Formation of Amphibole-Bearing Peridotite and Amphibole-Bearing Pyroxenite Through Hydrous Melt-Peridotite Reaction and In Situ Crystallization: An Experimental Study

Chunguang Wang1,2  ©, Yan Liang2 ©, and Wenliang Xu1 ©

1College of Earth Sciences, Jilin University, Changchun, China, 2Department of Earth, Environmental and Planetary Sciences, Brown University, Providence, RI, USA

Abstract  Amphibole is a common hydrous mineral in mantle rocks. To better understand processes leading to the formation of amphibole-bearing peridotites and pyroxenites in the lithospheric mantle, we conducted experiments by juxtaposing a lherzolite against hydrous basaltic melts in Au-Pd capsules. Two melts were examined, a basaltic andesite and a basalt, each containing 4 wt% of water. The experiments were run at 1200°C and 1 GPa for 3 or 12 h, and then cooled to 880°C and 0.8 GPa over 49 h. The reaction at 1200°C produced a melt-bearing orthopyroxenite-dunite sequence. Crystallization of the partially reacted melts during cooling lead to the formation of an amphibole-bearing gabbronorite-orthopyroxenite-peridotite sequence. Orthopyroxene in the peridotite and orthopyroxenite has a poikilitic texture enclosing olivines and spinels. Amphibole in the peridotite occurs interstitial to olivine, orthopyroxene, clinopyroxene, and spinel. Comparisons of texture and mineral compositions in the experimental products with those from field observations allow a better understanding of hydrous melt-rock reaction in the lithospheric mantle. Amphibole-bearing pyroxenite veins (or dikes) can be formed in the lithospheric mantle or at the crust-mantle boundary by interaction between hydrous melt and peridotite and subsequent crystallization. Hornblendite or amphibole gabbronorite can be formed in the veins when the flux of hydrous melt is high. Differences in reacting melt and peridotite compositions are responsible for the variation in amphibole composition in mantle xenoliths from different tectonic settings. The extent of melt-rock reaction is a factor that control amphibole composition across the amphibole-bearing vein and the host peridotite.

1. Introduction

Amphibole is commonly observed in mantle xenoliths from a range of geological settings, such as arcs (e.g., Bénard & Ionov, 2013; Ishimaru et al., 2007), ocean islands (e.g., Clague & Bohrson, 1991; Moine et al., 2001), continental margins (Francis, 1976a, 1976b), and intracontinental regions (e.g., Dautria et al., 1987; O’Reilly et al., 1991; Xu et al., 2010). Typical lithologies include amphibole-bearing peridotite, amphibole-bearing pyroxenite, and hornblende (sometimes referred as amphibolite, e.g., Dautria et al., 1987). Amphibole in these mantle lithologies have varying occurrences including individual grain in peridotite, fine-grained aggregate in peridotite and amphibole-bearing vein hosted by peridotite, and oikocryst enclosing anhydrous minerals in hornblende. Different occurrences of amphibole often coexist and show inter-lithological variations in texture and mineral compositions. Petrologic and geochemical studies attributed the formation of amphibole-bearing in mantle lithologies to hydrous melt or fluid metasomatism in the lithospheric mantle (e.g., Bénard & Ionov, 2013; Dautria et al., 1987). Since amphibole contains water, halogens, and accommodates a range of trace elements, studying the processes of melt- or fluid-rock reaction during mantle metasomatism that leads to the formation of amphibole-bearing lithologies is important for understanding volatile cycling and trace element distribution in the lithospheric mantle (e.g., Bénard et al., 2017a; Shimizu et al., 2017).

The stability of amphibole in the upper mantle has been extensively studied through laboratory phase equilibrium experiments (Fumagalli et al., 2009, 2017; D. H. Green et al., 2014; Grove et al., 2006; Mallik et al., 2015; Mandler & Grove, 2016; Mengel & Green, 1989; Niida & Green, 1999; Wallace & Green, 1991). By comparison, the formation of amphibole by melt-rock reaction has only been examined in a few studies.
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In a recent melt-peridotite reaction study (Wang et al., 2016), we reacted a layer of lherzolite or dunite with a layer of water-added (4 wt%) basaltic andesite in Au-Pd lined graphite capsules at 1200°C and 1 GPa. An orthopyroxenite-dunite sequence is produced by hydrous melt and peridotite reaction, and quenched amphibole crystals are observed in the interstitial melts around the orthopyroxenite-dunite boundary (Figure 1a). Experimental results of Wang et al. (2016) indicate that the interstitial melts must be near amphibole saturation and imply that an amphibole-bearing pyroxenite-peridotite sequence could be formed by hydrous melt and peridotite reaction followed by crystallization.

Intrigued by the above mentioned experimental studies, we expand the isothermal hydrous melt-peridotite reaction experiments of Wang et al. (2016) by considering two hydrous melts (basaltic andesite and basalt) and by adding a step cooling of the experimental charges (to 880°C and 0.8 GPa) after the isothermal
reaction at 1200°C and 1 GPa (Figure 1b). The cooling promotes in situ crystallization of minerals from the interstitial melts. Such thermal path corresponds to the process that a relatively hot hydrous melt infiltrates the lithospheric mantle and the metasomatized region cools progressively due to heat conduction. The primary aim of this study is to better understand the formation of amphibole-bearing pyroxenite and peridotite assemblage. Texture, mineralogy, and mineral compositional variation of the experimental results, combined with those from previous experimental investigations and data from field observations, provide insight into processes of hydrous melt metasomatism in the lithospheric mantle.

