



Strength of orthoenstatite single crystals at mantle pressure and temperature and comparison with olivine



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ABSTRACT

Oriented single crystals of orthopyroxenes (OPx) were deformed in axisymmetric compression in the D-DIA at pressure and temperature in excess of 3 GPa and 1040 °C. Two crystal orientations were tested with the compression axis parallel to either $[101]_c$ crystallographic direction, to investigate $[001](100)$ dislocation slip-system strength, or $[011]_c$ direction to investigate $[001](010)$ slip-system strength. These slip systems are the most active in orthopyroxenes. Applied differential stresses and specimen strain rates were measured in situ by synchrotron X-ray diffraction and radiography. We used these data and comparison with previously reported low-pressure flow laws for protoenstatite and orthoenstatite to determine the power law parameters for the deformation of orthoenstatite crystals, which characterize OPx dislocation slip-system strengths. Applying these laws at reasonable mantle stresses along oceanic and continental geotherms indicates that OPx $[001](100)$ slip system is weaker than OPx $[001](010)$ slip system to ~260 km depth where the strengths converge. It also indicates that both OPx slip systems are significantly stronger than olivine slip systems throughout the upper mantle, except in the upper most mantle, in the lithosphere, where OPx $[001](100)$ slip system may be as weak or even weaker than olivine $[100](010)$ easy slip system.

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1. Introduction

Thermal convection of ductile peridotites in the Earth's upper mantle drives plate tectonics. Orthopyroxenes (OPx) are the second most abundant constituent of peridotite in the top 200 km, with volume fraction in excess of 15% (Green and Ringwood, 1963; Frost, 2008). They are also major constituents of the mantles of planetary bodies with enstatite-chondrite origins, such as Mercury (Ebel and Alexander, 2011). $Mg_{0.9}Fe_{0.1}SiO_3$ orthoenstatite (*Pbca* space group), commonly called enstatite, is a good representative of Earth's mantle orthopyroxenes; yet, its high-temperature plasticity is still poorly constrained. This is because at low temperature (T) the stable phase is clinoenstatite (Presnall, 1995; Fig. 1), while at $T > 1000$ °C and pressure (P) lower than typically 1.0 GPa it transforms into protoenstatite, an unquenchable orthorhombic polymorph (*Pbcn* space group) which has similar b and c axes but an a axis almost twice as short as orthopyroxene (0.93 nm

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instead of 1.82 nm for the Mg end member). High shear stress on (100) plane also promotes the ortho-clinoenstatite transition, rarely observed in naturally deformed rocks, which renders difficult studying enstatite rheology at moderate temperature, even at high pressure (Raleigh et al., 1971; Coe and Kirby, 1975). Here, we present new experimental data on the high-temperature deformation of orthoenstatite single crystals, obtained in the *Pbca* stability field. These data allow estimating the effect of pressure on enstatite rheology in the dislocation creep regime and comparing orthopyroxenes and olivine strength at mantle conditions.

Dislocation creep was identified in early studies as an important deformation mechanism at high temperature in dry and wet enstatite (Raleigh et al., 1971; Kohlstedt and Vander Sande, 1973; Ross and Nielsen, 1977). This has been confirmed by further studies (Van Duysen et al., 1985; Nazé et al., 1987) which identified $[001](100)$ as the easiest slip system, together with two other slip systems: $[001](010)$ and $[010](100)$. Dissociation of $[001]$ edge dislocations in (100) plane, according to the reaction $[001] \rightarrow 1/6[001] + 5/6[001]$, is also reported in association with the ortho-clinoenstatite transformation at low to moderate temperature; glide of the strong $5/6[001]$ partial dislocation generates then the “untwinning” of one orthorhombic unit cell into two monoclinic unit cells. Dissociation of $[001]$ dislocation has also been recently

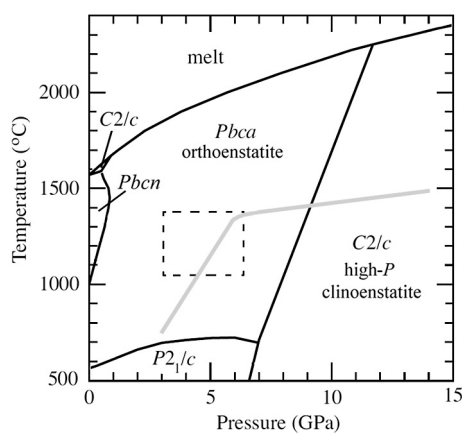


Fig. 1. Part of the MgSiO₃ phase diagram (after Presnall, 1995) indicating the stability field of five enstatite phases with their respective space groups. Low iron contents do not significantly affect the field boundaries (e.g., Woodland, 1998). The grey line shows a classic continental geotherm (Turcotte and Schubert, 2002). See text for further explanation.

observed in naturally deformed crystals, in association with sub-grain boundary formation (Kogure et al., 2014). Bruijn and Skemer (2014) estimate that, in the upper mantle, orthopyroxenes with grain size larger than ~150–500 μm deform in the dislocation creep regime.

