Aqueous fluid connectivity in pyrope aggregates: water transport into the deep mantle by a subducted oceanic crust without any hydrous minerals

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Abstract

The effect of pressure and temperature on the dihedral angles of aqueous fluid in a pyrope matrix was investigated. Experiments were performed on an H_2O–pyrope system in a multianvil apparatus over the pressure and temperature ranges of 4–13 GPa and 900–1200°C, respectively. The dihedral angle of the fluid in contact with the pyrope exhibited a significant change at pressures around 8–9 GPa. The dihedral angles increased with increasing pressure up to 9 GPa. At pressures above 9 GPa, the dihedral angles were greater than 60° at temperatures below 1000°C. Therefore, the efficient percolation of aqueous fluid in a pyrope matrix is not feasible in the upper mantle and the transition zone. The fluid released from the breakdown reactions of the hydrous minerals lawsonite and phengite exists in the oceanic crust, which mainly consists of garnet in the upper mantle and transition zone. We conclude that a part of the aqueous fluid released from the hydrous minerals may be retained in the subducted oceanic crust, and transferred into the deep mantle by the subduction process.
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1. Introduction

Owing to their ability to influence the melting

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behavior [1–4] and physical properties of mantle rocks, mantle fluids have been a longstanding topic of interest among geoscientists. For example, these fluids reduce the mechanical strength and modify the ionic diffusion and electric properties of mantle rocks [5–8]. Over the past 30 years, investigations of fluid behavior in the mantle have shed light on the dynamics and the chemical evolution of the Earth. The stability of hydrous minerals has been widely studied at high pressures and temperatures [9]. It is commonly believed that a significant amount of water could be re-

cycled into the mantle at subduction zones, where hydrated materials, altered Mid-Oceanic Ridge Basalts (MORBs), and sediments, are subducting into the mantle [10–12]. However, a part of the water in the slab is released as a result of the breakdown of some hydrous phases in these materials. If the released water percolates through the basaltic crust, it can hydrate the overlying mantle wedge and produce hydrous minerals such as dense hydrous magnesium silicates [11,13], and can be retained in the nominally anhydrous minerals, olivine, wadsleyite, and ringwoodite. Lawsonite is an important hydrous mineral at depths of only 80–300 km in a normal basaltic (N-MORB) layer of the slab, and there is no stable hydrous phase at depths greater than 300 km [11,14]. In the case of alkali-rich basalt, a small amount of phengite can appear as the hydrous mineral [15]. If the released water from lawsonite and phengite in the MORB layer cannot flow on the grain edge, it would be trapped in the subducted oceanic crust and then transported into the deep mantle. On dehydration of lawsonite and phengite, the host rock of basaltic composition consists mainly of majoritic garnet [16,17]. Therefore, it is important to investigate the wetting properties of garnet and aqueous fluids.

Watson and Brenan [18] and Watson et al. [19] reported the dihedral angles in a fluid–silicate system at 2 GPa. At higher pressures, up to 5 GPa, an aqueous fluid–olivine system was investigated, and the negative dependence of dihedral angles upon pressure was confirmed [19–21]. This means that the fluid forms an interconnected network in the upper mantle. Recently, many workers have investigated the dihedral angles of the near-equilibrium geometry of C–H–O fluids in monomineral aggregates of quartz [18,22–24], calcite [25], clinopyroxene [26] and anorthite [27]. In order to assess the effect of pressure on the wetting ability of pyrope garnet ($Mg_3Al_2Si_3O_{12}$) and thus the feasibility of percolation as the water transportation process in the slab, we have performed experiments over the pressure range 4–13 GPa on a pyrope–H_2O composition. The results make it possible to show the fate of water derived from the breakdown of hydrous minerals in the slab.

