Revision 1

Temperature and compositional dependences of H$_2$O solubility in majorite (Word Count: 5387)

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Abstract

We systematically investigated H$_2$O solubility in majorite as a function of temperature at 1670 – 2270 K under a pressure condition of 20 GPa using multi-anvil experimental techniques. The H$_2$O solubility in majorite decreases with increasing temperature. In addition, the H$_2$O content is relatively independent of the concentrations of Al$_2$O$_3$ and SiO$_2$ in majorite. Majorite can store more H$_2$O than bridgmanite in the lower mantle. Therefore, when a slab sinks into the lower mantle, hydrous melt could be produced not only by the phase transformation from ringwoodite to bridgmanite + ferropericlase near 660-km depth, but also by the majorite to bridgmanite transformation over a wide range of depth from 660 km up to ~800 km, at which depth majorite dissolves completely in bridgmanite.

Keywords: majorite; H$_2$O solubility; transition zone; hydrous melt

1 Introduction

The mantle transition zone is a potential H$_2$O reservoir in the Earth’s interior (e.g., Fei et al. 2017; Hirschmann 2006; Pearson et al. 2014) because the H$_2$O solubility of its dominant minerals, wadsleyite and ringwoodite, reaches 1 ~ 2 wt.% (Demouchy et al. 2005; Druzhbin et al. 2021; Fei and Katsura 2020a, 2021; Kohlstedt et al. 1996; Litasov et al. 2011; Purevjav et al. 2014, 2016). The H$_2$O solubility refers to the H$_2$O content of a mineral equilibrated with hydrous melt (Hirschmann et al. 2005). These values are distinctly higher than those of other major mantle minerals such as olivine, pyroxene, bridgmanite, and ferropericlase (Fei and Katsura 2020b; Férot and Bolfan-Casanova 2012; Fu et al. 2019; Guo et al. 2020; Kohlstedt et al. 1996; Litasov 2010; Liu et al. 2021; Purevjav et al. 2023; Smyth et al. 2006; Withers and Hirschmann...
Therefore, hydrous melts are expected to be formed by a back transformation at 410 km depth from wadsleyite to olivine and a forward transformation at 660 km depth from ringwoodite to bridgmanite + ferropericlase. The presence of hydrous melt may affect mantle dynamics significantly (Revenaugh and Sipkin 1994; Schmandt et al. 2014; Vinnik and Farra 2007).

Majorite, which contributes up to ~40 vol.% of the transition zone, persists in the lower mantle down to ~800 km depth until the dissolution of majorite in bridgmanite is completed (Ishii et al. 2018, 2019; Ringwood 1991; Stixrude and Lithgow-Bertelloni 2007). As majorite may contain higher amounts of H$_2$O than bridgmanite (Fu et al. 2019; Katayama et al. 2003; Liu et al. 2021), a hydrous melt may be produced by the majorite-bridgmanite transformation (Panero et al. 2020).

Nevertheless, the H$_2$O solubility of majorite is poorly constrained, while the H$_2$O solubility of wadsleyite and ringwoodite has been studied extensively. Katayama et al. (2003) reported an H$_2$O solubility of about 1200 wt. ppm in majorite, but their temperature range was limited (1670 - 1770 K). Accordingly, the temperature dependence is unknown despite the fact that the H$_2$O solubility in minerals is strongly correlated to temperature (e.g., Demouchy et al. 2005; Fei and Katsura 2020a, 2021; Litasov et al. 2011). Moreover, even though the composition of majorite can vary significantly in the Earth’s mantle (Frost 2008), the compositional dependence of H$_2$O solubility is also unknown. Therefore, a systematic study of the effects of temperature and composition on H$_2$O solubility in majorite is required to assess hydrous melting at the top of the lower mantle.

In this study, we determined the temperature and compositional dependences of H$_2$O solubility in majorite at temperatures of 1670 to 2270 K and a pressure of 20 GPa using a multi-
anvil press. Our results suggest that majorite can store 900 ~ 3000 wt. ppm H$_2$O in the deep mantle and may play an important role in the dehydration melting at the topmost lower mantle.

