



Experimental study of trace element release during ultrahigh-pressure serpentinite dehydration



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ABSTRACT

Subduction of serpentinite is envisaged to play a key role in volatile and element recycling at convergent plate margins, but there is currently little known about the composition of the fluid phase(s) released by devolatilisation of deeply subducted serpentinite. We have performed a series of ultrahigh pressure experiments to examine the phase relations and fluid compositions produced by reaction of a natural serpentinite under sub-arc conditions. We employ a novel technique of forming synthetic fluid inclusions in olivine at run conditions to preserve samples of experimental fluids for subsequent analysis. Our experiments confirm that the breakdown of antigorite and chlorite are the most important fluid-producing reactions from serpentinite at sub-arc depths. For our low CaO/Al₂O₃ peridotitic composition at 3.5 to 4.0 GPa we find that clinopyroxene reacts out below 750 °C and chlorite breaks down progressively between 700 and 800 °C to form garnet harzburgite.

Raman analysis of synthetic fluid inclusions indicates that all experiments contained a single aqueous fluid phase, which – together with a lack of textural or mineralogical evidence for hydrous melting – indicates that the water-saturated solidus for our starting composition is above 900 °C at 4.0 GPa. Element concentrations in the fluid for three experiments were determined in situ via laser ablation ICP-MS of individual fluid inclusions. In general, the fluids are enriched in trace elements compared to the bulk starting material, but particularly so for Li, B, LILE, LREE, and U. Chlorite dehydration fluids have high Li/B, LREE/HREE and Ce/Y due to retention of some B in olivine, and retention of Y and HREE in garnet. Our results indicate that fluids produced by serpentinite dehydration at sub-arc depths may carry some of the slab-derived trace elements required for arc magmatism, and may fractionate key trace element ratios in the dehydrated residues, which in turn may ultimately contribute to the geochemical heterogeneity of mantle-derived magmas.

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1. Introduction

The liberation of H₂O and trace elements from subducting oceanic plates is fundamental to the generation of arc magmas and is likely to have profound influence on the composition of material returned into the deep mantle. There is growing evidence that hydrated ultramafic rocks play a significant – if not dominant – role in deep transport and release of volatiles in subduction zones (Ulmer and Trommsdorff, 1995; Peacock, 2001; Kerrick, 2002; Scambelluri et al., 2004a; van Keken et al., 2011). Alteration of mantle peridotite by aqueous fluids prior to subduction, or at fore-arc depths during subduction (Kerrick, 2002; Savov

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et al., 2007), primarily forms serpentinite, that can retain ~13 wt.% H₂O (Ulmer and Trommsdorff, 1995), as well as high concentrations of Cl, As, B, Sb, Cs, Sr and U (Scambelluri et al., 2004a, 2004b; Savov et al., 2007; Deschamps et al., 2010; Kodolányi et al., 2012; Spandler and Pirard, 2013). A large fraction of the volatiles in subducted serpentinite is predicted to be released as aqueous fluid at sub-arc depths (Ulmer and Trommsdorff, 1995; van Keken et al., 2011). These fluids may be crucial for generation of intermediate-depth intraslab earthquakes (e.g., Peacock, 2001), and there is compelling evidence for a serpentinite-derived fluid component in some arc lavas (Singer et al., 2007; John et al., 2011; Scambelluri and Tonarini, 2012; Spandler and Pirard, 2013).

Nevertheless, we currently lack comprehensive knowledge of the geochemical effects of deep serpentinite dehydration. Known rock sequences that preserve evidence of high-pressure serpentinite dehydration are restricted to two localities: the Cerro del

Almirez Complex of Southern Spain (Trommsdorff et al., 1998; López Sánchez-Vizcaíno et al., 2005) and Cima di Gagnone in the Swiss Central Alps (Scambelluri et al., 2012, 2014). In both cases, constraints on the composition of antigorite dehydration fluids have been obtained from analysis of remnant fluid inclusions preserved within garnet and/or olivine and orthopyroxene that formed as products of the dehydration reactions (Scambelluri et al., 2004a, 2004b, 2012). However, interpretation of these results is complicated by the complex metamorphic history of these rock packages and potential interaction with external fluids (Scambelluri et al., 2004a, 2014; López Sánchez-Vizcaíno et al., 2005). Moreover, peak metamorphic conditions of Cerro del Almirez and Cima di Gagnone correspond to 50 to 80 km depth (Evans and Trommsdorff, 1978; Padrón-Navarta et al., 2010; Scambelluri et al., 2014), which represent fore-arc rather than sub-arc conditions.

Ultrahigh pressure (UHP) experiments have proved effective in advancing our knowledge of the nature and composition of deep subduction-zone fluids (e.g., Manning, 2004; Kessel et al., 2005; Hermann and Spandler, 2008; Skora and Blundy, 2010), but relatively few studies have targeted ultramafic components of the slab in part due to the experimental difficulties of recovering, analysing, and interpreting the fluid phase(s) present at run conditions. These issues are far from trivial, and, for example, are the root cause of ongoing controversy over the position of the wet peridotite solidus (Green et al., 2010; Till et al., 2012). To enable chemical analysis of experimental fluids, Tenthorey and Hermann (2004) and Dvir et al. (2011) ran experiments combining ultramafic starting material with layers of diamond aggregates to separate fluids from solid phases on experiment quenching. An alternative technique first devised by Roedder and Kopp (1975) for low-pressure hydrothermal systems, and later employed by Spandler et al. (2007) and Bali et al. (2013) for high-pressure experiments, involves synthesis of fluid inclusions in prefractured crystals of quartz or olivine that heal during experimental annealing. Preservation of experimental fluid samples by this technique avoids many of the complications associated with experiment quenching and allows analysis of experimental fluids using a range of techniques (e.g., Raman, FTIR, LA-ICP-MS).

