thus, corresponds to 2 room-P points 320 (same symbol on Figure 2.a) which were plotted using either the minimum stress (open 321 symbol) or the maximum stress (full symbol) as deduced from the stress values and 322 uncertainties reported in Table 2. A maximum slope (solid line) and a minimum slope (dashed line) can thus be obtained for each high-P experimental point. These two 323 slopes correspond to, respectively, maximum and minimum  $V^*$  values, and are 324 325 constrained by i) the size of the P-T- $\dot{\varepsilon}$  uncertainty rectangle around each high-P point, 326 and ii) the two calculated room-P strain rates corresponding to each high-P point. 327 Indeed, room-P strain rates are calculated using the room-P rheological law as 328 previously reported for crystal in orientation [2], with the high-P experimental T and

using either a minimum stress value (average stress minus its uncertainty) or a maximum stress value (average stress plus its uncertainty) as deduced from each high-*P* stress measurement. This method allows taking into account all sources of uncertainties, i.e. uncertainties on *P*, *T*,  $\sigma$  and  $\dot{\varepsilon}$ , when determining *V*\* values for crystal in orientation [2] as reported in Table 4.

From equivalent slopes obtained from  $\ln(\dot{\varepsilon})$  vs. (-*P/RT*) plots, we deduced the maximum  $V^*_{max}$  and minimum  $V^*_{min}$  values compatible with each high-*P* point, reported in Table 4. Finally, taking into account uncertainties on experimental values  $V_i^* = (V_i^*_{max} + V_{i\mbox{min}}^*)/2$ , i.e., applying to each of them a weight  $\omega_i = 1/(\delta V_i^*)^2$ where  $\delta V_i^*$  is the uncertainty  $(V_{i\mbox{max}}^* - V_{i\mbox{min}}^*)/2$ , and using the general weighted mean value formula:

340 
$$V^* = \frac{\sum_{i} \omega_i \times V_i^*}{\sum_{i} \omega_i}$$
(5),

341 we obtained the mean value  $V^* = 17 \pm 6 \text{ cm}^3/\text{mol}$  for the orientation-[2] crystal 342 rheological law (Equation 2). This  $V^*$  value completes the set parameters obtained at 343 room-*P* by Raterron and Jaoul (1991), and leads to the complete set of parameters 344 reported in Table 5. The large activation volume obtained here for crystals in 345 orientation [2] shows that *P* significantly inhibits glide of  $\frac{1}{2} < 110 >$  dislocations in {110} 346 planes.

347 Figure 2.b shows  $\log_{10}(\dot{\varepsilon})$  versus (-l/RT) for crystal in orientation [2], as obtained from Table 2 data points corrected to nominal P = 6 GPa and  $\sigma = 300$  MPa 348 using the  $V^*$  and *n* values reported in Table 5. Error rectangles around the experimental 349 350 points show uncertainties on experimental T and  $\dot{\varepsilon}$ , taking into account the uncertainties 351 in  $\sigma$ . The plain line on Figure 2.b is a plot of orientation-[2] rheological law using Table 352 5 parameters in Equation (2). The dashed line was obtained with the  $V^*$  and n values 353 reported in Table 5, fitting the experimental points (least square regression) to values for the pre-exponential factor A and the activation energy  $E^*$  in Equation (2). Figure 354

2.b shows that, although Table 4 parameters - with *A*, *n*, and *E*\* parameters obtained at room-*P* - account well for our high-*P* data, a fit through the high-*P* data alone leads to the higher activation energy  $E^* \sim 702$  kJ/mol (instead of 442 kJ/mol at room *P*). Such an increase with pressure in the apparent activation energy  $E^*$  may result from a change with pressure in the mechanism for  $\frac{1}{2} < 110$ > dislocation glide. Further investigation is however needed to confirm this preliminary result.

#### 361 ½<110> glide activity vs. [100], [010] and [001] glide activities

Room-*P* rheological data for crystals in orientations [3] and [4] are too sparse (see Raterron and Jaoul, 1991, and Raterron et al., 1994) to apply the analytical method described above for orientation-[2] crystals. Bi-crystal experiments however, such as runs Dio18, Dio21 and Dio24, are designed to compare slip-system activities by comparing the strain rates of crystals in different orientations with respect to principal stress directions.

