



Stability and crystal chemistry of iron-bearing dense hydrous magnesium silicates



Geertje Ganskow^a, Falko Langenhorst^{a,b,*}

^a Bayerisches Geoinstitut, Universität Bayreuth, D-95440 Bayreuth, Germany

^b Institut für Geowissenschaften, Friedrich-Schiller-Universität Jena, Carl-Zeiss-Promenade 10, D-07745 Jena, Germany

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ABSTRACT

Dense hydrous magnesium silicates (DHMS) are supposed to be key phases in planetary water cycles because of their ability to carry water to deep mantle regions in subduction slab environments. In order to understand water cycles in iron-enriched planetary systems such as Mars knowledge of the water content and stability of iron-bearing DHMS is required. Iron-bearing DHMS were synthesized based on two starting compositions, $\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$ system and a simple hydrous Martian mantle composition containing Fe, Mg, Al and Si + 12.35 wt% H_2O (hydrous FMAS system). Compared to literature data on phase D, iron-bearing phase D shows analogous variations in water contents as Mg-phase D but appears to be stable at higher temperatures than Mg-phase D for both starting compositions used in this study. Iron-bearing superhydrous phase B contains up to 7 wt% H_2O and shows an extended thermal stability in the hydrous FMAS system. The high-temperature stability of iron-bearing DHMS with a Mars-like bulk composition indicates that these hydrous phases could host significant amounts of water at core-mantle boundary conditions (1500 °C and 23 GPa) in a hydrous Martian mantle.

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1. Introduction

Water is an important volatile component which determines dynamic and physical properties of planetary mantles, especially on the terrestrial planets like Earth and Mars. Water transport and water storage as well as water release mechanisms are significant components of planetary water cycles which have major impacts on mantle processes. On Earth, subduction slabs are assumed to represent the most probable path to transport water in form of hydrous minerals into upper mantle, transition zone and potentially lower mantle regions. The hydrous low-pressure mineral serpentine breaks down to forsterite and enstatite at 5 GPa, and the released water causes subduction-related partial melting in the overlying mantle wedge (Ulmer and Trommsdorff, 1995). However, at temperatures below 600 °C and 7 GPa serpentine decomposes to the dense hydrous magnesium silicate (DHMS) phase A, which transforms to superhydrous phase B (ShyB) and phase D with increasing pressure. This allows the assumption that water could be transported into deep mantle regions by DHMS. It also suggests that

DHMS may remain in the mantle over a longer period of time than previously thought. Due to the potential importance for planetary water cycles, DHMS have been studied intensively in terms of *P-T* stability and crystal chemistry (Frost, 1999; Ohtani et al., 2000; Kawamoto, 2004). Superhydrous phase B, $\text{Mg}_{10}\text{Si}_3\text{H}_4\text{O}_{18}$ (5.8 wt% H_2O) shows the highest stability in respect to temperature (up to 1350 °C according to Kawamoto (2004) and up to 1300 °C for $\text{Mg}_2\text{SiO}_4 + 11 \text{ wt\% H}_2\text{O}$ system (Ohtani et al., 2000), while phase D, $\text{MgSi}_2\text{H}_2\text{O}_6$ has the highest pressure stability up to ~44 GPa (Shieh et al., 1998).

Phase D commonly shows chemical variations, which are related to water incorporation mechanisms involving Mg- and Si-vacancies and Mg–Si disorder to charge balance the variable water contents of 10–18 wt% H_2O (Frost, 1999). However, few data exist for iron-bearing dense hydrous magnesium silicates relevant for the Earth, while data for more iron-rich planetary systems such as Mars is completely missing. The knowledge of such data is not only relevant for Earth as a planet with active plate tectonics but also for Mars. Dense Network of valleys, deltas and paleo-oceans (Carr and Head, 2010), the detection of hydrous minerals and evaporates (e.g., zeolites, clays, and gypsum; Bibring et al., 2006), and recent D/H isotope measurements of Martian meteorites (Kurokawa et al., 2014) provide compelling evidence for the early existence of a water-rich Martian (sub-)surface. This water was probably released early on from the Martian interior.

* Corresponding author at: Institut für Geowissenschaften, Friedrich-Schiller-Universität Jena, Carl-Zeiss-Promenade 10, D-07745 Jena, Germany.
Tel.: +49 3641 948730.

E-mail address: Falko.Langenhorst@uni-jena.de (F. Langenhorst).

Furthermore, data about the ferric iron content of dense hydrous magnesium silicates is required to estimate the influence of Fe^{3+} on the water content and related water substitution mechanisms, which could affect the stability of dense hydrous magnesium silicates. This study provides insights into the stability, crystal chemistry and water substitution mechanisms of iron-bearing DHMS (phase A, superhydrous phase B, and phase D) for the first time also with respect to the ferric iron content. These results extend the present dataset on the stability and crystal chemistry of DHMS to iron-rich compositions (Kawamoto, 2004), which are potentially important for understanding the water inventory and cycle in iron-dominated planetary mantles such as that of planet Mars.