2. Methods

Hydrous melt-peridotite reaction and in situ crystallization experiments were conducted using the reaction couple method and a 19.1-mm piston-cylinder apparatus at Brown University. Reaction couples were made by juxtaposing a powdered starting basalt layer against a powdered spinel lherzolite layer at a length ratio of ∼5:3 in Au$_{75}$-Pd$_{25}$ lined graphite capsules. Each experiment consists of four stages: pressurization to 1 GPa at room temperature, a dwell at 1200°C and 1 GPa, a subsequent step cooling to 880°C and 0.8 GPa, and a final dwell at 880°C and 0.8 GPa. The reaction at 1200°C and 1 GPa simulates reaction of lithospheric mantle with a hydrous melt, which is not designated to reach equilibrium. The subsequent cooling allows crystallizations of partly reacted melts, making the final product more relevant to field observations. The starting melt and mineral compositions are listed in Table 1. Run conditions for each experiment are listed in Table 2, and the temperature-time paths are shown in Figure 1b.

2.1. Starting Materials

The starting lherzolite (sample WFY-2) was obtained by mixing optically clean olivine (60 wt%), orthopyroxene (23 wt%), clinopyroxene (12 wt%), and spinel (5 wt%) from a disaggregated fertile spinel lherzolite.

| Oxide (wt%) | Basaltic andesite MONT147 | MORB D44A | Spinel lherzolite WFY-2$^b$ |
|-------------|---------------------------|------------|----------------------------|
| SiO$_2$     | 54.86                     | 50.13      | 40.25                      |
| TiO$_2$     | 0.77                      | 1.25       | 0.01                       |
| Al$_2$O$_3$ | 18.78                     | 16.31      | 0.04                       |
| Cr$_2$O$_3$ | –                         | –          | 0.03                       |
| FeO$^a$     | 8.96                      | 8.85       | 9.82                       |
| MnO         | 0.21                      | 0.17       | 0.12                       |
| MgO         | 3.66                      | 8.72       | 48.41                      |
| CaO         | 8.80                      | 12.50      | 0.09                       |
| Na$_2$O     | 3.08                      | 2.63       | 0.00                       |
| K$_2$O      | 0.78                      | 0.02       | 0.00                       |
| NiO         | –                         | –          | 0.38                       |
| P$_2$O$_5$  | 0.14                      | 0.08       | –                          |
| Total       | 100.04                    | 100.66     | 99.15                      |
| Mg#         | 42.13                     | 63.72      | 89.78                      |
| Cr#         | –                         | –          | –                          |

Mg#, 100 × Mg/(Mg + Fe), atomic ratio; Cr#, 100 × Cr/(Cr + Al), atomic ratio; Opx, orthopyroxene; Cpx, clinopyroxene.  
$^a$Total Fe as FeO.  
$^b$WFY-2 consists of 60 wt% olivine, 23 wt% opx, 12 wt% cpx, and 5 wt% spinel.
xenolith entrained by alkali basalt from Huinan, China. The starting materials for reacting melts are a basaltic andesite (sample Mont147) from Montserrat and a mid-ocean ridge basalt (MORB, sample D44A) from Southern East Pacific Rise to which we added 4 wt% water during sample loading. We use the two water-added basaltic compositions as the starting materials for the following reason. Hydrous metasomatic melts in the lithospheric mantle are derived from partial melting of recycled crustal materials that have transformed into eclogite or garnet pyroxenite; melt-peridotite reaction is driven by the difference in major-element chemistry between the melt and the peridotite; major element compositions of the two starting basaltic compositions are within the range defined by melts obtained from eclogite and garnet pyroxenite partial melting experiments (see Figure 2 in Wang et al., 2013 and Figure S1 in Wang et al., 2020). The starting lherzolite and basaltic andesite (with 4 wt% water) were used in isothermal hydrous melt-rock reaction experiments of Wang et al. (2016). The MORB was used in anhydrous reactive crystallization experiments of Tursack and Liang (2012) and Saper and Liang (2014). Experiments from these isothermal or anhydrous melt-rock reaction studies serve as benchmarks or references for understanding the processes of hydrous melt-peridotite reaction and amphibole formation in the lithospheric mantle. The starting samples were ground separately in ethanol using an agate mortar and pestle for 2–4 h and stored at 110°C before use.

2.2. Experimental Procedures

The furnace assembly consists of a Au_{75}-Pd_{25} lined graphite capsule in a MgO sleeve sandwiched between crushable MgO spacers in straight-walled graphite heater, Pyrex tubing, and NaCl sleeve. The cylindrical graphite capsule (6.5 mm OD, 7 mm long) was lined with a Au-Pd inner capsule (3 mm OD, 2.8 mm ID, 5 mm long). To make a reaction couple, we first packed the peridotite powder in the lower part of the Au-Pd capsule and filled the remaining part of the capsule with the basaltic andesite or MORB powder. We then added deionized water using a micro syringe and sealed the Au-Pd capsule using a micro arc welder immediately after water loading. We weighed the capsule before and after each loading to ensure that the water-to-basalt weight ratio is around 4:96 (i.e., 4 wt% water). The arc welder has a thin tungsten electrode (0.6 mm in diameter) and operates under a stream of protective argon gas. It allows pinpoint welding with a very small heat affected area. The weight loss due to the welding is less than 0.5% (evaporation of water and Au-Pd alloy). Finally, we loaded the sealed Au-Pd capsule in the graphite capsule and capped the graphite capsule with a 0.5 mm thick graphite lid. The furnace assembly and capsule used in this study are the same as those used in experiments BAH8, BAH11, and BATH14 in Wang et al. (2016). Oxygen fugacity of the experiments is maintained at 0.5–1.5 logarithmic unit below the quartz-fayalite-magnetite buffer, estimated
using the method of Barr and Grove (2010), within the range defined by mantle xenoliths from supra-subduction zone and intracontinental lithospheric mantles (Frost & McCammon, 2008).