2. Experimental methods

The starting materials were prepared by mixing MgO, Al_2O_3, SiO_2 and $Mg(OH)_2$. Prior to mixing, reagent grade MgO, SiO_2 and Al_2O_3 powders were heated in a furnace at 1400°C for 1 h, and $Mg(OH)_2$ powder was heated at 120°C for 24 h. The H_2O content in the starting materials was about 4 wt%, controlled by the ratio of MgO/ $Mg(OH)_2$. The mixtures were ground in an agate mortar to grain sizes less than 10 μm, and were loaded into Pt capsules (1.2 mm outside diameter, 0.1 mm wall thickness) that were sealed by welding. The typical volume of sample in the capsule was 0.5–2.0 mm^3. High-pressure experiments were carried out with a 6–8 type multianvil apparatus [11]. Tungsten carbide anvils with truncations of 8 mm edge length were separated by compressible pyrophyllite gaskets. The octahedral pressure assembly was composed of pre-cast Cr_2O_3-doped MgO. A graphite furnace inside a ZrO_2 sleeve was used as a heater, and capsules were separated from the furnace by an MgO sleeve. The temperature was measured using a W3%Re/W25%Re thermocouple, and no correction for the effect of pressure on thermocouple emf was applied. After being held at a desired pressure and temperature for the desired duration, the samples were quenched by cutting off the electric power supply, resulting in a temperature drop to < 200°C in 2–5 s. The relationship between press load and sample pressure was calibrated using the following phase transformations: Fe_2SiO_4 (α–γ) at 5.8 GPa at 1200°C; and SiO_2 (coesite–stishovite) at 9.2 GPa at 1200°C. The experimental uncertainty in pressure was within 5%. The experimental charges were mounted in epoxy resin and carefully polished to prevent the grain plucking. The samples were investigated by a scanning electron microscope (SEM). The magnification used in the SEM was about 3000× to obtain good secondary-electron photomicrographs. The photomicrographs were scanned and examined using the image analysis software NIH Image, and the apparent dihedral angles were measured to within ± 10° using the protractor provided in the analysis package [28]. However, a large number of measurements was carried out for each sample

in this study. The uncertainty of the median of the distribution of apparent angles was much smaller than the error of each measurement of the apparent angles.

3. Results

A typical section of sample quenched from texture equilibrium is shown in Fig. 1. Dark regions are clearly evident at triple junctions of grain boundaries. Some fractures were produced, even during protracted quenching. The sample was composed of pyrope crystals, which was checked using micro-focused X-ray diffraction. Small and elongated pyrope grains, which were the quench product precipitated from the fluid, appeared in the grain boundaries. We measured the dihedral angles subtended by the fluid in contact with two well-rounded crystalline faces. An added complication to the characterization of the dihedral angles arose because the measured values were only the apparent angles formed by the intersection of a three-dimensional feature with the plane of the polished section. If a large number of measurements is made, however, the median angle will be the typical dihedral angle [29]. The apparent dihedral angles for the fluid in contact with pairs of pyrope crystals are presented as a histogram in Fig. 2. The distribution of the apparent angles of our experiments is broader than that from theoretical calculations. In the previous experiments, the differences between the distributions of experiments and those of theoretical calculations were also observed, because the solid–solid and solid–liquid interfacial energies are functions of crystal orientation (e.g. [30,31]).

It was important to establish whether the textural equilibrium was attained among the run products. The results at 13 GPa and 900°C for different run durations (23 and 67 h) are shown in Fig. 2. Although a difference of the frequency distribution between the 23 and 67 h runs was observed, the median from the distribution of the apparent angles does not show any difference. This suggests that the median had reached a steady-state value that approached near-textural equilibrium after 23 h at 900°C. At temperatures higher than 900°C, a heating time of shorter duration is probably adequate to obtain the near-equilibrium texture.

Our dihedral angle measurements are summarized in Table 1. Below 7 GPa, all the measured dihedral angles were less than 60°. The dihedral angle of 60° is an important value, because the penetration of fluid along grain edges occurs if
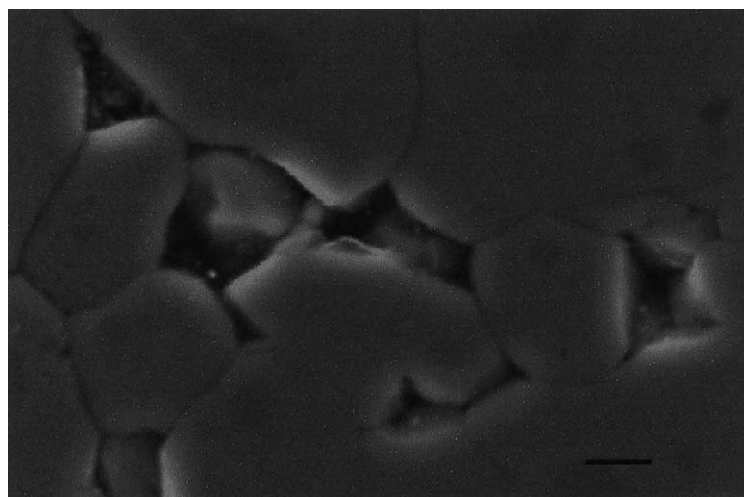

