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Invited research article

Helium incorporation and diffusion in polycrystalline olivine

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ABSTRACT

Helium is a key tracer of mantle geochemical and isotopic heterogeneities and can constrain our understanding of mantle geodynamics. Nevertheless, the mechanisms of helium storage and transport in mantle minerals remain poorly understood. Polycrystalline olivine was doped with helium at high temperature (1050 ± 25 °C) and high pressure (0.30 ± 0.01 GPa), followed by step heating extraction experiments to investigate helium storage and diffusion in Earth's upper mantle. We also tested the effect of heterogeneous initial concentrations on the extracted diffusivities, and demonstrate the robustness of diffusion parameters obtained in this study. Our results show that two diffusion processes are acting in polycrystalline olivine: (i) a high temperature process with high activation energy (E_a) where diffusion is only controlled by lattice diffusion, and (ii) a lower temperature process with lower E_a where diffusion is controlled by both grain boundary and lattice diffusion. These two diffusion processes are separated by a transition temperature that depends on the depletion of helium hosted in grain boundaries, i.e., the amount of helium stored at grain boundaries and the temperature and duration of the step heating sequence. Our results confirm that grain boundaries can represent a significant storage site for He. Moreover, we report two different populations of diffusion parameters in the lattice diffusion field, which are interpreted as diffusion in interstitials ($E_a = 95 \pm 15$ kJ·mol⁻¹ and $\log(D_0) = -8.26 \pm 2.13$) and Mg vacancies ($E_a = 168 \pm 19$ kJ·mol⁻¹ and $\log(D_0) = -3.59 \pm 2.12$). Similar diffusion parameters populations are observed in literature data after reprocessing the diffusivities. Furthermore, we determine grain boundary diffusion parameters: $E_a = 57 \pm 14$ kJ·mol⁻¹ and $\log(D_0) = -9.20 \pm 0.99$. Applying these results to the upper mantle reveals that an important amount of He can be stored at grain boundaries for typical mantle grain size (22% for a grain size of 1 mm) and that most helium can be stored at grain boundaries for relatively small grain sizes (≤ 290 μm and ≤ 10 μm for segregation factors of $1/10^{-5}$ and $1/0.0025$, respectively). As a consequence, bulk diffusivities can be significantly higher than lattice diffusivities. Although our study cannot be applied directly to the lower mantle, the similar storage sites and diffusion mechanisms are expected in lower mantle silicates if high pressure does not inhibit diffusion.

1. Introduction

From the earliest time of the Earth, it is now accepted that our planet has undergone a large-scale differentiation by producing an iron-rich core, a silicate mantle and a gaseous atmosphere. A proto-crust was also created by the partial melting of the Earth's mantle. Melt extraction during continental crust growth has removed major amounts of highly incompatible elements from the mantle. This is evidenced by the low trace element content and relatively homogeneous strontium (Sr) and neodymium (Nd) isotopic ratios of Mid-Oceanic Ridge Basalts (MORBs),

showing that MORBs sample a depleted mantle, homogenized by convection (e.g., Hofmann 2003). However, most Ocean Island Basalts (OIBs) show significant enrichments in incompatible trace elements (e.g., Eisele et al. 2002; McDonough and Sun 1995; Plank and Langmuir 1998; Rudnick 1995; Su 2002; White and Duncan 1996; Woodhead 1996), that cannot be explained solely by a low degree of partial melting of the OIB mantle source. Moreover, the isotopic ratios of OIBs are variable and systematically different from those of MORBs (e.g., OIBs typically contain less radiogenic neodymium and hafnium and more radiogenic strontium, Hofmann 2003). Therefore, OIBs sample a

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different mantle reservoir than MORBs (Hofmann and Hart 1978; Hofmann et al. 1978). Mantle isotopic compositions led to the definition of end-member compositions (or mantle components), which can be explained by a mixture between: (i) a depleted mantle, (ii) an enriched mantle and (iii) a recycled component such as recycled lithosphere during subduction process (Farley et al. 1992; Hanan and Graham 1996; Hart et al. 1992; White 1985; Zindler and Hart 1986). Moreover, some OIBs are very rich in primordial elements, suggesting the existence of a primitive mantle at depth, isolated from the bulk mantle and not homogenized by mantle convection (i.e. Xe isotopic heterogeneities are at least as old as 4.45 Ga; Mukhopadhyay 2012). The isolation of such primordial material in the deep Earth has inhibited the loss of incompatible and volatile elements by degassing and/or partial melting processes.

