

# Report for the Joint Use/Research of the Institute for Planetary Materials, Okayama University for FY2024

02/21/2025

**Category:** International Joint Research General Joint Research Joint Use of Facility  
Workshop

**Name of the research project:** Phase equilibria of bridgmanite and CaFe<sub>2</sub>O<sub>4</sub>-type  
aluminous phase in the Na<sub>2</sub>O-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system

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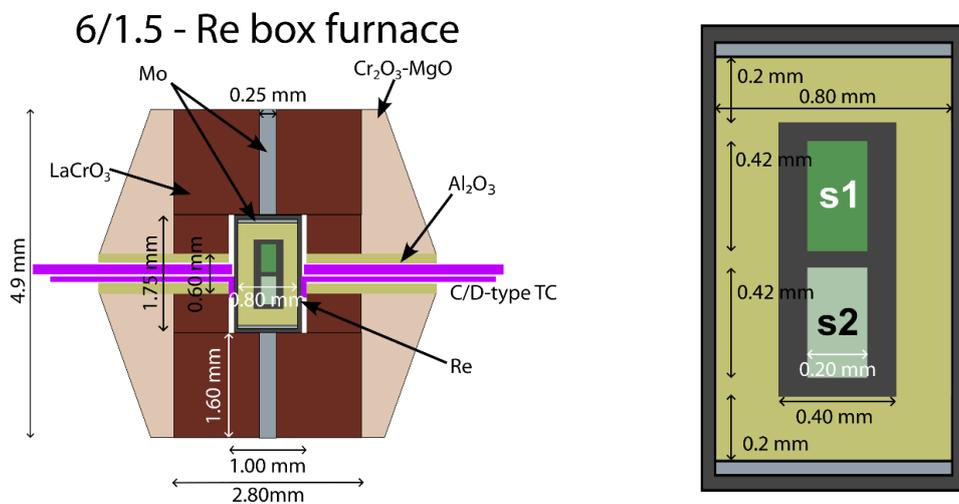
## Research report:

### 1. Research Purpose

Three dimensional models of the Earth's lower mantle show that subducted oceanic plates can be recycled in the lower mantle through deep subduction processes, enabling crustal materials to be transported into the Earth's lower mantle down to at least 1000 km. Metabasaltic rocks, comprising the top 10 km of the slab, are constituted of (Mg,Fe,Al)(Si,Al,Fe)O<sub>3</sub> bridgmanite, (Na,Mg,Fe)(Al,Si,Fe)<sub>2</sub>O<sub>4</sub> CaFe<sub>2</sub>O<sub>4</sub>-type (CF) phase, SiO<sub>2</sub> stishovite and CaSiO<sub>3</sub> davemaoite. In order to correctly interpret seismic data, it is critical to determine how pressure and temperature affect the chemical composition and element partitioning of these phases, especially those of bridgmanite and the CF phase owing to their complex crystal chemistry. In this project, I proposed to conduct phase equilibria experiments in the Na<sub>2</sub>O-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system using a multi-anvil apparatus to determine the major element partitioning between bridgmanite and the CaFe<sub>2</sub>O<sub>4</sub>-type (CF) aluminous phase at 33 GPa and 1700-2300 K. As bridgmanite and the CF phase comprise at least 50 vol.% of a basaltic phase assemblage at lower mantle conditions, results from this project aim to shed light on the mineralogical composition of subducted oceanic crust, in order to constrain its seismic properties and seismic detectability.

## 2. Conducted research

I conducted three experiments using the 5000-ton Kawai-type multi-anvil apparatus installed at the IPM. F05-grade tungsten carbide (WC) anvils with 1.5 mm truncations were used to compress an octahedral assembly with 6.0 mm edge length. In each experiment, two compositions were targeted simultaneously to determine the element partitioning under MgO- or SiO<sub>2</sub>-saturated conditions. Samples were loaded in Re capsules, which in turn were inserted in an Al<sub>2</sub>O<sub>3</sub> tube. A 0.07 mm-thick Re foil was wrapped around the Al<sub>2</sub>O<sub>3</sub> tube and served as a heater. Mo electrodes were used to connect the box furnace to the WC anvils. LaCrO<sub>3</sub> was used as thermal insulator. A type C thermocouple was inserted horizontally and connected through the Re heater. A schematic view of the assembly is shown in **Figure 1**.