2 Materials and Methods

2.1 Starting material

Four compositionally different starting materials, hereafter referred to as starting materials A, B, C, and D, were prepared from SiO$_2$, FeO, Al$_2$O$_3$, Mg(OH)$_2$, and Ca(OH)$_2$ powders (Table 1). The SiO$_2$ and Al$_2$O$_3$ powders were heated at 1270 K for two hours in an ambient-pressure furnace, whereas the Mg(OH)$_2$ and Ca(OH)$_2$ powders were heated at 400 K in a vacuum oven prior to use. The five powders were weighed and then well-mixed by grinding in an agate mortar. Starting material A had a bulk composition close to pyrolite-minus-olivine (Irifune and Ringwood 1987) plus H$_2$O. Starting material B had a lower Al$_2$O$_3$ content because the Al$_2$O$_3$ component tends to be incorporated in majorite rather than melt. Starting materials C and D had higher MgO and CaO contents and lower SiO$_2$ contents to suppress the formation of stishovite in the run products. The bulk H$_2$O contents were all 12 wt.%. All the mixed powders were stored in a vacuum oven at 400 K before use.

2.2 High-pressure experiments

High-pressure experiments were carried out in a 1000-ton multi-anvil press at the Bayerisches Geoinstitut, University of Bayreuth. The starting materials were welded into Pt$_{95}$Rh$_5$ capsules with inner and outer diameters of 1.0 and 1.2 mm, respectively. The capsule lengths after completion were 1.0 - 1.2 mm, except for run H5405, in which the capsule length was 2.0 mm. The sample assembly consisted of a Cr$_2$O$_3$-doped MgO octahedral pressure medium with an
edge length of 10 mm, a ZrO$_2$ sleeve for thermal insulation, and a LaCrO$_3$ heater. One or two capsules were loaded into an MgO sleeve in the heater. The assembly was pressurized to a pressure of 20 GPa using eight tungsten carbide anvils with truncated edge lengths of 4 mm, followed by heating to target temperatures of 1670 to 2270 K with a ramp rate of 70 K/min. Temperatures were monitored using a type-D (W97Re3/W75Re25) thermocouple. The run duration at the target temperature was 20 or 24 hours.

2.3 Sample analyses

The recovered run products were embedded in epoxy resin, and cross-sections were prepared by grinding using silicon carbide grinding paper and lapping using diamond powder. The phases present in the capsules were identified using a Bruker AXS D8 Discover micro-focused X-ray diffractometer (XRD) equipped with a two-dimensional solid-state detector and a Co-K$_\alpha$ radiation source operated at 40 kV and 500 $\mu$A. An example of the XRD patterns is shown in Fig. 1a.

The textures of the samples within the capsules were observed using a scanning electron microscope (SEM) with a backscattered-electron detector (BSE). The BSE images of all recovered capsules can be found in the Supplementary Materials (Fig. S1), with an example given in Fig. 1b. The phases identified by XRD were also confirmed by compositional measurements using an energy-dispersive X-ray spectrometer (EDS).

Chemical compositions of the run products were obtained using a JEOL JXA-8200 electron probe microanalyzer (EPMA) equipped with wavelength-dispersive spectrometers (WDS) operated at an acceleration voltage of 15 kV and a beam current of 15 nA. Enstatite was used as a standard for Mg and Si, and corundum, metallic Fe, and diopside for Al, Fe, and Ca,
respectively. A focused beam was used to analyze the solid phases, while a defocused beam was used for the melt phase. The counting time for each analysis was 20 sec.

2.4 Determination of H$_2$O contents in majorite

The H$_2$O contents in majorite were determined by Fourier-transform infrared (FTIR) spectroscopy analysis using a Bruker IFS 120 high-resolution spectrometer coupled with a Bruker IR microscope. After polishing to thicknesses of 50-100 μm, unpolarized FTIR spectra were taken on the samples. Any inclusions or microcracks visible under an optical microscope were avoided in the analysis. Infrared analyses were made using a visible light source, CaF$_2$ beamsplitter, and liquid-N$_2$-cooled MCT detector. Each spectrum was collected by accumulating 100 scans at a resolution of 2 cm$^{-1}$. Two to six spectra were collected for each sample.

Peak-fitting of the FTIR spectra was performed by fitting the sum of two Gaussian functions. The first peak at a wavenumber of 3615 cm$^{-1}$ is related to hydrogen in majorite (Bolfan-Casanova et al. 2000; Liu et al. 2021), while the second at a wavenumber of 3430 cm$^{-1}$ is expected to be from inclusions or sub grain boundaries which are invisible under the optical microscope (Katayama et al. 2003; Liu et al. 2021).

