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Evidence of phase nucleation during olivine diffusion creep: A new perspective for mantle strain localisation

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ABSTRACT

For the past decades, grain size reduction leading to diffusion creep in olivine is believed to be a very important process for strain localisation in the lithospheric mantle. However, the mechanisms of grain size reduction in this regime are still poorly understood (e.g., Platt, 2015). Here we show new experimental results that document grain size reduction and material weakening during wet olivine diffusion creep. While occurring for both, mono-phase and two-phase aggregates, grain size reduction is coeval with strain localisation and local phase mixing in olivine–pyroxene aggregates. Based on evidence of fluid inclusions and cracks filled with a fine-grained phase mixture, we conclude that grain size reduces as a result of fluid-assisted nucleation that takes place in the presence of an aqueous fluid during diffusion creep. Cavitation induced by grain boundary sliding (creep cavitation) can be inferred, and may play a critical role for olivine grain size reduction. Amongst their implications for rock rheology in general, our findings highlight a key process for strain localisation in the ductile uppermost mantle.

1. Introduction

In order for tectonic plates to be isolated from each other by high-strain plate boundaries, such as subduction zones or narrow rifts, strain localisation has to occur across the strong uppermost mantle (Burov and Watts, 2006; Bercovici and Ricard, 2014). The deformation of the lithosphere strongly depends on this presence or absence of strain localisation. Although some brittle behaviour may be expected in the shallow mantle, extrapolations of lab-based flow laws to corresponding depths give predictions of the uppermost mantle as mostly plastic (Kohlstedt et al., 1995). In this regime, strain localisation typically arises from a local weakening of the deforming rock, the origin of which remains very elusive today.

Olivine is by far the most abundant phase in the upper mantle. For this reason, experimental, field-based, and modelling studies have focused on the role of olivine for mantle rheology in the last decades, concluding that grain size reduction and dominant diffusion creep may be the best candidate to account for quasi-viscous strain localisation (Rutter and Brodie, 1988; Drury et al., 1991; Bercovici and Ricard, 2012). Their conclusion is based on the fact that (1) grain size reduction is a ubiquitous feature of ductile shear zones, and (2) diffusion creep (including grain boundary sliding) is grain size sensitive, unlike dislocation creep which does not – or to a lesser extent in the case of disGBS (Hansen et al., 2011) – depend on grain size (Karato et al., 1986; Hirth and Kohlstedt, 2003). However, grain size reduction is not a process which is defined or understood during dominant diffusion creep (e.g., Rutter and Brodie, 1988; De Bresser et al., 2001). Thus, although reducing grain size in this regime may be a key factor for strain localisation (Rutter and Brodie, 1988; Bercovici and Ricard, 2012; Platt, 2015), the processes are poorly known and require further investigation.

Many studies have pointed out the presence of minor phases, such as micas or pyroxenes, as a condition for small grain sizes during grain-size-sensitive creep (Hiraga et al., 2010; Herwegh et al., 2011; Farla et al., 2013; Linckens et al., 2014; Platt, 2015). Most of them attributed the reduction of grain size to a combination of dynamic recrystallization and grain boundary sliding (GBS), resulting in phase mixing. Following this approach, Bercovici and Ricard (2012) developed a theoretical model to propose that damages at olivine–pyroxene interfaces would indirectly drive grain size to reduction, provided that grain boundary pinning (Zener pinning) arises from phase mixing. However, natural evidence of grain size reduction induced by pinning of grain boundaries is still lacking; Zener pinning has been only shown to inhibit grain growth, not

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reducing grain size (Stünitz and Fitz Gerald, 1993; de Ronde et al., 2005; Warren and Hirth, 2006; Sundberg and Cooper, 2008). Furthermore, the link between phase mixing, grain size reduction and GBS during diffusion creep, as well as the transition from dislocation creep to diffusion creep, is still unclear (Kilian et al., 2011; Platt, 2015). And not all aspects are supported by available data. In recent high-temperature (>1200 °C) experiments on two-phase material (olivine + pyroxene), it has been shown that diffusion creep tends to aggregate phases because of its related component of GBS, promoting grain growth instead of grain size reduction (Hiraga et al., 2013).

In this study, we show new experimental results that document grain size reduction during diffusion creep in olivine at wet conditions of the uppermost mantle. The reduction can be understood in the presence of second phases, but not necessarily; we document it for both mono-phase (olivine) and two-phase (olivine + pyroxene) aggregates. In presence of pyroxene, strain localisation also goes along with grain size reduction and gives rise to typical ductile shear zones where complete phase mixing occurs. Both chemical and microstructural features suggest that grain size reduction results from fluid-assisted nucleation of olivine and pyroxene grains.