The strength contrast between olivine and orthoenstatite has significant implications for peridotite deformation and, hence, for mantle convection. However, experimental studies of olivine + enstatite aggregate plasticity, which are beyond the scope of this article, lead to confusing results (see Supplementary material for details). One approach to simplify the problem, and constrain olivine and orthoenstatite strengths in the dislocation creep regime, is to investigate the plasticity of oriented single crystals. Deforming crystals in specific crystallographic orientations allows activating one or several specific dislocation slip systems, and quantifying their individual plastic responses. The high-temperature rheology of olivine single crystals has been extensively studied (e.g., Mackwell et al., 1985; Bai et al., 1991; Raterron et al., 2012; Girard et al., 2013). In contrast, little work has been done on the rheology of orthopyroxene single crystals. Schloessin and Ranalli (1988) deformed protoenstatite single crystals by three-point bending at 1 atm and 1050 °C, which activated the [001](100) slip system; but their work was focused on demonstrating the feasibility of the techniques. Mackwell (1991) reports the investigation of protoenstatite single-crystal rheology at 1 atm. The crystals were axially deformed at temperature in excess of 1300 °C, with the compression axis at 45° angle from both [100] and [001] directions (along the so-called [101]_c crystallographic direction), which promotes the activation of the easiest [001](100) slip system alone with a maximum Schmid factor ($S = 0.5$). Mackwell observed that they deformed by dislocation creep and, from comparison with olivine single-crystal rheology, inferred that enstatite is likely stronger than olivine under mantle conditions. Although these results on protoenstatite crystals do not directly apply to mantle *Pbca* enstatite crystals, they provided a first quantification of [001](100) slip system strength at high temperature in an orthorhombic enstatite crystal. Interestingly, Mackwell (1991) observed that creep is insensitive to oxygen fugacity despite the presence of iron, unlike that of olivine single crystals (Bai et al., 1991). He also determined a stress exponent of $n = 3.8 \pm 0.5$, similar to that of olivine single crystals ($n = 3.5 \pm 0.1$, Bai et al., 1991). More recently, Ohuchi et al. (2011) carried out shear deformation experiments in a Griggs apparatus on oriented orthoenstatite single crystals in contact with polycrystalline olivine ($P = 1.3$ GPa and $T = 700$ – 1100 °C). They investigated the activity of [001](100)

and [001](010) dislocation slip systems. However, in their experiments shear strain and differential stress were not measured, but constrained after the runs by quantifying the rotation of a strain marker in the run products and measurement of dislocation density in adjacent olivine, which does not allow determination of whether steady-state conditions of deformation were achieved. Also, their data for [001](100) slip system were obtained after specimens had almost entirely transformed into low clinoenstatite; so the corresponding rheological laws characterize creep in clinoenstatite and not orthoenstatite. Nevertheless, Ohuchi et al. (2011) report a first rheological law quantifying the activity of [001](010) slip system in mantle (*Pbca*) orthoenstatite. From comparison of their flow law with that of olivine single crystals, as well as comparison of the single-crystal strain with that of the adjacent polycrystalline olivine, they conclude that orthoenstatite is significantly weaker than olivine at lithospheric conditions. This result contradicts Mackwell (1991) and is also inconsistent with estimates based on field observations of orthopyroxene versus olivine strength contrast (e.g., Tikoff et al., 2010). There is clearly a need for more experiments on orthoenstatite rheology to better constrain mantle deformation.

2. Experimental methods and run-product microstructures

2.1. Starting materials

Cylindrical specimens, ~1.2 mm in diameter, were cored from gem-quality enstatite crystals from Tanzania (En_{84.9}Fs_{14.7}Wo_{0.4}, Supplementary material, Table S1), then polished on both ends to a final length of 1 to 2 mm. Two crystallographic orientations were chosen – verified to within $\pm 5^\circ$ by transmission electron microscopy (TEM) – with the axis of the cylinder (the compression axis) along either the [101]_c direction mentioned above, or [011]_c direction which is at 45° angle from both [010] and [001] directions and promotes the activation of [001](010) dislocation slip system with $S = 0.5$. In order to compare enstatite strength with that of olivine, oriented olivine specimens were prepared in a similar way from gem-quality San Carlos olivine crystals, with their axis along either [110]_c direction or [011]_c direction which promote respectively [100](010) or [001](010) easy slip systems. In some of our experiments, enstatite crystals were deformed together with enstatite aggregates sintered from powders at the onset of the runs. The starting powders (grain size ranging from 1 to 5 μm), were prepared from Tanzanian crystals and a crystal of enstatite (En_{76.8}Fs_{22.6}Wo_{0.6}, Table S1) from Bamble, Norway (details in Supplementary material). In order to control silica activity during the runs, the enstatite crystals were enrobed with a thin layer (~20 μm) of San Carlos olivine powder prior to loading into the high-pressure cell. Similarly, olivine crystals were enrobed with a thin layer of enstatite powder.

2.2. Water content

Water contents were measured in the starting material and selected run products by collecting unpolarized infra-red absorbance spectra using the Nicolet Magna 560 FTIR spectrophotometer with NicPlan microscope at Texas A&M University (details in Supplementary material). Fig. 2 shows the absorbance spectra for the starting material and the single-crystal products of runs EN020 and EN022. The starting material is almost dry, as showed by the quasi absence of OH band in the corresponding spectrum. Run-product hydroxyl contents (C_{OH}) were calculated using the Paterson (1982)'s calibration leading to values of 380 and 780 ppm H/Si for the sample from run EN020, and 340 ppm H/Si for the sample from run EN022. See Supplementary material for details regarding the observed hydroxyls bands. The observed differences

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