In this paper we describe the technique for studying fluids in ultramafic systems by synthesis of fluid inclusions in forsteritic olivine during UHP experiments. We investigate the phase relations and fluid compositions of a natural serpentinite at conditions directly relevant to the sub-arc environment of subduction zones. The experiments were designed to focus on the breakdown of antigorite and chlorite, which arguably represent the most important devolatilisation reactions in subduction zones. The results inform on the fractionation of trace elements during slab dehydration, which is needed to understand trace element transfer to arc magmas as well as the composition of residual slab materials that may ultimately form geochemically distinct mantle reservoirs.

2. Experimental starting materials

We chose to use natural high-pressure serpentinite as our starting material for all experiments, as this material is representative of subducted serpentinite undergoing dehydration at sub-arc depths. We did not dope the experiments with high levels of trace elements, with the exception of Rb that serves as the internal standard for quantitative LA-ICP-MS analysis of fluid inclusions (see below). Working with the low trace element concentrations indigenous to ultramafic rocks presents challenges for precise elemental analysis of experimental products, but avoids unwanted artefacts of experimental design such as saturation in trace element rich accessory phases, or element partitioning behaviour that does not conform to Henry's law.

After petrographic and geochemical screening of a range of available serpentinite samples, we chose a sample labelled ALM127 from Cerro del Almirez (Spain) as our starting material. The geochemistry of ALM127 is outlined in Electronic Appendix 1. The sample is a medium-grained foliated serpentinite comprising 92% antigorite (as confirmed by XRD analysis), 4% metamorphic diopside, 4% magnetite, trace amounts of fine Ni-Fe sulfide, and very rare tiny apatite grains; it is free of carbonate. ALM127 has a peridotitic bulk composition (Table 1) with low TiO₂, CaO, Na₂O and K₂O concentrations and high MgO compared to lherzolitic compositions used in previous experimental studies (Electronic Appendix 1). The low CaO/Al₂O₃ of ALM127 is atypical of mantle peridotite, but is a feature of serpentinites formed in association with rodingites in abyssal settings (e.g., Mével, 2003). ALM127 is enriched in B and Cs, and slightly depleted in Sr, Ba, HFSE and REE compared to primitive mantle (McDonough and Sun, 1995), and is comparable to other serpentinites equilibrated under eclogite-facies conditions (Scambelluri et al., 2004a, 2004b). Following the methods of Spandler et al. (2007), a small amount (0.5% of the total mix) of 10–50 μm pyrope seeds were added to powdered ALM127 to avoid potential mineral metastability due to sluggish garnet nucleation in experiments (e.g., Green, 1977).

All of the experiments were loaded with powdered ALM127 mix, together with prefractured chips of San Carlos olivine (Fo⁹¹), which serve as traps for fluid inclusions formed during the experiments. San Carlos olivine has low concentrations of the trace elements of interest (Electronic Appendix 1; Spandler and O'Neill, 2010) and is expected to be in approximate equilibrium for major elements with ALM127 at experimental conditions. Gem quality, inclusion-free San Carlos olivine crystals were sliced into ~1 mm thick rectangular chips, heated to 460 °C for 15 min, and then quenched in water to generate microfractures. The chips were subsequently etched with 2% HF solution for 15 min and then cleaned in deionised water in an ultrasonic bath.

The third ingredient for the experiments was a 0.6 wt.% MgCl₂ aqueous solution with 200 μg g⁻¹ Rb. The solution was prepared from milliQ water with Mg and Rb added as chlorides. Subsequent analysis of the solution by ICP-MS confirmed the doping level of Mg and Rb, and revealed all other trace element concentrations to be below 0.01 μg g⁻¹, except for 0.32 μg g⁻¹ Ca (Electronic Appendix 1). Rubidium is present in very low concentrations in ALM127 (Table 1), and is expected to partition completely into the fluid during the experiments (e.g., Tenthorey and Hermann, 2004), so addition of this solution in controlled amounts to the experiments allows for quantification of fluid inclusion analysis by LA-ICP-MS using Rb concentration as the internal standard (Electronic Appendix 2).

3. Experimental techniques

All experiments were conducted in 12.7 mm end-loaded piston cylinder apparatus at the Research School of Earth Sciences, Australian National University. Each experimental charge was loaded with precisely measured quantities of ALM127 mix, fractured San Carlos chips, and Mg-Rb-Cl₂ solution (Table 2). Experiments at or below 800 °C used 6.5 mm diameter silver capsules designed to be cold sealed, as outlined by Hack and Mavrogenes (2006a) and Spandler et al. (2007). Cold sealing avoids evaporation of loaded solutions that may occur with standard capsule sealing by welding. Due to the low melting point of silver, experiments over 800 °C used standard 3.5 mm gold tube capsules that were sealed by conventional arc welding. To avoid uncontrolled devolatilisation from the capsule during welding, the loaded capsules were dried in a furnace overnight at 120 °C prior to final welding. This procedure is expected to evaporate the MgCl₂ solution to leave all MgCl₂ and RbCl as salt precipitates in the capsule. These salts redissolve

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