Report for the Joint Use/Research of the Institute for Planetary Materials, Okayama University for FY2023

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Category: ☑International Joint Research ☐General Joint Research ☐Joint Use of Facility ☐Workshop

Name of the research project: Determination of the Al_2O_3 solubility in $MgSiO_3$ bridgmanite

Principal applicant: Tomoo Katsura

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Collaborator

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3: Japan Synchrotron Radiation Institute

4: Center for High Pressure Science and Technology Advanced Research

Research report:

In 2023 fiscal year we had 2 beamtimes at SPring-8 in collaboration between Bayerisches Geoinstitut, (University of Bayreuth, Germany) and Institute for Planetary Materials (Okayama University, Japan). The aim of these beamtimes was to investigate solubility of Al_2O_3 in MgSiO3 bridgmanite coexisting with corundum as a function of pressure at 2000 K. Here we report the results of the beamtimes:

Beamtime #1 (19.06.2023-24.06.2023).

We conducted 5 experiments using SD anvils and 2 experiments using WC anvils (Table 1). Figures 1 and 2 show the cell assembly designs. In all experiments we used Cr_2O_3-doped MgO octahedral as a pressure medium, cylindrical CVD BDD heater, and TiC electrodes. Temperature was measured using D-type thermocouple. Combination of Mo + Cu electrodes was used for experiments with SD anvils. A fine-grained oxide mixture of composition $En_{50}Cor_{50}$ (En: $MgSiO_3$, Cor: Al_2O_3; the number represents mol %, oxide grain size: 50 nm) was used as a starting material. MgO + Pt (10:1 wt. %) oxide mixture was used as a pressure marker.

For experiments with SD anvils, the experimental procedure can be described as follows: the

sample was successively compressed up to 0.5, 1.0, 1.5, 2.0, 2.5, 3.1, 3.8, 4.6, 5.5, 6.5, 7.6 MN. The compression duration between these steps was 40 minutes. At each step, the sample was heated to 1000 K and then cooled to 300 K. Before heating, at 1000 K, and after cooling sample diffraction was taken and pressure was calculated. This procedure was applied to reduce the possibility of a blowout during compression. At target pressload (7.6 MN), sample was heated to ≈ 2250 K. However, only in M4031 experiment, sample was successfully heated to target temperature (Table 1). At ≈ 2250 K sample pressure was determined as 55.7(2) GPa. After 30 min explosion at these conditions, the sample was quenched and then decompressed to ambient pressure. After quenching, sample diffraction was taken (Figure 3).

The experimental procedure for experiments with WC anvils is different. In M4035 experiment, sample was successively compressed to 2.5, 5.0 and 15.0 MN. At 2.5 and 5.0 MN, the sample was heated to 800 K and then cooled to 300 K. Before heating, at 1000 K, and after cooling sample diffraction was taken and pressure was calculated. This procedure was applied to reduce the possibility of a blowout during compression. In M4036 experiment, sample was compressed directly to 15.0 MN. At target pressload (15.0 MN), sample was heated to 2300 K. However, only in M4035 experiment, sample was successfully heated to target temperature (Table 1). At 3000 K sample pressure was determined as 48.7(2) GPa. After 60 min explosion at these conditions, the sample was quenched and then decompressed to ambient pressure. After quenching, sample diffraction was taken (Figure 3).