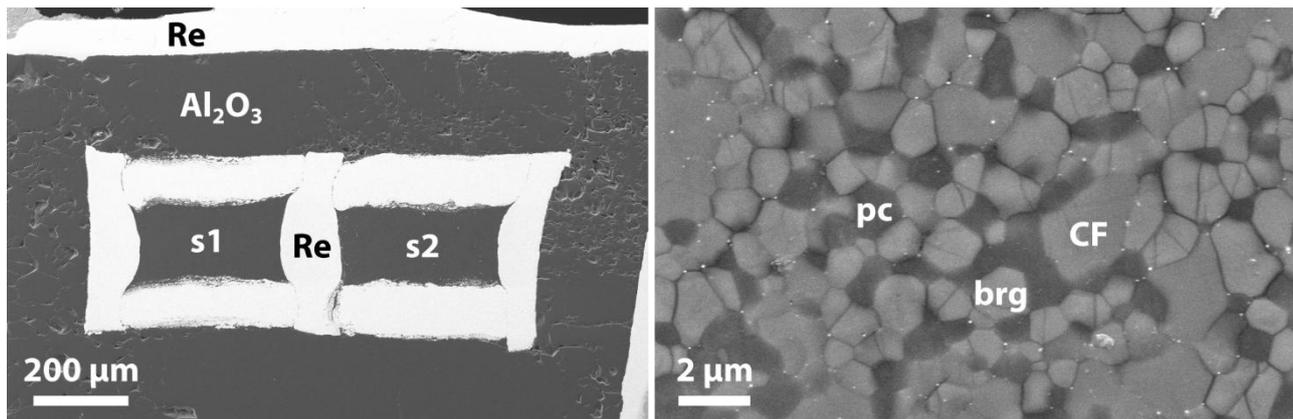
**Figure 1.** Cross section of the 6/1.5 multi-anvil assembly used for experiments at IPM. s1 and s2 indicate samples with different composition (i.e., MgO- or SiO<sub>2</sub>-saturated)

Each experiment was compressed to a target load of 7.5 MN in 4 h and heated to the target temperature at a rate of approximately 150 K/min. Due to malfunctioning of the thermocouple, temperature was estimated using power-temperature relations derived in the low temperature range. Initially, a linear relation was assumed and temperatures of ~1850, ~2000, and ~2200 K were targeted. However, after more careful consideration, a 2<sup>nd</sup> order polynomial seemed to better reproduce the low temperature data, resulting in temperatures of ~2173, ~2373, and ~2573 K. The power was kept constant for 2-3 h before samples were quenched by switching off the electrical power supply and slowly decompressed to room pressure over 12-15 h.

## 3. Research Outcomes

The Re box furnaces were then recovered from their respective assemblies and embedded in epoxy for textural observation and chemical analyses by electron microscopy at the Earth and Planets Laboratory, Carnegie Science (Washington DC, USA). SEM images were acquired using a Zeiss

Auriga field-emission scanning electron microscope (SEM). Secondary electron (SE) images show that the geometry of the assembly within the Re furnace was preserved under the high-pressure-temperature conditions, with minimal deformation of the capsules and samples within (). The grain sizes and textures of the run products were determined from backscattered electron (BSE) images, revealing grains of CF phase up to several micrometers, whereas bridgmanite, stishovite and periclase grains were generally below 2 micrometers. A representative sample texture is shown in **Figure 2b**.



**Figure 2.** a) SE image showing the two samples (s1, s2) synthesized in run 5k4233. b) BSE image of the run products of the MgO-saturated composition from the same run. Light grains with low relief are CF-type phase (CF), light grains with high relief are bridgmanite (brg), and dark grains are periclase (pc).