Subsequently, the H$_2$O content $C_{H_2O}$ in majorite, expressed as wt. ppm H$_2$O, was calculated using the Beer-Lambert law,

$$C_{H_2O} = \frac{10^6 \times 18.02}{\varepsilon \tau \rho} \times \int H(v) dv,$$

where $H(v)$ is the infrared absorbance of the sample at a wavenumber of $v$, $\varepsilon$ is the integral molar absorption coefficient (see below), $\tau$ is the sample thickness, and $\rho$ is the density (3670 g/L). The
integration was made for the infrared absorption bands at 3615 cm\(^{-1}\) given by the peak-fitting process.

Thomas et al. (2015) reported a wavenumber-dependent infrared absorption coefficient for majorite, which was \(\varepsilon = 6000 \text{ L/(mol.cm}^2\text{)}\) at a \(\nu = 3615 \text{ cm}\(^{-1}\)\. This value was used for the calculation of \(C_{H_2O}\) in this study. The \(C_{H_2O}\) was also calculated using the calibrations reported by Bell et al. (1995) (specific to garnet) and by Paterson (1982) (for general silicate minerals and glasses) and listed in Table 2 for comparison.

3 Results and discussion

3.1 Phase assemblages of the run products

Majorite crystals with grain sizes of 50 ~ 300 µm coexisting with hydrous melts (crystallized to small grains during quenching) were found in all of the recovered capsules (Table 2, Fig. 1, Fig. S1), indicating that experiments were H\(_2\)O-saturated. Stishovite crystals appeared in the experiments with high SiO\(_2\)-content starting materials A and B, while davemaoite (CaSiO\(_3\) perovskite) appeared in the experiments with low SiO\(_2\)-content starting materials C and D. Magnesite and dense hydrous magnesium silicate phases (superhydrous phase B and phase D) were formed under relatively low-temperature conditions (1670 K) with starting material of C (Table 2).
3.2 Chemical compositions of majorite

Since compositionally different starting materials were used, the composition of majorite and melt from different runs cannot be compared directly (Table 3, Table 4). However, the composition of majorite synthesized from the same starting material (B) shows a systematic change with temperature, i.e., the Si atomic concentration increases, while the Al and Fe concentrations decrease with increasing temperature. Meanwhile, the Mg concentration increases from 1670 to 1870 K and decreases at higher temperatures, whereas the Ca concentration shows an opposite trend to Mg (Fig. 2).

The atomic concentrations of Si and Mg+Fe+Ca, where Fe is assumed to be ferrous, are plotted against the Al concentration for all samples (Fig. 2). It is found that both Mg+Fe+Ca and Si concentrations are inversely correlated with the Al concentrations, indicating the dilution of Al₂O₃ by the incorporation of the (Mg,Fe,Ca)SiO₃ component in majorite.

3.3 FTIR spectra of majorite

All of the majorite samples obtained in this study show asymmetric infrared absorption bands between 3000 and 3800 cm⁻¹ with peak positions at 3610~3120 cm⁻¹ and a broad shoulder from about 3500 to 3000 cm⁻¹ (Fig. 3). Although the shapes and peak positions in the spectra are identical in different samples, suggesting the same dominant proton incorporation mechanisms in majorite with different compositions, the peak height decreases with increasing temperature, indicating temperature-dominated water contents of the samples in this study.

By deconvolution of the spectra, the infrared absorption peak at 3615 was obtained, which could be attributed to majorite (Bolfan-Casanova et al. 2000; Liu et al. 2021). In contrast,
Thomas et al. (2015) and Katayama et al. (2003) reported infrared absorption peaks at slightly lower wavenumbers (3550 ~ 3580 cm\(^{-1}\)). The 3430 cm\(^{-1}\) peak likely arises from H\(_2\)O in inclusions, so it was not considered for the calculation of water content in this study. Even though we have followed previous studies in assigning the broad absorption band to water H\(_2\)O from inclusions (Liu et al. 2021), we cannot rule out a contribution from bonded hydroxyl in majorite. If the broad absorption band is included in the calculation of \(C_{\text{H}_2\text{O}}\), the values are roughly doubled.

3.4 Temperature dependence of H\(_2\)O solubility in majorite

Although the water solubility in majorite has already been reported previously (Bolfan-Casanova et al. 2000; Katayama et al. 2003; Panero et al. 2020; Thomas et al. 2015), all previous studies focused on a fixed temperature or composition without a systematic investigation. Since majorite coexists with hydrous melt in all runs in this study, the H\(_2\)O contents obtained in this study should represent the H\(_2\)O solubility of majorite under the corresponding pressure and temperature conditions. Based on the FTIR calibration of Thomas et al. (2015), the H\(_2\)O content in our majorite samples decreases from about 2900 to 400 wt. ppm with increasing temperature from 1670 to 2270 K, regardless of the difference in starting materials (Table 1, Fig. 4). Therefore, our results indicate a systematic decrease of H\(_2\)O solubility in majorite with temperature.