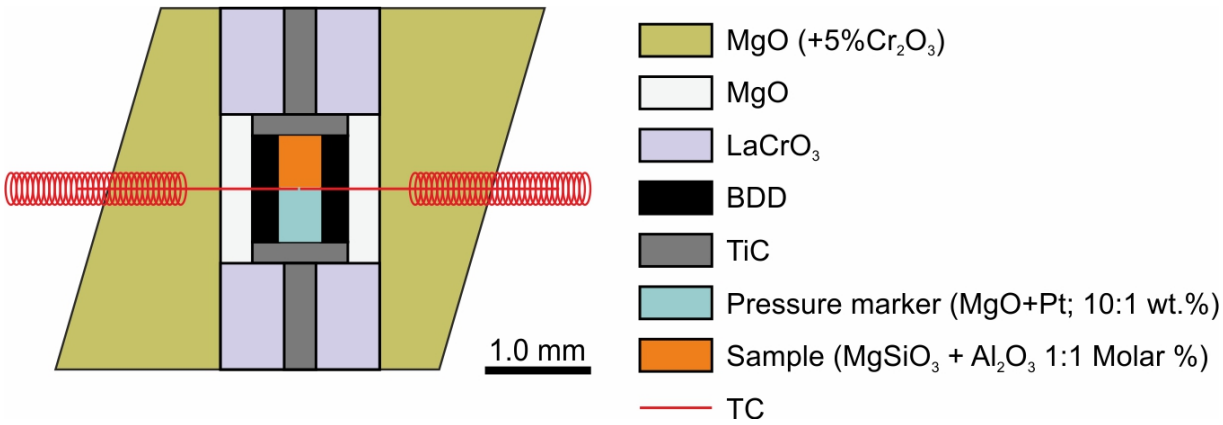

Figure 1. 4.05/1.0 experimental assembly

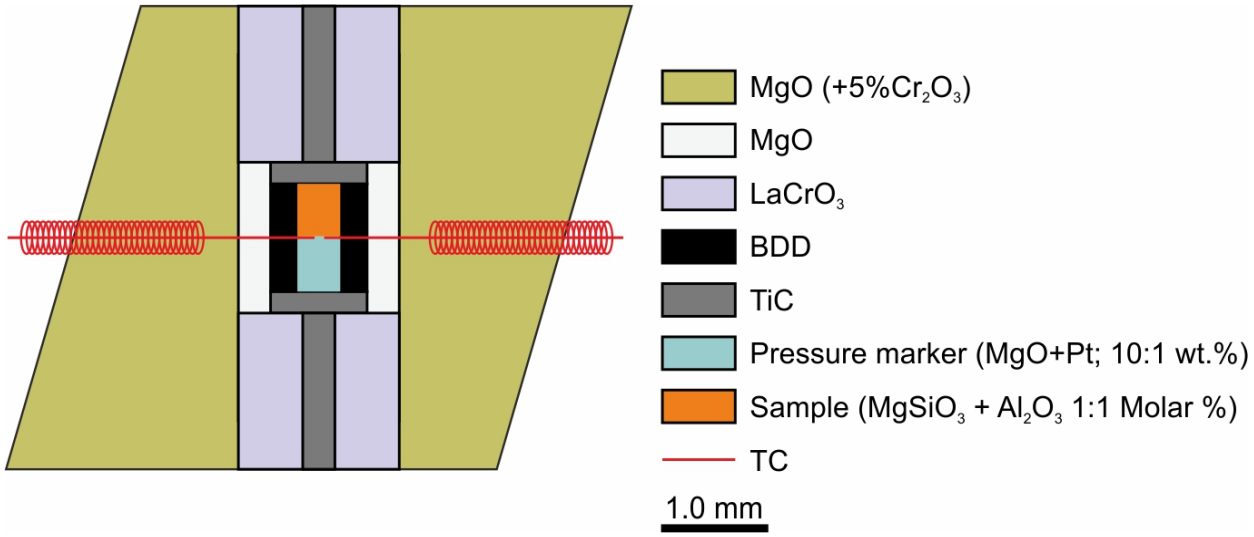

Figure 2. 5.24/1.0 experimental assembly

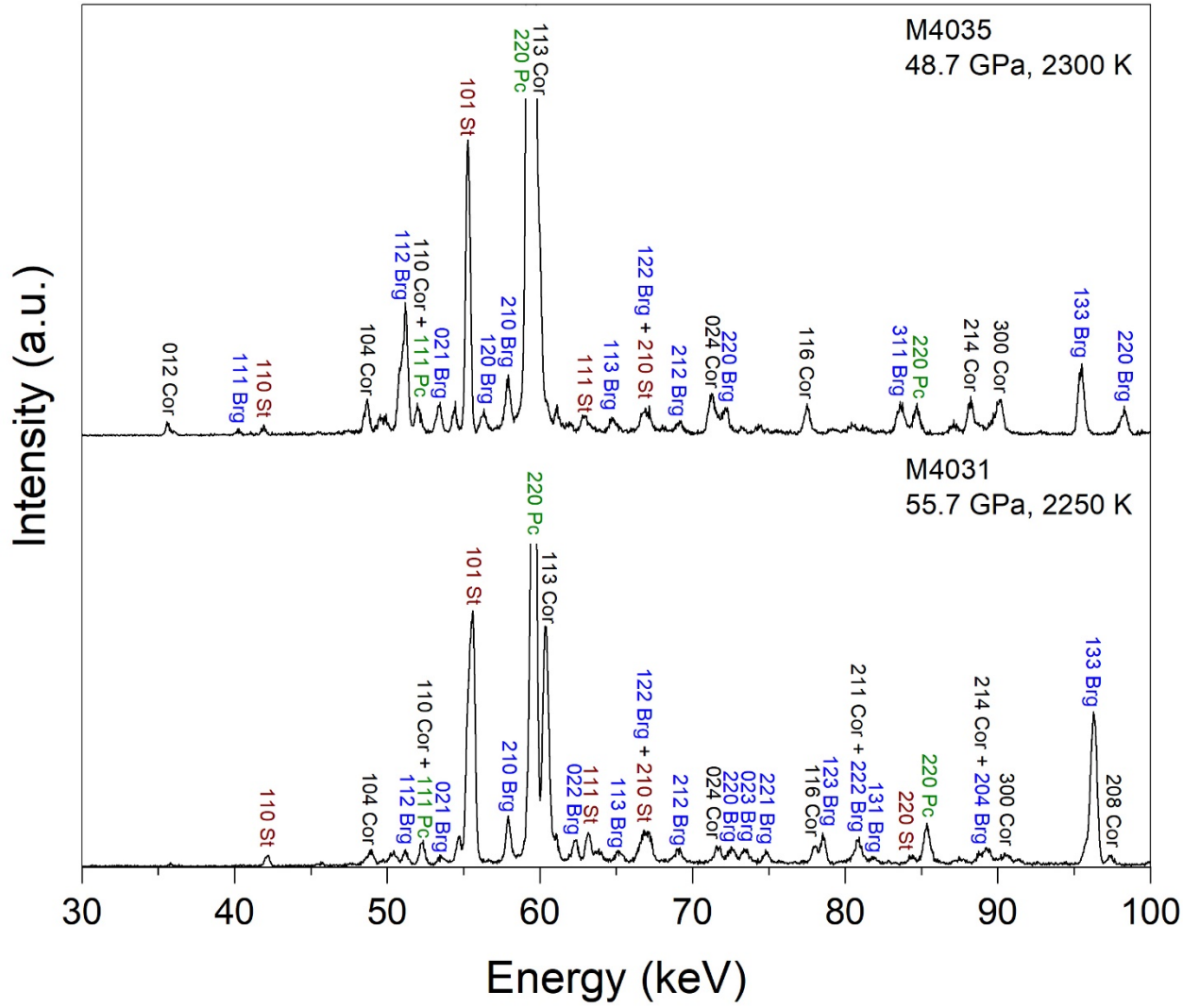

Figure 3. XRD of samples after quenching in experiments M4031 (bottom) and M4035 (top). The numbers above the peaks indicate the Miller indexes of bridgmanite, corundum, stishovite and periclase.

Table 1. Experimental summary.

Run #	Electrode material	Anvils	Assembly	Maximum pressload, MN	Number of heating cycles	Highest P, Gpa	Highest T, K	remarks
M4030	Mo+Cu	SD	4.05/1.0	0.7	1	9.9	1000	Blowout at 0.7 MN occurred during compression
M4031	Mo+Cu	SD	4.05/1.0	7.6	11	55.7(2)	2250	Kept at 2250 K in 30 minutes. Blowout during decompression; 6 SD anvils were broken.
M4032	Mo+Cu	SD	4.05/1.0	0.7	1	9.2	1000	Blowout at 0.7 MN occurred during compression
M4033	Mo+Cu	SD	4.05/1.0	0.85	1	8.3	900	Blowout at 0.85 MN occurred during compression
M4034	Mo+Cu	SD	4.05/1.0	0.5	1	9.7	≈1000	Experiment was terminated due to TC breakage during heating at 0.5 MN
M4035		WC	5.24/1.0	15	3	48.7(2)	2300	Kept at 2300 K in 60 minutes. Blowout during decompression; all anvils were broken. The sample was destroyed by blowout.
M4036		WC	4.05/1.0	15	1	43.1	300	At 15 MN heating was failed due to low resistance of the heater (≈0.003 Ohm). Possibly electrode material (TiC) was extruded in the anvil gap. Blowout during decompression; all anvils were broken.

Beamtime #2 (22.01.2024-27.01.2024).

We conducted 1 experiment using SD anvils and 4 experiments using WC anvils (Table 2). Figures 1 and 2 show the cell assembly designs. In all experiments we used Cr_2O_3-doped MgO octahedral as a pressure medium, cylindrical CVD BDD heater, and TiC electrodes. Temperature was measured using D-type thermocouple. Combination of Mo + Cu electrodes was used for experiment with SD anvils. A glass with composition $En_{50}Cor_{50}$ (En: $MgSiO_3$, Cor: Al_2O_3; the number represents mol %, oxide grain size: 50 nm) was used as a starting material. MgO + Pt (10:1 wt. %) oxide mixture was used as a pressure marker.