368 Figure 3.a shows specimen strain ( $\varepsilon$ ) versus time during run Dio18, where one 369 orientation-[2] crystal and one orientation-[4] crystal were loaded on top of each other 370 in the assembly compression column (Figure 1.b). Both crystals experienced identical 371 *P-T-\sigma* path during the run. Their resulting strains and strain rates are comparable - often 372 identical within uncertainties - which shows that the high-T activities of the duplex 373  $\frac{1}{2} \le 110 \ge \{1\overline{1}0\}$  systems (orientation [2]) and that of [001] glide in and  $\{110\}$ , (100), and to a lesser extent (010) (orientation [4]), are comparable at mantle pressure (6 < P <374 375 9 GPa). This differs from observations at room P, which show that  $\frac{1}{2} < 110 > \{1\overline{1}0\}$  systems dominate diopside deformation at high temperature (Raterron et 376 377 al., 1994). This difference in the relative activities of  $\frac{1}{2} < 110$  and [001] slips at room P 378 and high P suggests that pressure inhibits  $\frac{1}{2} < 110$  glide more than [001] glide. This 379 should translates into a lower activation volume  $V^*$  in the rheological law (Equation 2) 380 describing orientation-[4] crystal plasticity than that obtained for crystals in orientation [2] ( $V^* \sim 17 \text{ cm}^3/\text{mol}$ ), although further investigation is needed to establish this point. 381

382 Runs Dio21 and Dio24 (bi-crystal experiments) were designed to compare the activities of  $\frac{1}{2} < 110 > \{1\overline{1}0\}$  slip systems (orientation [2]) with activities of [100](010) 383 384 and [010](100) systems activated together (orientation [3]). Figure 3.b shows specimen  $\varepsilon$  versus time during run Dio21. During this run, the crystal in orientation [2] 385 386 deformed at strain rates 5 to 8 times higher than that of the crystal in orientation [3]. 387 This shows that  $\frac{1}{2} < 110 > \{1\overline{1}0\}$  activity dominates over those of [100](010) and 388 [010](100) at mantle pressures (7 < P  $\leq$  8 GPa, Table 2 and 3). Results from run Dio24, 389 where orientation-[2] crystal deformed more than twice as fast as orientation-[3] crystal, 390 confirm this observation. A similar observation was made for room-P experiments, in which  $\frac{1}{2} < 110 > \{1\overline{1}0\}$  systems dominate deformation over [100](010) and [010](100)391 392 systems (Raterron et al., 1994). Both [100] and [010] dislocations have longer Burgers 393 vectors ( $b_r$ , respectively 0.97 and 0.87 nm long) than that of  $\frac{1}{2} < 110$  dislocation (0.66 394 nm long), which increases the energy involved in dislocation glide by 2 (roughly  $\propto$  $b_r^{2}$ ). 395

We conclude that  $\frac{1}{2}<110>\{1\overline{1}0\}$  slip systems and [001] glide in  $\{110\}$ , (100), and to a lesser extend (010), have comparable activities at high *P* and *T*. These systems largely dominate diopside deformation over [100](010) and [010](100) systems at mantle *P* and *T* conditions.

#### 400 **4. Discussion and geological implications**

An important result of the present study is the quantification of the effect of *P* on  $\frac{1}{2} < 110 > \{1\overline{1}0\}$  slip system activity, which are dominant systems of diopside at room *P* and temperatures above 1000°C. Increasing pressure inhibits  $\frac{1}{2} < 110 >$  glide, which translates into the activation volume  $V^*=17\pm6$  cm<sup>3</sup>/mol (Equation (2)) for crystals in orientation [2]. This shows that orientation-[2] crystals significantly harden with pressure. Diopside viscosity, thus, significantly increases with *P* (e.g., Figure 2.a). Assuming this property extrapolates to all mantle cpx, one can expect cpx to be much

stronger in the deep upper mantle - leading to reductions in strain rate by several orders
on magnitude- than expected from room-*P* rheological laws. In this context, the
complete rheological law reported here for crystals in orientation [2] (Table 2) provides
a first clue to quantify cpx viscosity at mantle conditions.