To conduct a reaction experiment, the charge was cold pressurized to 1 GPa, followed by heating to 1200°C at 75°C/min while maintaining pressure. The run was held at 1200°C and 1 GPa for 3 h for BAH11 and MBH12 and 12 h for BAH13. The run was then cooled to 880°C over 7 steps in 49 h (Figure 1b). Each step cooling was performed at a rate of 1°C/min, followed by a 4 or 8 h dwell at the specific temperature. The 7-step cooling was applied to resolve the limitation of temperature controller of our piston-cylinder apparatus which uses °C/min in its ramp rate. The average cooling rate of the seven steps is 6.5°C/h (Table 2). The pressure, which was not adjusted during cooling, gradually decreased to 0.8 GPa at the final dwell (880°C). At the end of experiment, the charge was quenched by cutting the power supply while maintaining pressure at approximately 0.8 GPa. The heating and step cooling were operated through a Eurotherm controller, and temperature was measured using a W5Re5-W25Re25 thermocouple with an uncertainty of 10°C (Morgan & Liang, 2005). No friction correction was applied to the pressure. The retrieved experimental charge was checked under a binocular microscope to ensure no crack in the Au-Pd capsule. The capsule was then mounted in epoxy and polished gradually using a 600-mesh SiC polishing paper in water until the cylindrical axis was exposed. During polishing, the exposed surface was soaked in epoxy and stored in a vacuum.

Figure 2. Back-scattered electron (BSE) images showing the results of the reactive crystallization experiments. (a) BAH13 (hydrous basaltic andesite vs. lherzolite, 12 h reaction). (b) MBH12 (hydrous MORB vs. lherzolite, 3 h reaction). Ol, olivine; Opx, orthopyroxene; Cpx, clinopyroxene; D-cpx, disseminated clinopyroxene; Sp, spinel; Amp, amphibole; Pl, plagioclase; Opx-ite, orthopyroxenite.
chamber for four times, so that cracks formed during quench were puttied. The exposed surface was finally polished to 1 μm finish for electron microprobe analysis.

Iron-loss is a potential issue in hydrous experiments using Au-Pd capsules. Wang et al. (2016) addressed this issue by comparing hydrous melt-rock reaction experiments (their runs FBHNL3/4, FBH4, and FBH11) with the same starting materials and run condition (1300°C, 1 GPa, 3 h) using Pt, Au-Pd, and Fe-presaturated Au-Pd capsules. They did not observe any difference in reaction kinetics and reaction products among the three experiments. Given the low temperatures (1200°–880°C), large capsule size (and sample-to-metal volume ratio), and short dwell time at 1200°C (3 or 12 h) of the reactive crystallization experiments reported in this study, we expect the Fe-loss in these runs to be small, which should have a negligible effect on the reaction kinetics and very small effect on the Mg# (100 × Mg/[Mg + Fe], in molar) of crystallized minerals.

2.3. Microprobe Analysis

Collection of back-scattered electron (BSE) images and X-ray concentration maps and measurements of mineral compositions were carried out using a Cameca SX-100 electron microprobe at Brown University. An accelerating voltage of 20 kV and a beam current of 25 nA were used to collect X-ray concentration maps. A 10 μm diameter beam was used for glass analyses and a focused beam for mineral analyses. Accelerating voltage and beam current were 15 kV and 10 nA for glass analyses, 20 kV and 15 nA for plagioclase analyses, and 20 kV and 25 nA for olivine, pyroxene, amphibole, and spinel analyses, respectively. Sodium was analyzed first with counting times of 10 s for peak and 5 s for background to minimize volatilization. Counting times for other elements were 20–30 s for peak, and 10–15 s for background on each side of the peak. Natural standards were used for calibration. Uncertainties (±1σ) based on repeated measurements of standards used in the probe analysis are less than 0.16 wt% for oxides in minerals and melt (see Table S1 for details).

3. Results

Three hydrous melt-peridotite reaction and crystallization experiments were conducted using the two starting melts. Experiments BAH11 and BAH13 use the 4 wt% water-added basaltic andesite as the starting melt, and experiment MBH12 uses the 4 wt% water-added MORB as the starting melt. The experiments were built on three melt-peridotite reaction studies using similar setups and starting compositions that are either hydrous but isothermal (Wang et al., 2016) or step-cooling but anhydrous (Saper & Liang, 2014; Tursack & Liang, 2012). Lithological and mineralogical features of the experiments are summarized in Table 2. Representative back-scattered electron images of the run products are displayed in Figures 2 and 3. Mineral compositions are presented in Figures 4 and 5. Additional BSE images and mineral composition profiles (Figures S1–S9) can be found in the Supporting Information, and electron microprobe data (Tables S1–S4) are archived at Mendeley Data (http://dx.doi.org/10.17632/895f6y8chd.3). We did not measure water contents in melts because the small sizes of glass pools and quench cracks in them. Judging from the presence of amphibole and vesicle in the experiments and a mass balance calculation (Table S5), we conclude that these capsules retained a significant fraction of water in the system and our reacting melts were indeed hydrous at run conditions.

3.1. Lithologies and Textures

After reaction at 1200°C and 1 GPa and step cooling to 880°C and 0.8 GPa, each experimental charge consists of three distinct lithological zones: an amphibole gabbronorite, an amphibole-bearing peridotite, and an amphibole-bearing orthopyroxenite (100–300 μm thick) that separates the former two lithological zones (Figures 2 and S1). The amphibole gabbronorite, which is absent in the isothermal experiments of Tursack and Liang (2012) and Wang et al. (2016), consists of orthopyroxene, clinopyroxene, amphibole, plagioclase, and quenched melt (Figure 2). Orthopyroxenes are subhedral and often surrounded by large euhedral amphiboles; some orthopyroxenes are present as inclusions in the central part of amphiboles (Figures 2, S1, and S2). Clinopyroxene grains are anhedral and small. They are generally enclosed around rims of the
large amphiboles, and some clinopyroxenes in gabbronorite from run MBH12 are dispersed in melts or enclosed in plagioclases (Figure 2b). Plagioclases are tabular-shaped with modal abundance decrease from runs BAH11 and BAH13 to run MBH12. Ulvöspinel is observed in the gabbronorite section in experiment BAH11 (Figure S1), a byproduct of reactive crystallization at the reducing condition.