Noble gases are excellent tracers of geochemical heterogeneities since they are both incompatible and volatile elements, and are not easily recycled into the mantle by subduction as a subduction barrier exists (Moreira and Raquin 2007; Staudacher and Allègre 1988), an exception may be cold subduction zones and for heavy noble gases only (Smye et al. 2017). Noble gases are also highly mobile during rock deformation (Bauer et al. 2016). In particular, helium (He) diffusion constrains the minimum size of mantle heterogeneities since it is the fastest diffusing species in mantle silicates with the possible exception of H (i.e. Cherniak & Watson, 2012; Demouchy and Mackwell 2006). According to the characteristic distance of He diffusion at high pressure, temperature and timescale relevant for Earth's mantle, a threshold size of several kilometres is obtained, below which heterogeneities would have been homogenized by diffusion (Hart et al. 2008). Moreover, He isotopic compositions constrain mantle source components. Compared to MORBs, OIB $^3\text{He}/^4\text{He}$ values vary greatly (Allègre et al. 1995; Class et al. 2005; Graham et al. 1992; Hilton et al. 1999; Kurz et al. 1982; Kurz et al. 1983, 2004; Moreira et al. 1999; Parai et al. 2009; Stuart et al. 2003): $^3\text{He}/^4\text{He}$ values lower than MORB are attributed to U-rich (high U/ ^3He) recycled materials, in the OIB source, whereas those higher than MORB could result from a relatively undegassed (low U/ ^3He) source (Moreira & Kurz, 2013) or the incorporation of outer core material into mantle plumes (Bouhifd et al. 2013; Porcelli and Halliday 2001). Nevertheless, the exact settings and characteristics of these heterogeneities remain open questions.

To broaden the use of He as a key tracer of mantle heterogeneities, it is necessary to identify He storage sites and constrain the mechanisms of He diffusion in mantle minerals and rocks. In monocrystalline olivine, it is assumed that He is preferentially stored in atomic defects within the structure such as 0-dimension defects (i.e., vacancies and interstitials), 1-dimension defects (i.e., dislocations), 2-dimensions defects (i.e., interfaces and subgrain boundaries) and 3-dimensions defects (i.e., inclusions). In polycrystalline olivine, high temperature partitioning experiments have shown that incompatible elements are preferentially stored at grain interfaces (Hiraga et al. 2003, 2004, 2007). This observation was recently confirmed by atom probe tomography performed on natural samples (for grain boundaries, see Bachhav et al. 2015; for storage along dislocations, see Piazzolo et al. 2016). Concerning He, higher He concentration have been measured in fine-grained ultramylonites compared to coarser-grained mylonitic peridotites (Kurz et al. 2009; Recanati et al. 2012), suggesting a link between He concentration and grain size (thus surface area), which could significantly influence bulk He diffusion in the upper mantle.

It is generally accepted that noble gas diffusion in mantle materials is controlled by lattice diffusion as in a monocrystalline olivine. However, more complex diffusion processes are predicted in polycrystalline materials, with several diffusion regimes operating as a function of temperature: surface, grain boundary and lattice diffusion (see Chakraborty, 2008 for a review; Harrison 1961). If incompatible elements are preferentially stored at grain boundaries, they should be significantly affected by grain boundary diffusion in addition to lattice diffusion. This mechanism of diffusion has been modelled (Dohmen and