412 TEM investigation of the run products (see companion article for details) shows that glide of  $\frac{1}{2} < 110$  dislocations dominates deformation of orientation-[2] crystals. 413 This suggests that the  $V^*$  value mostly results from an increasing lattice friction with P 414 415 for  $\frac{1}{2} < 110$  dislocations glide in {110} planes. This interpretation, however, cannot be 416 definitive without proper modeling of  $\frac{1}{2} < 110$  dislocation core and elastic properties. 417 Indeed, empirical rheological laws such as Equation (2) account for differential stress 418 and pressure as two independent variables. In fact, both  $\sigma$  and P are partial expressions 419 of the stress tensor and their respective effects on crystal plasticity cannot be easily 420 separated. This is particularly true in the case of dislocation creep, where crystal 421 deformation results from dislocation mobility which strongly depends on their core 422 structures. According to computational studies, dislocation cores are complex and extended in silicates, likely in several directions (e.g., Walker et al., 2005; Carrez et al., 423 424 2008), which makes them sensitive to both  $\sigma$  and P. This was recently modeled in the 425 case of forsterite dislocations (Durinck et al., 2005; Durinck et al., 2007) which provided 426 the theoretical background to explain the inhibition effect of P on olivine [100](010) 427 slip system (Raterron et al., 2007; Raterron et al., 2009). Similar modeling is thus required to explain the inhibition effect of P on diopside  $\frac{1}{2} <110 > \{1 \overline{1} 0\}$  slip systems 428 429 observed here.

430 Another important result is that both  $\frac{1}{2} < 110$  and [001] glides have comparable 431 activities at mantle *P* and *T*. This observation differs from room-*P* observations 432 (Raterron et al., 1994), which show that  $\frac{1}{2} < 110$  glide is dominant at high temperature.

It is, however, consistent with the recent experimental study of omphacite rheology at 3 433 GPa reported by Zhang et al. (2006). These authors conclude that  $\frac{1}{2} <110 > \{1\overline{1}0\}$ , 434 435  $[001]{110}$  and [001](100) slip systems dominate deformation at high-T. Our result is 436 also consistent with cpx lattice preferred orientations (LPOs) observed in naturally 437 deformed eclogites from various origins (The Alpes, Norway, Spain, Mali, China, etc.). 438 Omphacite and augite LPOs in deformed eclogites have been extensively studied over 439 the past 25 years (e.g., Van Roermund et Boland, 1981; Van Roermund, 1983; Buatier 440 et al., 1991; Skrotzki, 1994; Godard et Van Roermund, 1995; Bascou et al, 2001, 441 Brenker et al., 2002). In nature, cpx develop marked LPOs characterized by a strong 442 concentration of [001]-axes sub-parallel to the lineation and of (010)-poles roughly 443 normal to the foliation. Such LPOs form in relation with the deformation of eclogites through dislocation creep, as revealed by TEM observations of naturally deformed cpx 444 445 grains. The LPOs are explained by self-consistent visco-plastic (VPSC) modeling of 446 cpx aggregate deformation (Bascou et al, 2001; Bascou et al., 2002). VPSC models show, in the case of omphacite, that such LPOs can develop by the dominant activity of 447  $\frac{1}{2} < 110 > \{1\overline{1}0\}, [001] \{110\}$  and [001] (100) dislocation slip systems, a result in 448 excellent agreement with the results reported here for diopside crystals deformed at 449 mantle P and T. 450

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Slip	Orientation	Orientation	Orientation
systems	[2]	[3]	[4]
[001](100)	0	0.14	0.36
[001](010)	0	0.13	0.34
[001](110)	0	0.19	0.49
[001](110)	0	0	0
[110](110)	0.49	0	0
[110](110)	0.49	0.09	0.07
[010](100)	0	0.48	0.30
[100](010)	0	0.5	0.20

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#### 604 **Table 1:** Schmid factors for potential slip systems