The amphibole-bearing peridotite consists of olivine, orthopyroxene, clinopyroxene, spinel, and amphibole (Figure 3). Olivines are round in shape and small in size (<60 μm in diameter). Orthopyroxenes are large (up to 350 μm in length) and core-to-rim zoned in the X-ray concentration map and BSE images (Figures 3 and S2). The orthopyroxenes enclose small rounded olivines and spinels forming a poikilitic texture. Clinopyroxenes are embayed in shape and low in modal abundance (<7 vol%). Clinopyroxene is more abundant in the peridotite when the reacting melt is the hydrous MORB (run MBH12, Figure 3c) than those when the reacting melt is the hydrous basaltic andesite (BAH11 and BAH13, Figures 3a and 3b). Some spinels have euhedral darker rims in the BSE images (inset in Figure 3b). Amphibole is crystallized interstitial to other minerals throughout the peridotite. Empty vesicles are present where small olivine grains are abundant, but absent in the poikilitic orthopyroxenes. Plagioclase and interstitial melt are not observed in the peridotite region, in contrast to the anhydrous experiments of Saper and Liang (2014).

The amphibole-bearing orthopyroxenite layer is mainly composed of large euhedral orthopyroxenes (up to 200 μm in length) (Figures 3 and S2). These orthopyroxene grains contain small olivine and spinel inclusions, similar to those formed in the isothermal hydrous reaction experiment BAH8 from Wang et al. (2016). Clinopyroxene and amphibole are observed around the gabbronorite-orthopyroxenite boundary, and interstitial melt is observed around the orthopyroxenite-peridotite interface. The orthopyroxenite layer in run BAH13 (12 h reaction at 1200°C) is wider than that in BAH11 (3 h reaction at 1200°C) (Figures 3a and
suggesting the role of kinetics in orthopyroxene growth. Interestingly, on the gabbronorite side of the orthopyroxenite in MBH12, orthopyroxenes are partially dissolved and replaced by a layer of clinopyroxene (Figures 3c and S2), suggesting a reaction relationship between the two minerals.

### 3.2. Variations in Mineral Composition

Compositional variations in minerals from the hydrous melt-peridotite reaction experiments are observed both within grain and across lithological regions. Grain-scale variation exists in orthopyroxene, amphibole, plagioclase, and spinel. Cores of large orthopyroxene and amphibole grains have higher Mg# than the rims (Figures S3 and S4). Plagioclase displays oscillatory zoning with an overall decreasing in An# (100 × Ca/[Ca + Na], in molar) and Al2O3 content from core to rim (Figure S5). Spinel grains exhibit core-to-rim zoning in the BSE images (inset in Figure 3b). The darker rims likely have lower Cr# (100 × Cr/[Cr + Al], in molar) and higher Mg# than the lighter cores. These core-to-rim variations are consistent with crystallization trends.

Figures 4 and 5 display compositional variations of orthopyroxene and amphibole across the gabbronorite, orthopyroxenite, and peridotite sequence. (Compositional profiles for other minerals can be found in Fig-
Zero distance is the approximate position of the gabbronorite-orthopyroxenite interface at the end of the run. This interface is also the boundary separating the crystalline region (melt-bearing orthopyroxenite and dunite) and the crystal-free reacting melt during the reaction at 1200°C (Figure 1a). Mineral compositions are distinct across the interface for both orthopyroxene and amphibole, although the trends are somewhat obscured by core-to-rim variations. The differences are more pronounced in run BAH11 (basaltic andesite as reacting melt, 3 h reaction at 1200°C, cyan symbols) than the other two runs (blue and red symbols). For longer reaction time (BAH13) and more primitive reacting melt composition (MBH12), compositions of amphiboles and orthopyroxenes in gabbronorite become more similar to those in the orthopyroxenite and peridotite. SiO$_2$ content in amphibole and Mg# in orthopyroxene and amphibole from the gabbronorite layer are lower than those from the peridotite and orthopyroxenite layers, whereas TiO$_2$ and MnO contents are higher in the gabbronorite. Na$_2$O in amphibole increases from gabbronorite to peridotite in run BAH11 (Figure 5h). Orthopyroxene and amphibole compositions from each experiment exhibit large variabilities at the same distance in the compositional profiles, consistent with the core-to-rim compositional variations (Figures 4 and 5).

4. Discussion

4.1. Processes of Hydrous Melt and Peridotite Reaction Followed by Crystallization

Lithology and mineralogy resulting from melt-rock reaction are controlled by near-liquidus phase relationship of the reacting melt and reaction kinetics. Reaction between the hydrous basaltic andesite and lherzo-
lite at 1200°C and 1 GPa produced a high-porosity orthopyroxenite-dunite sequence (Figure 1a). The dunite is formed by melting of lherzolite that is infiltrated by water and hydrous melt, and the orthopyroxenite is formed by reaction between olivine and the interface melt with orthopyroxene as the liquidus phase (Wang et al., 2016, Figure 6a). The hydrous melting also produces Cr-rich spinel. The infiltration takes place at the onset of experiment, and is a common phenomenon of melt-rock reaction experiment involving powdered starting materials. After the initial infiltration, mass transfer across the reaction couple is facilitated by diffusion-limited dissolution, precipitation, and re-precipitation similar to those observed in previous melt-peridotite reaction experiments (e.g., Morgan & Liang, 2003, 2005).