The temperature dependence of H\(_2\)O solubility can be understood thermodynamically. Majorite is equilibrated with melt by the reaction,

\[
\text{Silicate (Majorite) + H}_2\text{O (melt)} = \text{H}_2\text{O (majorite) + silicate (melt)} \tag{2}
\]
Assuming that activity is proportional to mole fraction, the change in Gibbs energy of the above reaction \( \Delta G(2) \) is,

\[
\Delta G(2) = -RT \ln \frac{c_{\text{H}_2\text{O}}^{\text{maj}} \times c_{\text{silicate}}^{\text{melt}}}{c_{\text{H}_2\text{O}}^{\text{melt}} \times c_{\text{silicate}}^{\text{maj}}} \quad (3)
\]

where \( c_{N}^{M} \) is the fraction of the component of M in the phase N. Because of \( c_{\text{silicate}}^{\text{maj}} \approx 1 \), we have,

\[
c_{\text{H}_2\text{O}}^{\text{maj}} = c_{\text{H}_2\text{O}}^{\text{melt}} \times \exp \left( -\frac{\Delta G(2)}{RT} \right) \quad (4)
\]

\( c_{\text{H}_2\text{O}}^{\text{melt}} \) should decrease with increasing temperature as the melt fraction increases (e.g., Fei 2021; Hirschmann et al. 2005), resulting in the decrease of \( \frac{c_{\text{H}_2\text{O}}^{\text{maj}}}{c_{\text{silicate}}^{\text{melt}}} \) with temperature. On the other hand, \( \text{H}_2\text{O} \) is preferentially incorporated in melt rather than solid minerals, thus, \( \Delta G(2) > 0 \).

As a result, \( c_{\text{silicate}}^{\text{melt}} \) decreases with temperature approximately following a logarithmic function. The fitting of data points gives,

\[
c_{\text{H}_2\text{O}}^{\text{maj}} = e^{9810/T + 1.90} \quad (5)
\]

where \( c_{\text{H}_2\text{O}}^{\text{maj}} \) is the \( \text{H}_2\text{O} \) content in majorite based on the FTIR calibration of Thomas et al. (2015).

We emphasize that the \( c_{\text{H}_2\text{O}} \) determined from infrared spectroscopy relies on the FTIR calibrations, i.e., the infrared absorption coefficients reported previously. Using different absorption coefficients (e.g., Bell et al. 1995; Paterson 1982) linearly affects the absolute values of \( \text{H}_2\text{O} \) solubility (Table 2). However, the temperature dependence should remain the same. We also note that the \( \text{H}_2\text{O} \) solubility in majorite determined in this study is within the experimental...
uncertainty comparable with those of Katayama et al. (2003), Thomas et al. (2015), Panero et al. (2020), and Liu et al. (2021) at identical temperatures. In the referenced studies, H$_2$O contents were determined by secondary ion mass spectrometry, electron recoil detection analysis, and infrared spectroscopy. Therefore, the different techniques for $C_{H2O}$ determination should not affect our conclusion significantly.

3.5 Compositional dependence of $C_{H2O}$ and the proton incorporation mechanism

Although different starting materials were used in this study, resulting in compositionally different majorite samples, the $C_{H2O}$ in the run products do not show systematic variations with composition (Fig. 5). This suggests that protons in majorite are incorporated into the dodecahedral and/or tetrahedral sites ($M^{2+}_{\text{VIII}} \leftrightarrow (2H^+)_{\text{VIII}}$ and $\text{Si}^{4+}_{\text{IV}} \leftrightarrow (4H^+)_{\text{IV}}$, respectively) rather than the coupled $H^+ + Al^{3+}$. This is because if $\text{Si}^{4+}$ or divalent cations ($M^{2+}$) were substituted by $H^+ + Al^{3+}$, the H$_2$O content would be positively correlated with the Al$_2$O$_3$ content, which is not the case in this study (Table 3).