2. Sample characterization

Iron-bearing dense hydrous magnesium silicates were synthesized based on a stoichiometric $\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$ starting composition ($\text{Fe}_2\text{SiO}_4 + \text{Mg}(\text{OH})_2 + \text{SiO}_2$) and a simplified Martian mantle model composition (Wänke and Dreibus, 1988), which is referred to as hydrous FMAS system (Table 1). Both represent model compositions for planetary systems in the first approximation to study the effect of iron and water content on the stability and compositions of coexisting mineral phases. The experiments were conducted at 9–23 GPa and 750–1300 °C ($\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$ system) and 18–23 GPa and 1150–1450 °C (hydrous FMAS system) using 1000 t and 1200 t multi-anvil presses at Bayerisches Geoinstitut, Bayreuth, Germany. The starting material was loaded into welded Pt-capsules with 2 mm, 1.6 mm and 1.2 mm diameter for 18/11 (up to 10 GPa), 14/8 (15 GPa), 10/5 (18 GPa) and 10/4 (20.5–23 GPa) multi-anvil assemblies, respectively. Heating durations range between 1 and 8 h (Table 1). The following temperature

gradients of the particular assemblies were reported by Walter et al. (1995). These values represent the temperature gradient from the furnace midline toward the end of the cylindrical furnace unit for the 18/11 assembly -50 °C/mm (high-T stepped LaCrO_3 heater), 14/8 assembly -70 °C/mm (high-T stepped LaCrO_3 heater) and 10/5 and 10/4 assembly -200 °C/mm (straight LaCrO_3 heater). These gradients result in a heterogeneous phase distribution inside the capsule as shown in Fig. 1 (e.g., sample 2990 and 2540). According to Gibbs' phase rule $F = P - C + 2$ (number of present phases – P , number of components – C) chemical equilibrium was reached for all experiments even with short heating durations. Chemical compositions were measured on polished surfaces of several crystals mounted in epoxy-resin using a JEOL JXA-8200 microprobe at Bayerisches Geoinstitut. Several point analyses on different crystals were performed using wavelength dispersive spectroscopy at 15 kV and 20 nA. Standards used for calibration were hematite (Fe_2O_3) for Fe, spinel (MgAl_2O_4) for Al and diopside ($\text{CaMgSi}_2\text{O}_6$) for Mg and Si. Raw intensities were corrected by ZAF matrix-correction and converted into oxide weight percent. Results of the chemical analyses are given in Tables 2 and 3.

In order to determine the amount of Fe^{3+} , polished thin sections (30 μm thickness) of the run products were glued on 100 mesh molybdenum grids and thinned down to electron transparency ($\sim 100 \text{ nm}$) using Argon-milling method (GATAN dual ion mill 600). Selected area electron diffraction (SAED) and electron energy-loss spectroscopy (EELS) were performed on a Philips CM20 FEG transmission electron microscope operating at 200 kV. Electron energy-loss near-edge structure (ELNES) spectra were collected using energy-loss spectrometer (Gatan PEELS 666) with 0.8 eV energy resolution as described by Langenhorst and Solozhenko (2002) and Stoyanov and Langenhorst (2014). Quantification of $\text{Fe}^{3+}/\Sigma\text{Fe}$ ratio was done following the procedure described by

Table 1
Experimental run conditions and observed phase assemblages (rw – ringwoodite, A – phase A, D – phase D, SHyB – superhydrous B); “assembly” refers to the applied multi-anvil assembly according to the desired pressure.

Sample/assembly	P [GPa]	T [°C]	Heating duration	Bulk composition	Phases present
2580/ 18/11	9	750	2	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	rw + A + brucite
2895/ 18/11	10	750	8	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	rw + A + brucite
2539/ 14/8	15	750	3	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	rw + A
2540/ 10/5	18	750	2	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	rw + D
2827/ 10/4	20.5	950	4	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	D + SHyB + iron oxide
2737/ 10/4	20.5	1150	8	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	D + SHyB + rw + iron oxide
2795/ 10/4	20.5	1300	5	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	D + rw + iron oxide + brucite + melt
2613/ 10/4	23	950	1	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	D + SHyB + iron oxide + brucite
2736/ 10/4	23	1150	8	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	D + rw + iron oxide + brucite
2793/ 10/4	23	1300	8	$\text{MgFeSiO}_4 + 9.5 \text{ wt\% H}_2\text{O}$	D + rw + iron oxide + brucite + melt
4376/ 10/5	18	1250	4	hy FMAS + 12.5 wt% H_2O	D + SHyB + iron oxide + brucite + residual phase
4345/ 10/4	20.5	1150	4	hy FMAS + 12.5 wt% H_2O	D + SHyB + iron oxide + brucite
2945/ 10/4	20.5	1300	4	hy FMAS + 12.5 wt% H_2O	D + SHyB + iron oxide + melt
4348/ 10/4	20.5	1450	4	hy FMAS + 12.5 wt% H_2O	D + SHyB + stishovite + melt
2947/ 10/4	23	1150	4	hy FMAS + 12.5 wt% H_2O	D + SHyB + iron oxide + brucite
4377/ 10/4	23	1350	4	hy FMAS + 12.5 wt% H_2O	D + SHyB + iron oxide + residual phase + melt

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