Oversaturation of orthopyroxene in the reacted melt and interstitial melts leads to the crystallization of orthopyroxene upon subsequent cooling, forming the chemically zoned orthopyroxene oikocrysts in the peridotite (Figures 3, 6b, S2 and S3). Spinel is also saturated in the peridotite interstitial melt after the reaction stage, and spinel grains grow during cooling, forming the darker rims in the BSE images (inset in Figure 3b). Clinopyroxene and amphibole are near-liquidus phases of interstitial melts in the peridotite and orthopyroxenite. The stability of clinopyroxene on the liquidus increases from hydrous basaltic andesite to hydrous basalt (e.g., Weaver et al., 2011). This explains the more abundant clinopyroxene in run MBH12 than in runs BAH11 and BAH13, and the replacement of orthopyroxene by clinopyroxene around the gabbronorite-orthopyroxenite interface in MBH12 (Figures 3, 6c, and S2). Orthopyroxene and clinopyroxene are surrounded by amphiboles (Figures 2, 3, 6d, and S2), suggesting the later crystallization of amphibole. In the anhydrous reactive crystallization experiments, clinopyroxene is the mineral crystallized during cooling (Tursack & Liang, 2012). The presence of water in reacting melt in the hydrous reactive crystallization experiments stabilizes amphibole which crystallizes throughout the peridotite layer (Figure 3). Crystallization of amphibole is at the expenses of clinopyroxene and hydrous melt, resulting in the embayed clinopyroxenes in the peridotite and the small clinopyroxene inclusions near the rims of large amphiboles in the gabbronorite (Figures 3, 6d, and S2). The euhedral shape of plagioclase grains in the gabbronorite region suggests that plagioclase is the last phase of crystallization. The empty vesicles may represent the exsolution of water from the hydrous silicate melt upon quench.

4.2. Comparison of Melt-Rock Reaction Experiments

The most remarkable difference between the hydrous reactive crystallization experiments presented here and the anhydrous ones reported by Tursack and Liang (2012) and Saper and Liang (2014) is the formation

![Figure 6](image_url)
of amphibole-bearing orthopyroxenite and amphibole-bearing peridotite in the hydrous experiments (Figures 2 and 3). The two anhydrous reactive crystallization studies (reaction at 1 GPa and 1300°C–1320°C, followed by a step cooling to 1200°C or 1050°C) used the same starting basalt (sample D44A) as the hydrous run MBH12 but without addition of water. With clinopyroxene on the liquidus of the reacting melt and interstitial melt after the reaction stage, these anhydrous experiments produced a gabbro-wehrlite or a gabbro-wehrlite-peridotite sequence.

Figures 7–11 compare mineral compositions obtained from the present experiments with data from previous experimental studies (melt-rock reactions and phase equilibria). Spinel is present throughout the orthopyroxenite and peridotite layers in the hydrous reactive crystallization experiments (Figure 3). This is different from the anhydrous cases in which the abundance of spinel decreases dramatically toward the reaction front (Saper & Liang, 2014; Tursack & Liang, 2012). Such difference is most likely because that the presence of water in melt depresses crystallization temperatures of silicate minerals but has little effect on the crystallization temperature of spinel (Sisson & Grove, 1993). Cr# is negatively correlated with Mg# in spinel (Figure 7a). Starting with high Cr# at 1200°C (e.g., the isothermal run BAH8 in Figure 7), continuous crystallization of spinel with falling temperature depletes Cr2O3 in the residual melt, resulting in the negative correlation between spinel Cr# and Mg# and the core-to-rim variations observed in BSE images (inset in Figures 3b and S2). TiO2 in spinel from the reaction experiments in this study (0.19–0.40 wt%) is lower than that from the alkali basalt-peridotite reaction experiments of Tursack and Liang (2012), which is mainly attributed to the lower TiO2 in starting MORB (1.25 wt%) and basaltic andesite (0.77 wt%) used in the present experiments than the alkali basalt (2.49 wt%) used in Tursack and Liang (2012).

Olivine from hydrous reaction experiments is relatively high in Mg#, compared with that from the anhydrous reaction experiments (Figure 8). This is consistent with the high melting degree of the lherzolite at the isothermal hydrous melt-rock reaction stage that forms a dunite (Figure 1a, Wang et al., 2016). Variations of NiO and MnO with Mg# in olivine follow similar trends in the hydrous and anhydrous reaction experiments (Figures 8a and 8b), whereas Cr2O3 and CaO in olivines from the hydrous experiments are lower than those from the anhydrous experiments (Figures 8c and 8d). The low olivine Cr2O3 content is likely due to the formation of high-Cr spinels at the reaction stage of the hydrous experiments. Since most olivines
from the hydrous experiments were formed at the reaction stage at 1200°C (i.e., the dunite in BAH8), the CaO content in olivine is generally lower than that from the anhydrous experiments which were reacted at a higher temperature (1300°C or 1320°C). The solubility of CaO in olivine decreases with the decrease of temperature (e.g., Adams & Bishop, 1982).

Pyroxene compositions of the hydrous reactive crystallization experiments expand the variation trends of the anhydrous ones to lower Mg#'s (<70, Figure 9). The low Mg#'s are in part attributed to the evolved starting melt composition (basaltic andesite) used in the BAH experiments and the low final temperature. CaO is lower in orthopyroxene and higher in clinopyroxene from the hydrous experiments than those from the anhydrous experiments (Figures 9a and 9b). This is attributed to the temperature effect on CaO content in the pyroxene solvus (e.g., Lindsley, 1983; Nickel & Brey, 1984; Nickel et al., 1985). TiO₂ and Na₂O in clinopyroxene from experiments using basaltic andesite or MORB as starting melt are lower than those using alkali basalt (Figures 9d and 9f), reflecting the effect of reacting melt composition on the composition of clinopyroxene.

Amphibole is crystallized later than pyroxenes in the hydrous reactive crystallization experiments. We tested the extent of equilibrium between melt and amphibole using the composition-temperature relationship of Shimizu et al. (2017). As is shown in Figure S10, amphiboles in the orthopyroxenite and peridotite regions are close to chemical equilibrium with melt at 880°C. In addition, the temperature- and pressure-sensitive species Na₂O in amphibole (e.g., Niida & Green, 1999) from the present experiments are within the range defined by phase equilibrium experiments at 880°C and 0.8 GPa (Figures 10a and 10b). The above lines of evidence indicate that the amphiboles are close to equilibrium with melt at the final stage.