2. Material and methods

2.1. Sample preparation

In all experiments, we used olivine (Fo_{91}) from San Carlos (Arizona, USA) and clinopyroxene ($Wo_{51}En_{48}Fs_1$) from Cranberry lake (Canada). To produce the starting material, we crushed gem-quality single crystals in an agate mortar and sorted the grain size using a decantation method with distilled water. For olivine, we used a grain size ranging from 1 to 10 μm with a mean of 2 μm in all experiments. As for clinopyroxene (CPx), two types of grain sizes were used: one with fine CPx between 5 and 20 μm, and a second one with coarser CPx grains between 40 and 125 μm. Olivine and pyroxene powders were mixed at a ratio of 70 to 30% (by weight) in a solution of ethanol, following the procedure described in de Ronde et al. (2005). The powder was then placed between two alumina shear pistons cut at 45° to the piston axis, wrapped in a nickel foil and enclosed in a platinum jacket welded at both ends. The shear piston surfaces have been previously roughened using 120-grit corundum paper. Before welding, 0.1 or 0.2 wt.% of distilled water were added. These amounts exceed the solubility of water in olivine and pyroxene at our experimental conditions (Zhao et al., 2004; Bromiley et al., 2004). Such wet conditions and grain size have been chosen to ensure that (1) olivine will deform in the diffusion creep regime (Hirth and Kohlstedt, 2003; Sundberg and Cooper, 2008; Hansen et al., 2011), and (2) initial grains will be large enough to see significant grain size reduction, if applicable. We also have chosen to use clinopyroxene – instead of orthopyroxene – to ensure that secondary phases will be stronger than the olivine matrix (Bürgman and Dresen, 2008). Few grains of orthopyroxene (enstatite; ∼0.2 wt.%) and amphibole (tremolite; ∼0.8 wt.%) were present in the CPx powder despite initial hand-picking (Fig. 1).

2.2. Deformation experiments

The deformation experiments were conducted in a solid medium (NaCl) Griggs-type apparatus at Tromsø University (Norway). Temperature and pressure were alternately increased over several hours up to the conditions of experiment (900 °C and 1.2 GPa). Samples were hot-pressed *in situ* for 24 h (microstructures of hot-pressed material are available in supplementary Fig. 1). The deformation piston (σ_1-piston) was then advanced at a constant rate, first through the top lead piece, and then touching the alumina piston (Fig. 1A). Samples started to deform at this point, several hours after starting to advance the σ_1-piston. At the end of the experiment, the samples were quenched from 900 °C to 200 °C in 2 min. Both, pressure and differential stress decreased rapidly until the temperature stabilised at 200 °C. Subsequently, the sample was decompressed at a rate of ∼5 MPa per minute, keeping the differential stress ∼100 MPa above the confining pressure to reduce the formation of unloading cracks. Finally, temperature was dropped to 30 °C when the confining pressure was lower than 100 MPa. After the experiment, the samples were impregnated with epoxy, and then sectioned along the piston axis for thin sections.

2.3. Analytical procedures

Sample microstructures were analysed with a scanning electron microscope (SEM) of ISTO/BRGM (Orléans, France) and a transmission electron microscope (TEM) at the University of Minnesota (Minneapolis, USA). SEM work has been performed on carbon-coated (20 nm thickness) thin section using a TESCAN MIRA 3 XMU at 25 kV and a working distance of 25 mm. TEM images were taken using a FEI-TECNAI T12 at 120 kV through a focused ion beam (FIB) foil of 50–100 nm thickness. In the TEM, Energy dispersive X-ray spectroscopy (EDS) was used to analyse phases. Electron backscatter diffraction (EBSD) was employed for Lattice Preferred Orientation (LPO) and grain size determination on polished surfaces (diamond paste of 0.25 μm followed by colloidal silica) with an EDAX Pegasus system at 25 kV and a working distance of 18 mm. Lower-hemisphere pole figures of LPO were constructed considering both, the whole map dataset (available in supplementary Fig. 2) and one measurement per grain acquired manually. The data from EBSD maps have been processed with the OIM^{TM} – EDAX software, and the grain size distributions are shown with respect to the percent area of each category in order to compare between sample types. For LPO, the texture (J) and misorientation (M) indices are also given, which both quantify the degree of minerals alignment between $J = 1/M = 0$ for a uniform distribution, and $J = \infty/M = 1$ for a crystal-like distribution considering one point per grain (Bunge, 1982; Skemer et al., 2005). Phase composition, including starting material, was measured with an electron microprobe CAMECA SX five at ISTO on carbon-coated (20 nm thickness) thin sections at 15 keV and 10 nA. The step size of the microprobe map was 0.5 μm.

To measure *in situ* water content of both olivine and pyroxene grains, we performed ion probe measurements on gold-coated (20 nm thickness) thin sections using a Cameca IMS 1280-HR at the CRPG (Nancy, France). A primary beam of Cs^+ was used with a current of 3 nA and a beam diameter of 10 μm. The positive secondary beam was centred in a 2500 μm image field aperture. The voltage offset was stable at −50 eV and the energy window has been set to 20 eV. The counting elements were ^{16}O, ^{17}O, ^{16}OH, ^{17}OH, ^{28}Si and ^{30}Si with a counting time between 3 s (for Si) and 12 s (for OH). The total counting time was 12 min per point. To calibrate the ratio H/Si, we used the ratio H_2O/SiO_2 from standards, including basaltic glass (M35, M40, M48, T1G, StHs, Kl2G), synthetic forsterite (Fo_{100}), clinopyroxene (cpxNSH), pyrope (MON 9) and quartz (Suprasil). The very good quality of vacuum into the chamber ($6 - 7 \times 10^{-10}$ Torr) indicated that no epoxy degassing occurred during the analyses.

3. Results

3.1. Mechanical data

The strain–stress curves of all experiments show an increase of differential stress to peak stress at a bulk shear strain of