The $\text{Si}^{4+}_{\text{IV}} \leftrightarrow (4H^+)_{\text{IV}}$ substitution (hydrogarnet substitution) is more likely in this study because it shows an infrared absorption band at 3630 cm$^{-1}$ in pyrope (Ackerman et al. 1983; Geiger and Rossman 2018), similar to the infrared absorption peaks for majorite (Fig. 3). The $\text{Si}^{4+}_{\text{IV}} \leftrightarrow (4H^+)_{\text{IV}}$ substitution is also suggested by first-principle calculations, which show that the $(4H^+)_{\text{IV}}$ defect is more energetically favorable than the $(2H^+)_{\text{VIII}}$ defect in MgSiO$_3$-majorite (Pigott et al., 2015).
4 Implications for the role of majorite for water storage in the deep mantle

Majorite is an important mineral in the mantle transition zone and the topmost lower mantle (Irifune and Ringwood 1987; Ringwood 1991), corresponding to a temperature condition of 1800 ~ 2000 K according to the typical mantle geotherm (Katsura 2022). Over this temperature range, majorite can contain about 900 ~ 1500 wt. ppm. This value is much lower than wadsleyite and ringwoodite, the dominant minerals in the mantle transition zone (~1.0 wt.%, Demouchy et al. 2005; Druzhbin et al. 2021; Fei and Katsura 2020, 2021; Kohlstedt et al. 1996; Litasov et al. 2011). However, H$_2$O solubility is significantly higher than in bridgmanite (<100 ~ 900 wt. ppm, Fu et al. 2019; Liu et al. 2021; Purevjav et al. 2023) and ferropericlase (<100 wt. ppm, Bolfan-Casanova et al. 2002, 2003; Litasov et al. 2010) in the lower mantle. Therefore, majorite is expected to be the major H$_2$O reservoir in the topmost lower mantle within its stability field, i.e., down to ~800 km depth before complete phase transformation to bridgmanite (Ishii et al. 2018, 2019; Ringwood 1991; Stixrude and Lithgow-Bertelloni 2007).

H$_2$O can be transported into the deep mantle by slab subduction. When slabs sink into the lower mantle, hydrous ringwoodite within the slabs transforms to bridgmanite and ferropericlase, forming a hydrous melt layer just below the 660-km discontinuity due to the contrasting H$_2$O solubilities in ringwoodite, bridgmanite, and ferropericlase (Schmandt et al. 2014). The hydrous melt should saturate majorite within the slabs. Due to the relatively low temperature of slabs (~1600 K near the 660 km discontinuity, Litasov et al. 2013; Tan et al. 2002), majorite can contain about 3000 wt. ppm H$_2$O, which is significantly higher than bridgmanite (Fu et al. 2019; Liu et al. 2021; Purevjav et al. 2023). As majorite transforms to bridgmanite gradually, a thick hydrous melt layer is expected at the top of the lower mantle depths of 660 ~ 800 km (Panero et al. 2020), which may lower the slab viscosity significantly.
Acknowledgments

We appreciate the help of H. Fisher for high-pressure cell assembly preparation and R. Njul for sample polishing. The manuscript benefited from comments and suggestions from reviewers Steve Jacobsen and Joshua Muir. D.L. is funded by the China Scholarship Council. The project was supported by the "CUG Scholar" Scientific Research Funds at China University of Geosciences (Wuhan) (Project No. 2022117) and China Postdoctoral Science Foundation (No.2023M733297). N.P. and H.F. are supported by the BGI. The BGI covered all experimental costs. The raw EPMA, FTIR and XRD data are available in the online Supporting Information and at https://zenodo.org/uploads/10073791.

Reference


Litasov, K. (2010). The influence of Al_{2}O_{3} on the H_{2}O content in periclase and ferropericlase at 25 GPa. Russian Geology and Geophysics, 51 (6), 644–649. doi:10.1016/j.rgg.2010.05.005


Table 1. Nominal compositions of starting materials A, B, C, and D.