For experiment with SD anvils (M4211), the experimental procedure can be described as follows: the sample was successively compressed up to 0.5, 1.0, 1.5, 2.0, 2.5, 3.1, 3.8, 4.6, 5.5, 6.5, 7.6, 8.8 MN. The compression duration between these steps was 40-45 minutes. At each step, the sample was heated to 1000 K and then cooled to 300 K. Before heating, at 1000 K, and after cooling sample diffraction was taken and pressure was calculated. This procedure was applied to reduce the possibility of a blowout during compression. At target pressload (8.8 MN), sample was heated to 1773 K. The further heating was suspended due to limit of AC power supply. After replacement of AC to DC heating system, cell assembly was accidentally overheated, which caused blowout.

The experimental procedure for experiments with WC anvils was different. In M4212, M4213 and M4215 experiments, samples were initially compressed to 2.0 MN and heated to 1000 K. After that, samples were compressed to 9.0 (M4212, M4213) and 12.0 (M4215) MN. At target pressload, samples were heated to 2300 K. After exposure at this temperature in 30-60 min, samples were quenched and then decompressed to ambient pressure. After quenching, sample diffractions were taking, confirming that run products consist of bridgmanite and corundum (Figure 4).

Experiment M4214 with WC anvils was failed due to blowout during compression.

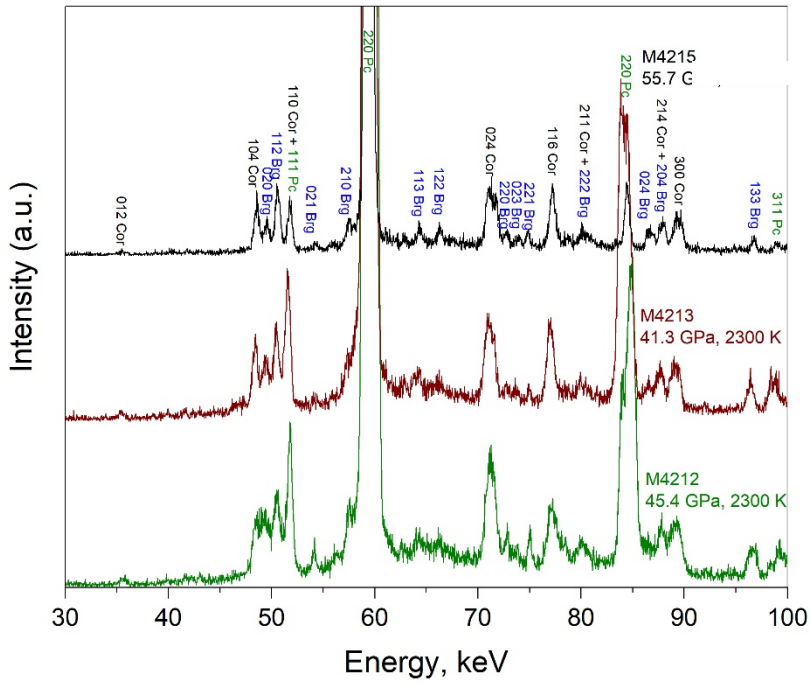

Figure 4. XRD of samples after quenching in experiments M4212 (green), M4213 (wine) and

M4215 (black). The numbers above the peaks indicate the Miller indexes of bridgmanite, corundum and periclase.

Table 2. Experimental summary.

Run #	Electrode material / Taper angle	Anvils	Assembly	Maximum pressload, MN	Number of heating cycles	Highest P, Gpa	Highest T, K	remarks
M4211	Mo+Cu / 1°	14mm SD	4.05/1.0	8.8	12	55.2	1773	At 8.8 MN 2 heating cycles: 1. Using AC heating system. Due to power limit on a power source, heating was suspended at 1773 K and sample was cooled down 2. Using DC heating system. Sample was overheated which caused blowout
M4212	1.7°	14mm WC TJS01	5.24/1.0	9.0	2	44.4	≈2300 (698 W)	Kept at ≈2300 K in 30 minutes. Blowout during decompression
M4213	1.5°	14mm WC TJS01	5.24/1.0	9.0	2	41.3	≈2300 (605 W)	Kept at ≈2300 K in 60 minutes. Blowout during decompression
M4214	1.7°	14mm WC TJS01	5.24/1.0	1.5	0	-	300	Blowout at 1.5 MN occurred during compression
M4215	1.7	14mm WC TJS01	4.05/1.0	12.0	2	45.4	≈2300 (605 W)	Kept at ≈2300 K in 35 minutes. Blowout during decompression