Chakraborty 2003; Lee 1995; Maier 2004) and reported experimentally for major and trace elements in metals (e.g., Herzog et al., 1993; Herzog and Divinski 2003; Herzog and Mishin 2005; Mishin and Herzog 1999), in carbonates (e.g., Farver and Yund 1996, 1998; McCaig et al. 2007), and in simple oxides, such as NiO (Joesten 1991) and MgO (Hayden and Watson, 2007; Van Orman et al. 2003). For grain boundary diffusion in silicates, only few experimental data are available: OH⁻ in muscovite and quartz (Lasaga 1986), O in silicates (Farver and Yund 1991, 1995; Joesten 1991), H in spinel (Demouchy, 2010b) and H, Mg and Mg-Fe in olivine (Demouchy 2010a; Dohmen 2008; Farver et al. 1994). More recently, bulk diffusion of He and Ar in experimentally doped polycrystalline olivine has been reported to occur via two diffusion processes corresponding to (i) low temperature grain boundary diffusion characterized by low activation energies ($< 100 \text{ kJ}\cdot\text{mol}^{-1}$) and (ii) high temperature lattice diffusion characterized by higher activation energies ($\geq 100 \text{ kJ}\cdot\text{mol}^{-1}$; Burnard et al. 2015). Nonetheless, He storage and diffusion in polycrystalline olivine remains underconstrained, especially regarding grain boundaries as the sole dominant preferential storage site (i.e., quantification of the segregation factor between grain boundaries and the olivine lattice remains to be quantified). Finally, few experimental data are available on He diffusion in polycrystalline olivine, and the parameters that control grain boundary diffusion (i.e., activation energy, transition temperature), are very poorly quantified. In this study, we report diffusion experiments designed to better understand and quantify He distribution and diffusion in polycrystalline olivine, and discuss consequences for helium mobility at larger scale.

2. Material and methods

2.1. Starting material

Polycrystalline olivine was prepared by sintering at (i) $0.30 \pm 0.01 \text{ GPa}$ with a gas-medium HP-HT apparatus (also called Paterson press) at Geosciences Montpellier (GM, University of Montpellier, France) and (ii) $1.00 \pm 0.02 \text{ GPa}$ with a piston cylinder apparatus at the Laboratoire Magmas et Volcans (LMV, Université Clermont Auvergne, Clermont-Ferrand, France). In both cases, inclusion-free gem-like olivines $\text{Mg}_{0.904}\text{Fe}_{0.092}\text{Ni}_{0.0035}\text{Cr}_{0.0003}\text{SiO}_4$ from San Carlos (Arizona, USA) were carefully selected, crushed, and sieved to obtain powders of different grain sizes.

A Helos BF laser diffraction granulometer with a He-Ne red laser was used to determine powder grain sizes at Laboratoire Interdisciplinaire des Environnements Continentaux (LIEC, Université de Lorraine, Nancy, France). Olivine powder was introduced into a water tank before being agitated and sent to a R5 lens (determination range of 2.5–875 μm , focal depth of 500 mm) or R2 lens (determination range of 0.25–87.5 μm , focal depth of 50 mm) for laser diffraction. A second analysis was performed after 60 s of ultrasound in the tank to avoid grain agglomeration that may lead to an overestimation of grain sizes. The resulting olivine grain size ranges were 0.5 to 100 μm (average grain size around 14 μm) for RDPC1, 0.3 to 80 μm (average grain size around 14 μm) for RDPP1, and 40 to 340 μm (average grain size around 140 μm) for RDPC150.

For sintering at 0.3 GPa in static conditions, olivine powder was first cold pressed into a 20-mm-long Ni sleeve (inner diameter of 9.45 mm, outer diameter of 9.95 mm). The sleeve was closed at each end by a Ni disc (100 μm thick) using a small amount of superglue to lock it in place. The sleeve was then encapsulated in an iron jacket with zirconia pistons, alumina pistons and alumina spacers (Mackwell et al. 1985). Then, the sample was annealed for 5 h at $300 \pm 10 \text{ MPa}$ and $1250 \pm 25 \text{ }^\circ\text{C}$ using a gas medium high pressure high temperature apparatus (see Paterson 1990) to sinter the olivine grains. Independent calibration of the furnace shows that temperature varies by $< 2 \text{ }^\circ\text{C}$ along the Ni sleeve. After sintering, the sample was quenched at high pressure before releasing the isostatic Ar pressure.

For sintering at 1 GPa, olivine powder was loaded into a 10-mm-

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