The hydrous reactive crystallization experiments produce amphiboles with higher TiO₂ and low Mg# than the reaction experiments of Sen and Dunn (1994), due to the high starting melt/peridotite ratio (and thus, the evolved bulk compositions). In terms of amphibole TiO₂ and Mg#, the present reaction experiments are comparable to phase equilibrium experiments on basalt to dacite compositions, and experiments of Sen and
Dunn (1994) are comparable to phase equilibrium experiments on mantle compositions (Figures 10c–10f). A slight difference in amphibole Na₂O can be observed between the BAH and MBH experiments (Figures 10a and 10b), which can be attributed to the different starting compositions used (Table 1). Amphiboles from the hydrous reactive crystallization experiments have higher Al₂O₃ and lower Cr₂O₃ than those from the phase equilibrium experiments (Figures 10g–10j), mostly due to the formation of Cr-rich spinels at the reaction stage.

Plagioclase from the reaction experiments is relatively low in An# (<67, Figure 11) and overlaps plagioclase composition from phase equilibrium experiments run at pressures ≥0.6 GPa (Borghini et al., 2010; Fumagalli et al., 2017). Composition of plagioclase from the hydrous reactive crystallization experiments mimics that from the anhydrous ones of Saper and Liang (2014), except for the lower MgO for the hydrous runs. This is likely caused by the late crystallization of plagioclase that is suppressed by the presence of water (e.g., Borghini et al., 2010; Namur et al., 2012; Takagi et al., 2005).

5. Geological Applications

The experiments reported here and those in the previous melt-rock reaction and phase equilibrium studies underscore the importance of water and reacting melt and peridotite compositions in determining lithological and mineral compositional variations produced by melt-rock reaction. Because of the simplified
experimental setup, the mass transfer mechanism may be different between melt-peridotite interaction in the laboratory experiments and those in nature (e.g., diffusion vs. advection). Extensive water infiltration into the powdered starting peridotite leads to a higher degree of partial melting and faster reaction. The rate of cooling in the experiments is considerably faster than that takes place in nature. Nevertheless, the reaction kinetics should be similar between the laboratory experiments and those in nature, because it is determined by chemical potential gradients of the major components and phase relationships in the hydrous basalt-peridotite system. In this section, we compare lithology and mineral compositions obtained from laboratory melt-rock reaction and phase equilibrium experiments with those from field studies of amphibole-bearing peridotites (Figures 7–10, and 13) to assess the effect of hydrous melt-peridotite interaction.

**Figure 10.** Plots of oxide abundances (in wt%, on water-free basis) and Mg# in amphibole against temperature and pressure for melt-peridotite reaction experiments from this study, Sen and Dunn (1994, SD94) and phase equilibrium experiments on mantle compositions (Phase equilibria 1: Fumagalli et al., 2009, 2017; D. H. Green et al., 2014; Grove et al., 2006; Mallik et al., 2015; Mandler & Grove, 2016; Mengel & Green, 1989; Niida & Green, 1999; Wallace & Green, 1991) and basalt to dacite compositions (Phase equilibria 2: Alonso-Perez et al., 2009; Blatter et al., 2013; Costa et al., 2004; Feig et al., 2006; Foden & Green, 1992; Gardner et al., 1995; T. H. Green and Pearson, 1985; Grove et al., 1997, 2003; Holtz et al., 2004; Kawamoto, 1996; Latourrette et al., 1995; Mercer & Johnston, 2008; Moore & Carmichael, 1998; Nandedkar et al., 2014, 2016; Nekvasil et al., 2004; Nicholls & Harris, 1980; Parat et al., 2008; Pichavant et al., 2002; Pietranik et al., 2009; Pilet et al., 2010; Prouteau & Scaillet, 2003; Sato et al., 2005). Compositional range of amphiboles in each experiment from this study are shown as bars at temperature and pressure of the final stage (880°C and 0.8 GPa).
in modifying the mineralogy and mineral chemistry of the lithospheric mantle. Petrologic features of the amphibole-bearing mantle rocks from literatures for comparison are briefly summarized in Table 3.

5.1. Formation of Amphibole-Bearing Rocks in the Lithosphere

We have demonstrated that hydrous basalt-peridotite reaction and in situ crystallization can produce spatially associated amphibole-bearing garnorite-orthopyroxenite-peridotite sequence at conditions examined. One field example is from the lower crust section of the Ivrea-Vermbano Zone in Italian Alps described in details by Antonicelli et al. (2020). In the deepest levels of the Ivrea Magmatic Complex, harzburgites are intruded by amphibole gabbronorite dikes, with millimeter-scale thick contact zones of orthopyroxenite toward the harzburgite and hornblende websterite toward the gabbronorite. The harzburgites are amphibole-bearing, and contain poikilitic orthopyroxenes enclosing partially dissolved olivine and minor spinel (Antonicelli et al., 2020). Amphibole, orthopyroxene, and clinopyroxene have increasing Mg#' and Cr# from gabbronorite to pyroxenites and harzburgite, and spinel have an increasing Cr# from websterite to orthopyroxenite and harzburgite. The lithological sequence, petrological features, and mineral chemical variations of the Ivrea samples are remarkably similar to those observed in the reactive crystallization experiments reported in the present study (Figures 2 and 3).