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Run	Point #	Pressure (GPa)	Temperature (°C)	Stress (MPa)	Strain rate (10 <sup>-6</sup> s <sup>-1</sup> )	Total strain (%)	
orientation [2]							
Dio17	17-1	7.8 ± 0.4	1400	333 ± 74 <sup>a</sup>	15.4 ± 1.5	5.0	
1800µm	17-2	$7.6 \pm 0.4$	1400	290 ± 100 <sup>a</sup>	8.7 ± 1.5	3.2	
	17-3	$6.4 \pm 0.6$	1200	505 ± 100 <sup>a</sup>	11.9 ± 1.6	5.0	
Dio18	18-1	8.8 ± 0.1	1400	236 ± 100 <sup>ab</sup>	14.2 ± 2.2	5.8	
1070µm	18-2	$7.9 \pm 0.3$	1400	210 ± 48 <sup>ab</sup>	6.1 ± 1.7	3.4	
	18-3	$7.0 \pm 0.2$	1200	928 ± 35 <sup>ab</sup>	16.3 ± 2.0	8.2	
	18-4	6.9 ± 0.1	1200	864 ± 52 <sup>a</sup>	7.1 ± 2.1	3.6	
	18-5	6.4 ± 0.1	1200	499 ± 65 <sup>0</sup>	7.1 ± 2.1	3.6	
Dio19	19-1	7.5 ± 0.6	1300	500 ± 80 <sup>a</sup>	18.9 ± 1.7	6.3	
1700µm	19-2	$6.9 \pm 0.2$	1300	373 ± 80 <sup>a</sup>	9.0 ± 1.5	3.7	
	19-3	7.3 ± 1.0	1100	1660 ± 140 <sup>ab</sup>	31.0 ± 2.2	9.3	
	19-4	6.3 ± 1.0	1100	1606 ± 230 <sup>ab</sup>	10.2 ± 1.7	4.5	
Dio21	21-1	8.0 ± 1.1	1400	282 ± 46 <sup>ab</sup>	21.2 ± 1.5	6.5	
1250µm	21-2	$7.3 \pm 0.8$	1400	276 ± 75 <sup>ab</sup>	12.5 ± 1.2	5.5	
Dio24	24-1	8.0 ± 0.7	1300	354 ± 82 <sup>ab</sup>	12.7 ± 1.1	5.6	
1050m	24-2	$8.2 \pm 0.3$	1300	252 ± 66 <sup>ab</sup>	7.4 ± 1.0	4.3	
orientation [3]							
Dio06	6-1	8.3 ± 0.2	1200	1347 ± 200 <sup>ad</sup>	28.5 ± 3.5	6.4	
1970µm	6-2	8.1 ± 0.1	1200	975 ± 330 <sup>ad</sup>	11.0 ± 2.2	4.2	
Dio21	21-1	8.0 ± 1.1	1400	282 ± 46 <sup>a</sup>	3.8 ± 1.4	1.2	
1280µm	21-2	7.3 ± 0.8	1400	276 ± 75 <sup>b</sup>	1.5 ± 1.0	0.7	
Dio24	24-1	8.0 ± 0.7	1300	354 ± 82 <sup>ab</sup>	4.4 ± 1.7	1.8	
710µm	24-2	$8.2 \pm 0.3$	1300	252 ± 66 <sup>ab</sup>	3.2 ± 1.3	1.9	
orientation [4]							
Dio11	11-1	5.7 ± 0.2	1300	493 ± 150 <sup>c</sup>	10.8 ± 2.0	2.6	
1110µm	11-2	$5.0 \pm 0.2$	1300	347 ± 160 <sup>c</sup>	7.8 ± 1.3	2.9	
	11-3	3.8 ± 1.7	1100	943 ± 220 <sup>ac</sup>	4.6 ± 1.2	2.0	
	11-4	$4.5 \pm 2.0$	1100	1010 ± 220 <sup>ac</sup>	37.5 ± 2.3	9.0	
Dio12	12-1	7.7 ± 0.2	1400	213 ± 180 <sup>c</sup>	14.5 ± 1.3	4.2	
1380µm	12-2	$8.5 \pm 0.9$	1400	349 ± 86a <sup>c</sup>	6.7 ± 1.0	3.5	
	12-3	7.0 ± 0.1	1200	608 ± 280 <sup>c</sup>	5.0 ± 1.2	3.6	
	12-4	7.2 ± 0.1	1200	645 ± 170 <sup>c</sup>	34.4 ± 1.6	10.1	
Dio18	18-1	8.8 ± 0.1	1400	236 ± 100 <sup>ab</sup>	11.8 ± 1.8	4.9	
1220µm	18-2	7.9 ± 0.3	1400	210 ± 48 <sup>ab</sup>	5.5 ± 1.4	3.0	
	18-3	$7.0 \pm 0.2$	1200	928 ± 35 <sup>ab</sup>	23.7 ± 2.7	7.4	
	18-4	6.9 ± 0.1	1200	864 ± 52 <sup>a</sup>	7.1 ± 2.1	3.0	
	18-5	6.4 ± 0.1	1200	$499 \pm 65^{b}$	7.1 ± 2.1	3.0	