Fig. 1. Back-scattered microphotograph of run product quenched at 13 GPa and 900°C. Scale bar is 3 μm. The black patches are void space representing pores that were filled with aqueous fluid at run conditions. The fine-grained materials are considered to be quench phases from fluids.
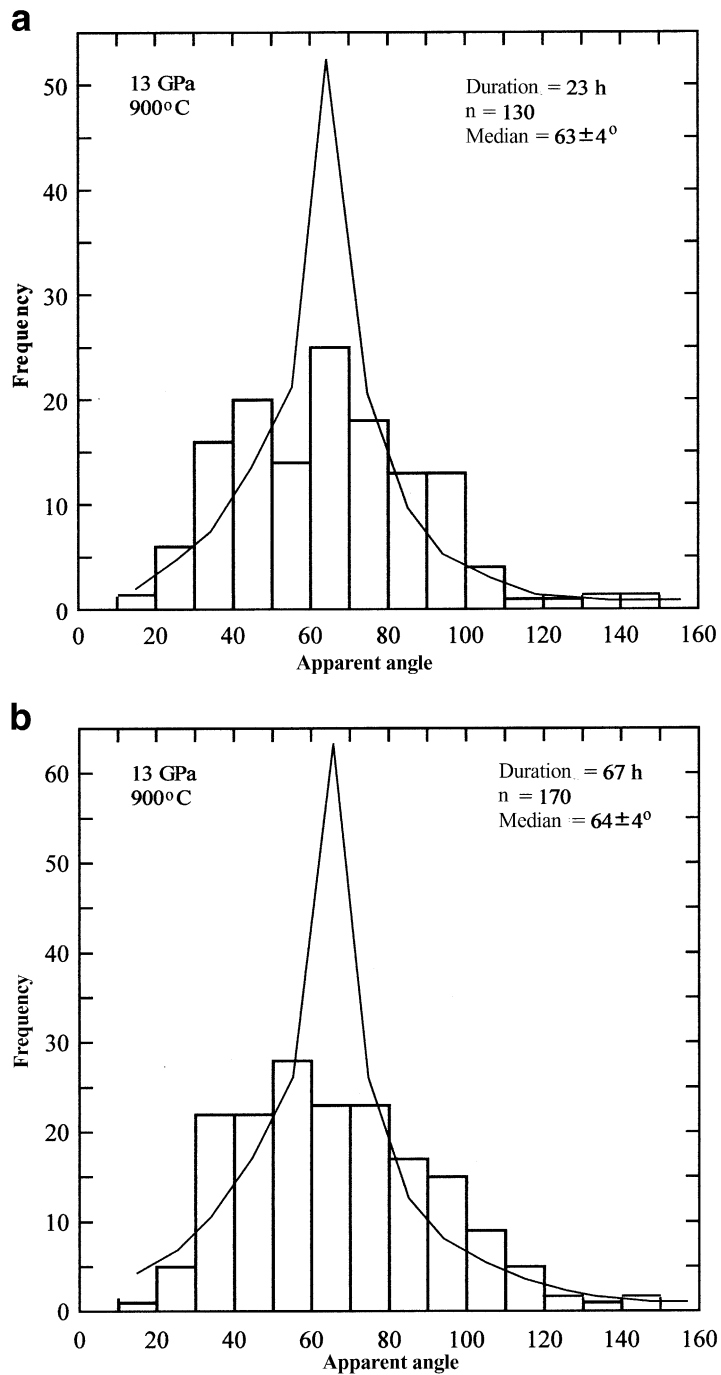
Fig. 2. Relative frequency distributions of apparent dihedral angles at fluid–pyrope–pyrope triple junctions at 13 GPa and 900°C. (a) 23 h; (b) 67 h. The theoretical distribution [45] for a monomineral, isotropic system is shown for comparison (thin lines). The error of dihedral angle is the 95% confidence interval around the median, calculated as described in [46].

Table 1
Summary of run conditions

Pressure (GPa)	Temperature (°C)	Duration (h)	Median angle (°)	N^a
4(0.2)	900(30)	75	46(7)	76
4(0.2)	1200(50)	20	39(3)	90
5(0.3)	900(30)	71	52(6)	112
6.5(0.3)	800(30)	137	n.d.[b]	
6.5(0.3)	900(30)	67	54(3)	195
6.5(0.3)	1000(40)	50	47(7)	48
7.5(0.4)	900(30)	68	59(5)	67
9(0.5)	900(30)	54	67(3)	201
9.5(0.5)	1000(40)	21	64(3)	243
9.5(0.5)	1100(40)	24	52(3)	232
13(0.7)	900(30)	23	63(4)	130
13(0.7)	900(30)	67	64(4)	170
13(0.7)	1000(40)	25	59(4)	243

Values in parentheses represent the experimental errors.
[a] Number of measurements.
[b] We could not observe the equilibrium texture.

the angle is less than 60°. The dihedral angle seems to be less than 60° at 900°C and pressures below 7 GPa, because the angles increase with decreasing temperature. At higher pressures, we could observe the boundary between the wet and the dry cases. The 60° angle appeared at temperatures of about 1000°C and pressures above 8 GPa.