<table>
<thead>
<tr>
<th></th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂ (wt.%)</td>
<td>51.89</td>
<td>54.58</td>
<td>42.96</td>
<td>48.08</td>
</tr>
<tr>
<td>Al₂O₃ (wt.%)</td>
<td>12.17</td>
<td>7.01</td>
<td>5.52</td>
<td>6.17</td>
</tr>
<tr>
<td>FeO (wt.%)</td>
<td>2.94</td>
<td>2.97</td>
<td>3.98</td>
<td>3.54</td>
</tr>
<tr>
<td>MgO (wt.%)</td>
<td>23.67</td>
<td>19.07</td>
<td>25.57</td>
<td>22.71</td>
</tr>
<tr>
<td>CaO (wt.%)</td>
<td>9.33</td>
<td>16.37</td>
<td>21.95</td>
<td>19.49</td>
</tr>
<tr>
<td>H₂O (wt.%)</td>
<td>11.94</td>
<td>12.11</td>
<td>12.09</td>
<td>12.17</td>
</tr>
</tbody>
</table>
Table 2. A list of run conditions, phases in the recovered capsules, and water contents in majorite based on various infrared calibrations. Mj: majorite, St: stishovite, Dm: davemaoite (CaSiO$_3$ perovskite), hy-PhB: superhydrous phase B, PhD: phase D, Mgs: magnesite.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Starting material</th>
<th>T (K)</th>
<th>Duration (h)</th>
<th>Phases present</th>
<th>$\tau$ (μm)</th>
<th>$^aC_{H_2O}$ (wt. ppm)</th>
<th>$^bC_{H_2O}$ (wt. ppm)</th>
<th>$^cC_{H_2O}$ (wt. ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>H5450</td>
<td>B</td>
<td>1670</td>
<td>24</td>
<td>Mj, St, Dm, melt</td>
<td>82</td>
<td>2591 (277)</td>
<td>2318 (248)</td>
<td>910 (92)</td>
</tr>
<tr>
<td>H5450</td>
<td>C</td>
<td>1670</td>
<td>24</td>
<td>Mj, hy-PhB, Mgs, PhD, melt</td>
<td>82</td>
<td>2913 (453)</td>
<td>2606 (405)</td>
<td>999 (122)</td>
</tr>
<tr>
<td>H5405</td>
<td>B</td>
<td>1870</td>
<td>24</td>
<td>Mj, St, melt</td>
<td>75</td>
<td>1423 (30)</td>
<td>1272 (27)</td>
<td>492 (16)</td>
</tr>
<tr>
<td>H5423</td>
<td>C</td>
<td>1870</td>
<td>20</td>
<td>Mj, Dm, melt</td>
<td>70</td>
<td>930 (157)</td>
<td>832 (140)</td>
<td>322 (64)</td>
</tr>
<tr>
<td>H5423</td>
<td>D</td>
<td>1870</td>
<td>20</td>
<td>Mj, Dm, melt</td>
<td>70</td>
<td>812 (168)</td>
<td>727 (151)</td>
<td>275 (57)</td>
</tr>
<tr>
<td>H5416</td>
<td>A</td>
<td>2070</td>
<td>20</td>
<td>Mj, St, melt</td>
<td>104</td>
<td>871 (64)</td>
<td>779 (57)</td>
<td>314 (17)</td>
</tr>
<tr>
<td>H5416</td>
<td>B</td>
<td>2070</td>
<td>20</td>
<td>Mj, St, melt</td>
<td>104</td>
<td>894 (90)</td>
<td>800 (81)</td>
<td>322 (32)</td>
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<td>H5419</td>
<td>A</td>
<td>2270</td>
<td>4</td>
<td>Mj, St, melt</td>
<td>69</td>
<td>412 (74)</td>
<td>368 (66)</td>
<td>149 (27)</td>
</tr>
<tr>
<td>H5419</td>
<td>B</td>
<td>2270</td>
<td>4</td>
<td>Mj, melt</td>
<td>70</td>
<td>636 (289)</td>
<td>569 (259)</td>
<td>248 (136)</td>
</tr>
</tbody>
</table>

$C_{H_2O}$: the H$_2$O content in majorite.

$^a$: Based on Thomas et al. (2015) calibration.

$^b$: Based on Bell et al. (1995) calibration.