607 **Table 2**. Experimental conditions and results for crystals in orientation [2], [3] and [4]

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Sample	OH <sup>-</sup> content (H/10 <sup>6</sup> Si)
Starting material	8960
Dio19 (orientation [2])	1640
Dio11 and Dio18 (orientation [4])	2480
Dio24 (orientation [3])	3950

#### 611

612 **Table 4:** *V*\* values obtained for crystal in orientation [2]

Run	Point #	V* <sub>min</sub> (cm³/mol)	V* <sub>max</sub> (cm <sup>3</sup> /mol)
Dio17	17-1 17-2	11 9 1	23 24
	17-3	9.8	22
Dio18	18-1	4.4	18
	18-2	7.8	19
	18-3	16	26
	18-4	16	28
	18-5	11	28
Dio19	19-1	12	23
	19-2	11	24
	19-3	15	24
	19-4	17	32
Dio21	21-1	9.7	19
	21-2	9.6	23
Dio24	24-1	8.2	18
	24-2	1.1	18

613	$V_{\min}^*$ and	$V^*_{max}$ :	respectively	minimum	and maximum	V* obtained
614	in ln(strain	rate) v	versus -P/RT	plots, takin	ng into account	uncertainties

619 **Table 5:** Equation (2) parameters for crystals in orientation [2]

$\operatorname{Ln}(A)^{\dagger}$	$n^{\dagger}$	$E^*(kJ/mol)^{\dagger}$	$V^*(\text{cm}^3/\text{mol})$
-7.8	6.46	442	17 ± 6

620  $\ddagger$  from Raterron et al. (1991); *A* is in MPa<sup>-n</sup>

<sup>615</sup> on P, T,  $\sigma$ , and fO<sub>2</sub>. The weighted mean V\* value is obtained using

<sup>616</sup> Equation (5).

<sup>617</sup> 

<sup>618</sup> 

621 **Figure 1**: schematic drawing of the compression column within a) the single-crystal

- 622 experiment cell and b) the bi-crystal experiment cell. Stress calibrants used here
- 623 include polycrystalline olivine, forsterite, spinel and alumina (see text and Table 2).



**Figure 2**: Orientation-[2] crystal high-*P* experimental points with corresponding uncertainty rectangles. a) log(strain rate) vs. (P/RT) plot and corresponding *V*\* value in Equation (2); at each high-*P* point correspond 2 room-*P* points, as obtained taking into account  $\sigma$  uncertainty on high-*P* data and using Raterron and Jaoul's (1991) rheological law. b) log(strain rate) vs. (1/RT) plot as extrapolated at the indicated *P*,  $\sigma$  conditions using Table-5 parameters (indicated *V*\* value). See text for further explanation.





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Figure 3: Specimen total strain ( $\varepsilon$ ) versus time during bi-crystal runs a) Dio18 and b) Dio21, showing the deformation of orientation-[2] crystals (black squares) with that of crystals in orientation [4] and [3] (colored symbols), respectively. Vertical lines separate different regimes of deformation at the indicated *P* and *T* conditions. The numbers next to the deformation curves indicate corresponding steady-state strain rates (in 10<sup>-6</sup> s<sup>-1</sup>). The corresponding stress values are listed in Table 2. See text for further information.