Fig. 3 shows the effect of pressure on the dihedral angles. The dihedral angle gradually increases up to pressures of 9 GPa and then slightly decreases. This implies that the relationship of the interfacial energy between the pyrope crystals and aqueous fluids can change, because the dihedral angle (θ) is given by $\gamma_{mm} = 2\gamma_{fm} \cos(\theta/2)$, where γ refers to the interfacial energy per unit area of the subscripted interface, either mineral/mineral (mm) or fluid/mineral (fm). We cannot explain clearly why the relationship of the interfacial energy changes at about 9 GPa and the dihedral angle increases. One possibility is the change in the packing density of adsorbed fluid molecules on the crystal surface compared to that in the bulk [24]. These molecules seem to have a semi-solid type of structure with a density fixed by the density of possible adsorption sites. Holness [24] suggested that the change of slope in the quartz–H_2O system is caused by the presence of the adsorbed layer of H_2O on the quartz–fluid interface and the

quartz grain boundary. Another possibility is the change of chemical composition of the aqueous fluid. It has been reported that the silicate component dissolved in the aqueous fluid changes with increasing pressure [32,33]. Bureau and Keppler [34] reported that silicate melts and aqueous fluids became completely miscible in the upper mantle conditions. In their study, the com-
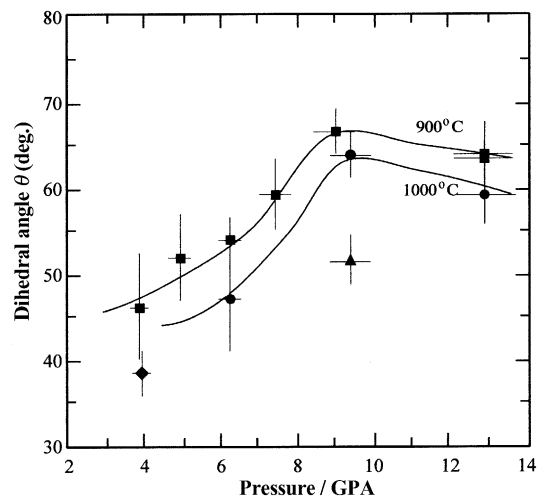

Fig. 3. Dihedral angle versus pressure diagram. The plotted value of the dihedral angle is the median angle. Squares, 900°C; circles, 1000°C; triangle, 1100°C; diamond, 1200°C. The contours are schematic.
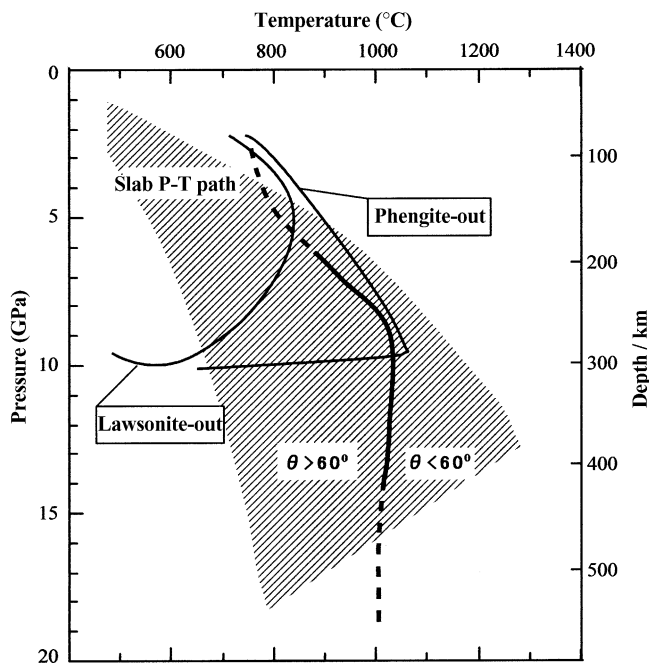
Fig. 4. 60° isopleth of the dihedral angle in fluid–pyrope–pyrope in *P–T* space. The limit of lawsonite and phengite breakdown in the system MORB–H$_2$O [11,15] is also shown. The possible *P–T* path of the upper part of the subducted oceanic crust is shown as the shaded region.

position of the fluid changed dramatically close to the miscible point between the aqueous fluid and the silicate melt. Therefore, the chemical composition of the fluid may change at the high pressures used in this study and the interfacial energy between the pyrope and the fluid may be sufficiently changed. However, it seems to need more studies to understand the dihedral angle change.