$^c$: Based on Paterson (1982) calibration.
Table 3. Chemical composition of the recovered majorite analyzed by EPMA.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>No.</th>
<th>Al₂O₃ (wt.%)</th>
<th>SiO₂ (wt.%)</th>
<th>MgO (wt.%)</th>
<th>CaO (wt.%)</th>
<th>FeO (wt.%)</th>
<th>Total (wt.%)</th>
<th>Al (atomic)</th>
<th>Si (atomic)</th>
<th>Mg (atomic)</th>
<th>Ca (atomic)</th>
<th>Fe (atomic)</th>
</tr>
</thead>
<tbody>
<tr>
<td>H5450B</td>
<td>19</td>
<td>16.25 (0.3)</td>
<td>46.26 (0.2)</td>
<td>26.10 (0.29)</td>
<td>7.68 (0.32)</td>
<td>2.86 (0.11)</td>
<td>99.15 (0.38)</td>
<td>1.35 (0.02)</td>
<td>3.25 (0.01)</td>
<td>2.73 (0.02)</td>
<td>0.58 (0.03)</td>
<td>0.17 (0.01)</td>
</tr>
<tr>
<td>H5450C</td>
<td>21</td>
<td>15.88 (0.36)</td>
<td>46.10 (0.20)</td>
<td>25.41 (0.32)</td>
<td>8.40 (0.38)</td>
<td>3.71 (0.26)</td>
<td>99.49 (0.33)</td>
<td>1.32 (0.03)</td>
<td>3.25 (0.01)</td>
<td>2.67 (0.03)</td>
<td>0.63 (0.03)</td>
<td>0.22 (0.02)</td>
</tr>
<tr>
<td>H5405B</td>
<td>12</td>
<td>15.81 (0.29)</td>
<td>46.53 (0.26)</td>
<td>21.50 (0.45)</td>
<td>12.96 (0.27)</td>
<td>2.20 (0.05)</td>
<td>99.01 (0.41)</td>
<td>1.32 (0.02)</td>
<td>3.31 (0.02)</td>
<td>2.28 (0.04)</td>
<td>0.99 (0.02)</td>
<td>0.13 (0.00)</td>
</tr>
<tr>
<td>H5423C</td>
<td>19</td>
<td>9.75 (0.48)</td>
<td>50.12 (0.31)</td>
<td>26.25 (0.27)</td>
<td>10.47 (0.21)</td>
<td>3.06 (0.10)</td>
<td>99.65 (0.33)</td>
<td>0.81 (0.04)</td>
<td>3.53 (0.02)</td>
<td>2.75 (0.03)</td>
<td>0.79 (0.02)</td>
<td>0.18 (0.01)</td>
</tr>
<tr>
<td>H5423D</td>
<td>16</td>
<td>10.86 (0.50)</td>
<td>48.26 (0.40)</td>
<td>24.39 (0.41)</td>
<td>11.97 (0.35)</td>
<td>3.90 (0.14)</td>
<td>99.37 (0.24)</td>
<td>0.92 (0.04)</td>
<td>3.44 (0.02)</td>
<td>2.59 (0.04)</td>
<td>0.91 (0.03)</td>
<td>0.23 (0.01)</td>
</tr>
<tr>
<td>H5416A</td>
<td>10</td>
<td>17.54 (0.35)</td>
<td>46.38 (0.36)</td>
<td>26.96 (0.27)</td>
<td>6.34 (0.2)</td>
<td>2.48 (0.12)</td>
<td>99.70 (0.39)</td>
<td>1.44 (0.03)</td>
<td>3.22 (0.02)</td>
<td>2.79 (0.02)</td>
<td>0.47 (0.02)</td>
<td>0.14 (0.01)</td>
</tr>
<tr>
<td>H5416B</td>
<td>13</td>
<td>15.47 (0.90)</td>
<td>46.50 (0.37)</td>
<td>22.15 (0.38)</td>
<td>12.49 (0.53)</td>
<td>2.48 (0.18)</td>
<td>99.09 (0.39)</td>
<td>1.30 (0.07)</td>
<td>3.31 (0.03)</td>
<td>2.35 (0.04)</td>
<td>0.95 (0.04)</td>
<td>0.15 (0.01)</td>
</tr>
<tr>
<td>H5419A</td>
<td>16</td>
<td>20.51 (0.41)</td>
<td>44.72 (0.18)</td>
<td>26.78 (0.15)</td>
<td>4.77 (0.20)</td>
<td>1.94 (0.10)</td>
<td>98.70 (0.33)</td>
<td>1.68 (0.03)</td>
<td>3.11 (0.01)</td>
<td>2.78 (0.02)</td>
<td>0.36 (0.02)</td>
<td>0.11 (0.01)</td>
</tr>
<tr>
<td>H5419B</td>
<td>15</td>
<td>11.06 (0.72)</td>
<td>49.21 (0.36)</td>
<td>26.39 (0.40)</td>
<td>10.47 (0.57)</td>
<td>1.92 (0.10)</td>
<td>99.06 (0.25)</td>
<td>0.92 (0.06)</td>
<td>3.47 (0.03)</td>
<td>2.77 (0.03)</td>
<td>0.79 (0.05)</td>
<td>0.11 (0.01)</td>
</tr>
</tbody>
</table>