Figure 12 summarizes possible scenarios in which amphibole-bearing lithologies are formed. Hydrous melt-peridotite reaction may happen when an asthenosphere-derived hydrous melt infiltrates peridotite in the lithospheric mantle or at the crust-mantle boundary along a pressure gradient or fracture network (Figure 12a). The infiltration of the hot hydrous melt may induce a partial melting of the surrounding peridotite. Reaction between the hydrous melt and the partially molten peridotite produces a melt-bearing orthopyroxenite vein (Figure 12b, Wang et al., 2016). A new generation of orthopyroxene, clinopyroxene, and amphibole then precipitates from the interstitial melts as the metasomatized region is cooled and/or the infiltrating melt is depleted in water (by diffusional loss or change of pressure). The precipitation occurs in the melt-bearing orthopyroxenite and the partially molten peridotite, forming an amphibole-bearing pyroxenite vein in the amphibole-bearing peridotite wall-rock (Figure 12c). Poikilitic orthopyroxene enclosing olivine and spinel inclusions can be produced in the peridotite by the reactive crystallization (Figure 12c). It can be inferred that a hornblendite or amphibole gabbronorite may form at the center of pyroxenite vein when the extent of reaction or the flux of hydrous melt is high (Figure 12d). These processes are similar to those in the present reactive crystallization experiments. Hydrous melt-peridotite reaction may also happen when the hydrous melt infiltrates peridotite at a low-temperature region in the lithosphere. The infiltration dose not trigger a partial melting of the peridotite, and the reaction is less extensive. Crystallization of the partially reacted melt forms an amphibole-rich vein in the fracture with or without a thin orthopyroxene-rich layer between the vein and the peridotite wall-rock (Figure 12e). This scenario is similar to those in the low-temperature reaction experiments of Sen and Dunn (1994) and Rapp et al. (1999).

5.2. Amphiboles in Mantle Xenoliths from Different Tectonic Settings

Compositions of the supra-subduction zone lithospheric mantle and the intraplate lithospheric mantle are distinct. The supra-subduction zone lithospheric mantle has a more refractory composition than the
intraplate lithospheric mantle, and melts migrating in supra-subduction zones are higher in SiO<sub>2</sub>, Mg# and lower in TiO<sub>2</sub> than intraplate magmas (e.g., Bénard et al., 2017b). Based on the effect of bulk composition on amphibole chemistry, Coltorti et al. (2007) recognized the difference between amphiboles from supra-subduction zone lithospheric mantle (S-amphiboles) and amphiboles from intraplate lithospheric mantle (I-amphiboles). In general, S-amphiboles (light blue field in Figure 13) have higher Mg# and lower TiO<sub>2</sub> and Na<sub>2</sub>O than I-amphiboles (light red field in Figure 13), although an overlap exists. In addition to amphibole composition, TiO<sub>2</sub> in spinel and Mg#, TiO<sub>2</sub>, and Na<sub>2</sub>O in pyroxenes show corresponding differences between xenoliths from the two settings. Spinel in mantle xenoliths from supra-subduction zone are commonly lower in TiO<sub>2</sub> than those in intraplate mantle xenoliths (Figure 7). Pyroxenes in supra-subduction zone xenoliths generally have higher Mg# and lower TiO<sub>2</sub> and Na<sub>2</sub>O than those in the intraplate ones (Figure 9). It should be noted that mineral compositions obtained from the reaction experiments are more similar to those in mantle xenoliths of the I-affinity (Figures 7, 9, and 13), which is mainly due to the starting peridotite (i.e., lherzolite) used in these experiments (Table 1).

Amphiboles in harzburgite xenoliths carried by andesites from Avacha, Kamchatka have high SiO<sub>2</sub>, Mg# and low TiO<sub>2</sub>, Na<sub>2</sub>O (Bénard & Ionov, 2013; Ishimaru et al., 2007; Ionov, 2010), their compositions vary along the S-amphibole trend (Figure 13). These compositional features can be produced by reaction between the depleted lithospheric mantle and a boninitic melt which is rich in SiO<sub>2</sub>, MgO, H<sub>2</sub>O and poor in TiO<sub>2</sub> (Bénard & Ionov, 2013). Harzburgite and clinopyroxene-poor lherzolite xenoliths in diorites from Japan Island Arc.

Table 3

| Reference | Lithology or lithological assemblage | Main amp occurrence(s) | Locality |
|-----------|-------------------------------------|-------------------------|----------|
| Takahashi (1980) | Sp lherzolite | – | Japan Island Arc |
| Abe et al. (1998) | Sp harzburgite | – | Japan Island Arc |
| Blatter and Carmichael (1998) | Sp lherzolite; chr ol websterite | Interstitial grains replacing cpx | Central Mexico |
| Coltorti et al. (2007) | – | – | Japan Island Arc |
| Ishimaru et al. (2007) | Sp/chr harzburgite | Discrete; interstitial grains replacing cpx | Avacha |
| Xu et al. (2009, 2010) | Sp harzburgite; chr dunite | Interstitial grains replacing cpx; grains at margin of sp | North China Craton |
| Ionov (2010) | Sp harzburgite | In fine-grained pocket and vein | Avacha |
| Bénard and Ionov (2013) | Sp harzburgite with opx-rich and amp-rich veins | Discrete (host); disseminated grains in fine-grained pockets (host); anhedral to subhedral grains (vein) | Avacha |

Classification of supra-subduction zone (S)-amp and intraplate (I)-amp is after Coltorti et al. (2007).
the central and the eastern parts of the North China Craton contains secondary amphiboles (Xu et al., 2009, 2010). These amphiboles occur around clinopyroxenes and spinels, similar to those observed in the peridotite layer of hydrous reaction experiments from this study (Figures 3, and S2), and their compositions follow the variation trend of S-amphiboles (Figure 13). They can be formed by interaction between the refractory harzburgites and hydrous siliceous melts derived from delaminited continental crust (Xu et al., 2009, 2010). Amphibole-bearing lherzolite and pyroxenite xenoliths carried by alkali basalts from Nunivak Island, Alaska (Francis, 1976a, 1976b) contain amphibole with the I-amphibole affinity (Coltorti et al., 2007) (Figure 13). Such compositional feature of amphibole in mantle xenoliths is consistent with the regional setting of intraplate continental margin at Nunivak.