4. Discussion

It is widely recognized that the slab is transported by subduction into the transition zone of the mantle at least as far as the 660 km discontinuity, and perhaps into the lower mantle. In order to shed light on water transport in subduction zones, the water content of the basaltic layer in the slab must be considered. Previous studies reported the stability fields of hydrous phases in the normal and alkali-rich basaltic composition [10,11,15]. In the normal and alkali-rich basaltic layer, lawsonite and phengite can transfer water

up to pressures of about 10 GPa, corresponding to a depth of 300 km. After lawsonite and phengite breakdown reactions occur, there is no hydrous phase in the basaltic layer of the slab, and the basaltic composition crystallized into an assemblage of garnet+clinopyroxene+stishovite ± K-hollandite [11,35]. Therefore, in the upper mantle and transition zone, the basaltic layer consists mainly of garnet, which possesses a majoritic composition. In order to assess the interconnection properties accurately, the effect of the chemical composition of garnet should be considered in the natural rocks. If the fluid is interconnected in texturally equilibrated garnet rocks, which form the major constituent of the basaltic layer, then the water released from the breakdown reaction of lawsonite and phengite can move into the overlying mantle. On the contrary, if textural equilibrium is attained in the basaltic layer and the fluid cannot form the interconnection, there is a possibility that the subducted slab is able to retain the water.

Many workers have proposed models for the

temperature distribution within the slab (e.g. [36,37]). Based on the present results, the fate of water in the slabs using hypothetical thermal paths for the uppermost part of the slab, as shown in Fig. 4, can be determined. In the case of a relatively hot slab, which means young oceanic crust and lithosphere, with a small dip angle of subduction and a low velocity subduction rate, the water from the breakdown reaction of lawsonite can form an interconnection at pressures higher than 5 GPa, corresponding to a depth of 160 km, because the dihedral angles are nearly 60° or less than 60°. In this case, the water migrates to the overlying mantle and cannot be transferred into the deep mantle by the basaltic layer. In the case of a relatively cold slab, the breakdown reaction of lawsonite finishes at pressures of about 10 GPa, corresponding to a depth of 300 km. If texture equilibrium in the basaltic layer is attained, the dihedral angles are larger than 60° and the water cannot form an interconnection of the garnet crystals. Although a certain amount of water may move by the fracturing of the rock, some amounts of water can be retained in the basaltic layer of the slab. As garnet is a major constituent down to depths of about 800 km [17], some amounts of water may be transferred into the lower mantle by a subducted slab without any hydrous minerals.

According to the high pressure experimental results, there is a possibility that the subducted slab may stagnate at depths between the transition zone and the lower mantle because of the buoyancy of the subducted slab [17,38,39]. If the basaltic layer in the slab can retain water, the density of the slab may decrease, because the density of water could be less than those of surrounding minerals. This effect seems to enhance the buoyancy of the subducted slab. Although the porosity of the slab is small, the buoyancy is not negligible. Moreover, the stagnated slab containing the basaltic layers, which mainly consist of majoritic garnet, interrupts the water infiltration. Therefore, it may have a significant effect on the water circulation in the mantle.

The present work is still preliminary as regards the understanding of quantitative water transportation of the subducting slab, for the following reasons. First, the chemical composition of garnet may affect the dihedral angle. Natural garnets in the mantle have the majoritic composition (e.g. [11,35]). However, we have investigated a pyrope composition which does not contain the Ca, Fe, Al, Na components. Secondly, subducted oceanic crust is not a monomineral garnet rock. The presence of additional phases has been shown experimentally to have an influence on the dihedral angles in other systems [40]. Thirdly, the present study has explored the distribution of fluid under hydrostatic conditions. Recently, it has been demonstrated that the deformation affects the dihedral angles (e.g. [41–44]). In the subducted slab, the effect of the deformation may play an important role on the distribution of fluid. Finally, the activities of the fluid components are unconstrained in this study. The fluid composition was changing in the range of pressures and temperatures in the present experiments. The change of the fluid composition seems to affect the relative interfacial energies between the crystal and the fluid. Further careful experiments, especially near 60° dihedral angle, are needed to assess these issues.

Acknowledgements

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