The data in parentheses are one standard deviation of the N analyzed points for each sample. The FeO content in wt.% is obtained by assuming all iron to be ferrous.
Table 4. Chemical composition of the melt analyzed by EPMA.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>N</th>
<th>Al₂O₃ (wt. %)</th>
<th>SiO₂ (wt. %)</th>
<th>MgO (wt. %)</th>
<th>CaO (wt. %)</th>
<th>FeO (wt. %)</th>
<th>Total (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>H5450B</td>
<td>8</td>
<td>1.9(0.3)</td>
<td>17.9(2.8)</td>
<td>16.9(1.7)</td>
<td>4.5(0.6)</td>
<td>26.1(2.4)</td>
<td>67.2(1.4)</td>
</tr>
<tr>
<td>H5450C</td>
<td>14</td>
<td>1.9(0.5)</td>
<td>13.4(1.6)</td>
<td>20.0(2.7)</td>
<td>5.0(0.7)</td>
<td>24.5(2.3)</td>
<td>64.7(1.3)</td>
</tr>
<tr>
<td>H5405B</td>
<td>17</td>
<td>2.4(1.0)</td>
<td>31.7(2.2)</td>
<td>18.5(2.2)</td>
<td>3.9(0.3)</td>
<td>21.7(2.0)</td>
<td>78.1(2.5)</td>
</tr>
<tr>
<td>H5423C</td>
<td>10</td>
<td>0.8(0.1)</td>
<td>28.8(1.3)</td>
<td>20.4(1.9)</td>
<td>3.4(0.5)</td>
<td>26.3(1.9)</td>
<td>79.7(1.5)</td>
</tr>
<tr>
<td>H5423D</td>
<td>11</td>
<td>0.8(0.1)</td>
<td>20.4(1.3)</td>
<td>24.2(1.7)</td>
<td>3.6(0.2)</td>
<td>25.8(1.6)</td>
<td>74.8(1.3)</td>
</tr>
<tr>
<td>H5416A</td>
<td>10</td>
<td>1.9(0.2)</td>
<td>21.1(2.0)</td>
<td>17.6(2.4)</td>
<td>4.7(0.2)</td>
<td>23.9(1.7)</td>
<td>69.2(2.1)</td>
</tr>
<tr>
<td>H5416B</td>
<td>10</td>
<td>2.2(0.4)</td>
<td>30.2(1.5)</td>
<td>21.0(1.7)</td>
<td>3.8(0.2)</td>
<td>19.2(0.8)</td>
<td>76.4(1.2)</td>
</tr>
<tr>
<td>H5419A</td>
<td>8</td>
<td>4.2(0.3)</td>
<td>28.4(0.9)</td>
<td>13.7(0.5)</td>
<td>3.9(0.3)</td>
<td>20.2(0.5)</td>
<td>70.5(1.8)</td>
</tr>
<tr>
<td>H5419B</td>
<td>15</td>
<td>1.3(0.1)</td>
<td>35.0(1.1)</td>
<td>18.3(1.5)</td>
<td>2.7(0.1)</td>
<td>30.6(1.2)</td>
<td>87.8(0.7)</td>
</tr>
</tbody>
</table>

The data in parentheses are one standard deviation of the N analyzed points for each sample. The FeO content in wt.% is obtained by assuming all iron to be ferrous.
Figure 1. (a) Micro-focused XRD pattern taken on the recovered sample H5405B. (b) BSE image of the sample H5405B. Majorite, stishovite, and quenched melt coexist in the sample capsule. Mj: majorite. St: stishovite.
Figure 2. (a) The relationships of Si (blue symbols) and Mg+Fe+Ca (red symbols) to Al atomic contents in majorite. (b) The temperature dependences of Si, Mg, Fe, Ca and Al contents in majorite synthesized from the same starting material.
Figure 3. FTIR spectra of the samples after baseline subtraction and thickness normalization to 1 cm. The peaks at ~3615 cm\(^{-1}\) and ~3430 cm\(^{-1}\), and fitting curves are obtained by least-squares fitting of the FTIR spectra, and represent the infrared absorption bands of majorite, noncrystalline OH, and their summation, respectively.
Figure 4. Temperature dependence of H$_2$O contents in majorite in the recovered samples and comparison with previous studies. The data points from this study are based on the Thomas et al. (2015) FTIR calibration.
Figure 5. Compositional dependence of H$_2$O content in majorite. (A) Al content dependence. (B) Si content dependence.