5.3. Amphiboles in Different Lithologies in Mantle Xenoliths

Results of the hydrous melt-peridotite reactive crystallization experiments exhibit variations in amphibole composition across lithological regions. The amphibole compositional variations are attributed to the variation in the amount of reacting melt relative to the peridotite component, which essentially reflects the variation in the extent of reaction. Harzburgite xenoliths from Avacha (Bénard & Ionov, 2013) contain amphiboles in the host and two types of veins. Amphiboles in the host harzburgite either form fine-grained pockets with clinopyroxene, orthopyroxene, and spinel or occur as coarse grains. Type 1 veins are orthopyroxene-rich with a mantle origin. The contact between the vein and the host harzburgite is either straight (Type 1A) or irregular (Type 1B). The irregular margin of Type 1B veins is similar to the orthopyroxene-peridotite contact in the hydrous melt-peridotite reactive crystallization experiments. Type 2 veins are amphibole-rich and were likely crystallized from the carrier andesite, because they can be traced back to selvages at xenolith rims. Amphiboles in the host harzburgite and Type 1 veins varied along the S-amphibole trend (Figures 13a–13c). Amphiboles in Type 1B veins have lower Mg# and higher Na$_2$O than amphiboles in Type 1A veins (Figures 13a and 13c). The straight margin and high amphibole Mg# of Type 1A veins were interpreted to be products of injection of a high-Mg boninitic melt into fractures of subsolidus peridotite with a less extent of melt-peridotite reaction (Bénard & Ionov, 2012). By contrast, the irregular margin and relatively low Mg# in amphibole suggest that Type 1B veins were products of grain-scale percolation of the boninitic melt through the partially molten host peridotite. The extent of melt-peridotite reaction is larger for the Type 1B veins than that for the Type 1A veins. Amphiboles in Type 2 veins and selvage amphiboles have low Cr# and vary along the Mg# vs. TiO$_2$ trend similar to that established by the hydrous melt-peridotite reaction experiments and phase equilibrium experiments on basalt to dacite compositions (Figures 13d and 13e). This is consistent with the interpretation that these amphiboles were products of reaction between xenoliths and carrier andesite that infiltrated into fractures of xenoliths during ascent or storage in crustal magma chambers (Bénard & Ionov, 2013). Different occurrences of amphiboles are also observed in a suite of mantle xenoliths and megacrysts in basanites and nephelinites from Ahaggar, southern Algeria (Dautria et al., 1987). Amphiboles are present in hornblende xenoliths, a lherzolite-hornblende composite xenolith,
Figure 13. Compositions of amphiboles (oxides in wt%, on water-free basis) from the melt-rock reaction and phase equilibrium experiments and amphibole-bearing mantle xenoliths. The experimental data are the same as in Figure 10. The division of supra-subduction zone (S)-amphibole and intraplate (I)-amphibole is after Coltrotti et al. (2007). Light blue hexagons are S-amphiboles in xenoliths from the North China Craton (Xu et al., 2009, 2010). Bluish squares are S-amphiboles in different lithological regions of xenoliths from Avacha, and light green squares are amphiboles formed by host magma-xenolith reactive crystallization (Bénard & Ionov, 2013). Red crosses are I-amphiboles in xenoliths from Nunivak (Francis, 1976a, 1976b). Dark red and orange diamonds are I-amphiboles in different lithological regions of xenoliths from Ahaggar, and yellow diamonds are amphibole megacrysts in the host alkali basalts (Dautria et al., 1987). Other S- and I-amphibole data are shown as light blue and light red fields, respectively, with sources listed in Table 3. Calculation of chemical formula and division of compositional boundaries (in g) follow Leake et al. (1997). See text for discussion.
and as megacrysts in the host basalt. In the composite xenolith, amphibole abundance increase toward the hornblendite section where it has a poikilitic texture enclosing chadacrysts of anhydrous minerals, similar to that observed in the peridotite region in our experiments (Figure 3). In terms of compositional variations, amphiboles in the hornblendites are lower in Mg# and higher in TiO₂ (Figures 13a and 13b) than those in the peridotites, similar to the trends observed in the melt-rock reaction experiments. Amphiboles in hornblendites have similar compositions to amphibole megacrysts in the host lava (Figure 13). The variations in occurrence and composition suggest that the amphibole-bearing lithologies were formed by melt-peridotite reactive crystallization; the hornblendites were crystallized at regions where reacting melt was concentrated (Dautria et al., 1987).

6. Conclusions

The role of hydrous melt-peridotite reaction and subsequent crystallization in the formation of amphibole-bearing mantle rocks is examined by reacting a spinel lherzolite with a hydrous basaltic andesite and a hydrous basalt (with 4 wt% water in the melts) in Au-Pd capsules using the reaction couple method. The experiments include a reaction at 1200°C and 1 GPa and a further reaction/crystallization through a series of step cooling to 880°C and 0.8 GPa. Results of the hydrous melt-peridotite reaction experiments together with those conducted under anhydrous conditions are used to deduce processes related to the formation of amphibole in mantle rocks. The main conclusions of this study are as follows:

1. Reaction between hydrous basaltic melt and lherzolite followed by in situ crystallization produces an amphibole-bearing gabbronorite-orthopyroxenite-peridotite sequence.
2. A melt-bearing orthopyroxenite-dunite sequence is formed in the reaction at high temperature (1200°C in this study). Crystallization of orthopyroxene continues at the early stages of cooling, followed by clinopyroxene, and finally amphibole in the peridotite and orthopyroxene.
3. Lithology, texture, and mineral compositional variation of run products formed by the hydrous melt-peridotite reactive crystallization experiments are similar to the hornblendite, amphibole-bearing gabbronorite-pyroxenite, and amphibole-bearing pyroxenite veins in amphibole-bearing peridotites observed in mantle xenoliths and magmatic complexes. This underscores the importance of melt-peridotite reaction and in situ crystallization in the formation of amphiboles in the lithosphere.
4. Variation in composition of amphiboles in the lithospheric mantle from different tectonic settings (subduction zone vs. intraplate) is attributed to different compositions of the metasomatic melt and the peridotite. Amphibole compositional variation in spatially accompanying amphibole-bearing lithological assemblage is mainly controlled by the extent of reaction.

Data Availability Statement

Electron microprobe data are archived at Mendeley Data (http://dx.doi.org/10.17632/895f6y8chd.3).

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