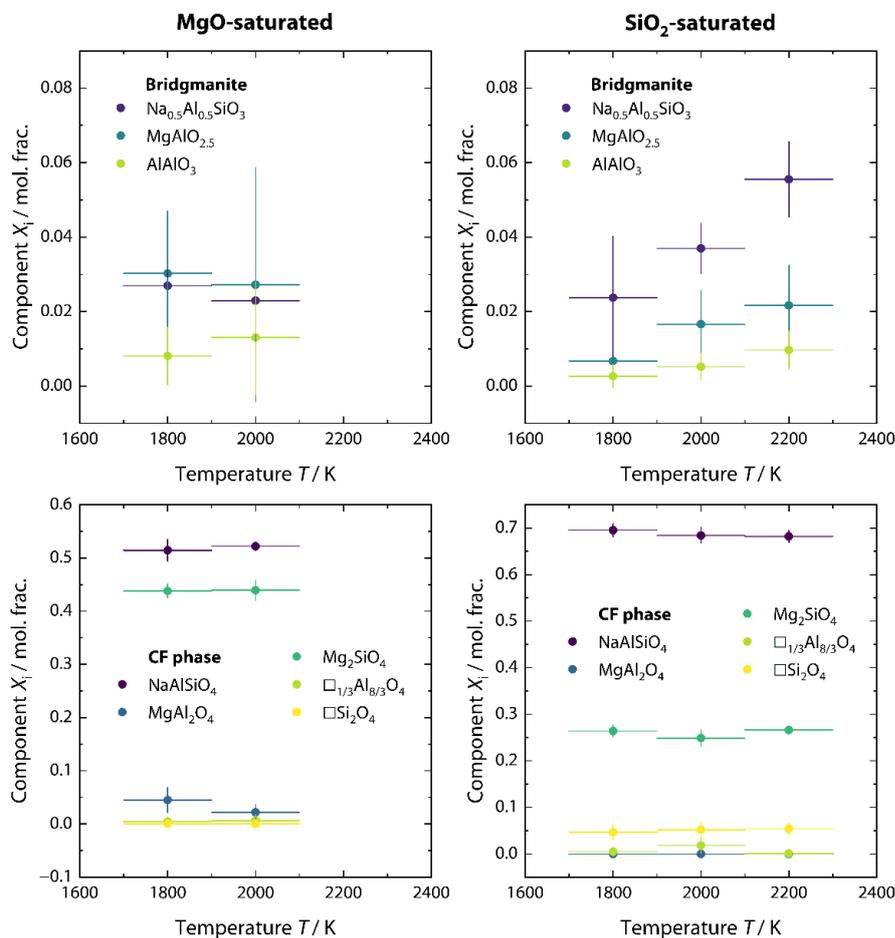
The chemical compositions of the run products were determined using the JEOL JXA-8530F field-emission electron probe micro-analyzer (EPMA). Due to the small size of the recovered samples, a low acceleration voltage of 5 kV and beam current of 5 nA were used to minimize the interaction volume between the electron beam and the sample, as well as the damage from electron irradiation. The beam diameter was adjusted between 1 and 3  $\mu\text{m}$  depending on the grain size. Natural albite was used as a standard for Na, synthetic corundum for Al, and synthetic enstatite for Mg and Al. High quality analyses were collected on all samples, except the MgO-saturated sample at 2300 K, where extensive contamination from Re has so far prevented conclusive analyses. Molar abundancies for the relevant bridgmanite and CF phase components are shown as a function of temperature in **Figure 3**.

The main components in the CF phase are  $\text{NaAlSiO}_4$  and  $\text{Mg}_2\text{SiO}_4$  in both systems. Interestingly, the concentration of  $\text{NaAlSiO}_4$  increases with temperature in the MgO-saturated system, but decreases with temperature in the  $\text{SiO}_2$ -saturated system. The molar fraction of  $\text{Mg}_2\text{SiO}_4$ , on the other hand, seem to be temperature independent. Cation vacancies of the  $\square\text{Si}_2\text{O}_4$  type were only observed when the CF phase coexisted with stishovite. This does not exclude that Al-related vacancies can be formed through the  $\square_{1/3}\text{Al}_{8/3}\text{O}_4$  component in more Al-rich compositions.

The oxygen vacancy component  $\text{MgAlO}_{2.5}$  is observed in the presence of both stishovite and periclase, supporting the idea that basaltic bridgmanite may be oxygen vacancy-rich. Although Na is generally considered to be incompatible in bridgmanite, the  $\text{Na}_{0.5}\text{Al}_{0.5}\text{SiO}_3$  substitution (up to 0.06 mol. frac. or 6 mol.%) is observed under both MgO- and  $\text{SiO}_2$ -saturated conditions. The solubility of this component displays a positive temperature dependence in the  $\text{SiO}_2$ -saturated system but remains relatively constant in the MgO-saturated system. This difference might be explained by the relatively high concentration of Na and Al in periclase, which follows the  $\text{Na}_{0.5}\text{Al}_{0.5}\text{O}$  substitution mechanism (up to 0.13 mol. frac. or 13 mol.%) and displays positive temperature dependence, whereas  $\text{SiO}_2$  stishovite is virtually Al- and Na-free.

#### 4. Preliminary implications

- Na can be incorporated in basaltic bridgmanite in equilibrium with stishovite and CF phase, although this substitution is likely to be relevant mainly in hot regions of the mantle (e.g., plumes).
- Na preferentially partitions in ferropericlase over bridgmanite in the pyrolitic lower mantle.
- Stishovite coexisting with bridgmanite and the CF phase is essentially pure  $\text{SiO}_2$  (Al < 0.005 apfu), suggesting that Al-bearing silica phases may be formed primarily in hydrous basaltic systems.
- Additional runs will be needed to better constrain the power temperature relations.



**Figure 3.** Molar fractions of relevant end-member components of bridgmanite and CF phase as a function of the estimated temperature at 33 GPa.