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

2022 fiscal year first term / second term / others

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

Name of the research project: Electrical conductivity measurement of partially molten peridotite under high pressure with implications for the high conductivity anomalies in the deep Earth

Principal applicant: Haibo Liu

Affiliated institution and department: Zhejiang University

Collaborator

Name: Professor Takashi Yoshino

Affiliated institution and department: Institute for Planetary Materials

Research report:

1) Please write the research report with free format, but include followings: research purpose, actually conducted research, and research outcomes. If necessary, you can add another pages.
2) For the workshop, please write the report for the workshop. Also, attach the program, abstracts, and list of the participants etc.
3) Please add Collaborator's Name, Affiliated institution and department as needed.

Research purpose:

1. The conductivity of hydrous KLB-1 was measured at 13 and 15 GPa, respectively, to check the difference in conductivity across the 410 km discontinuity.
2. Checking the difference in conductivity between dry and hydrous KLB-1 systems.
3. Confirm the hypothesis that the dehydration melting at the top of 410 km caused the high conductivity anomaly.

Experimental procedures:

1. Preparation of starting materials

The composition of KLB-1 sample is shown in Table 1, reagent grade oxide and carbonate powders are weighed and mixed, and ground in an agate mortar with alcohol. The ground fine-grained powder was then placed in a crucible in a muffle furnace with decarburization at 800 °C and 12 h. Subsequently, the decarburized powder was pressed into pellets, and the hydrous

and dry KLB-1 were baked in a gas mixing furnace with QFM buffer at 1350 °C for 43 h and 8 h, respectively. The recovered pellets were ground again with alcohol in an agate mortar to obtain dry KLB-1 starting material, and brucite was added to obtain 0.5 wt% hydrous KLB-1 starting material.

Table 1. Composition of KLB-1 sample

	KLB-1	**weight (g)**	**transform**	**weight (g)**	**remark**
SiO_2	44.48	0.4448			
TiO_2	0.16	0.0016			
Al_2O_3	3.59	0.0359			
FeO	8.10	0.0810	Fe_2O_3	0.0900	$Fe_2O_3=2FeO+0.5O_2$
MnO	0.12	0.0012			
MgO	39.22	0.3922	MgO	0.3811	$Mg(OH)_2=MgO+H_2O$
CaO	3.44	0.0344	$CaCO_3$	0.0614	$CaCO_3=CaO+CO_2$
Na_2O	0.30	0.0030	Na_2CO_3	0.0051	$Na_2CO_3=Na_2O+CO_2$
K_2O	0.02	0.0002	K_2CO_3	0.0003	$K_2CO_3=K_2O+CO_2$
Cr_2O_3	0.31	0.0031			
NiO	0.25	0.0025			
Total	99.99	1.0000			

2. Synthesis and characterization of dry and hydrous KLB-1 samples

As shown in Table 2, the cell assembly of the nine experiments were similar with minor differences. We tried Au, Fe, and Mo capsules successively. In general, dry or hydrous KLB-1 powder was loaded into a capsule with 2 mm diameter and 3 mm length, and inserted them into MgO sleeve with 3 mm diameter, $LaCrO_3$ heater with 4 mm diameter, ZrO_2 insulator with 6 mm diameter and 14 mm octahedron in sequence. $W_{97}Re_3$-$W_{75}Re_{25}$ thermocouple was set in the middle of the sample capsule to monitor temperature. Before compression, the whole cell assembly was kept in 200 ºC vacuum furnace overnight to minimize absorbed water. 32 mm anvils with 8 mm truncations was later adopted as the second stage in a multi-anvil apparatus. Sintering experiments were conducted at 7 MN (15 GPa). After reaching the target pressure, increase to the target temperature at a rate of 50 ºC/min. The duration and other detailed experimental conditions are summarized in Table 2.

The recovered samples after cutting and polishing were used to determine the phase structure using X-ray diffraction (XRD), and the grain size, composition, and microtexture were determined using scanning electron microscopy (SEM) with Electron Backscatter Diffraction (EBSD).

Results

The compositions and typical images of the recovered samples for all experiments are shown in Table 2. We obtained hydrous and dry KLB-1 samples with the ideal composition using Au capsules, but the samples were almost indistinguishable macroscopically and microscopically.

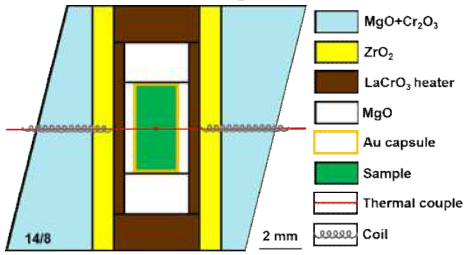
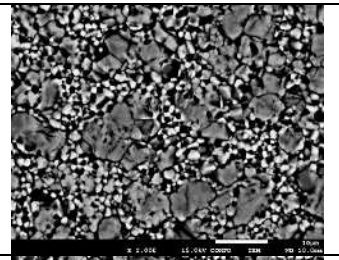
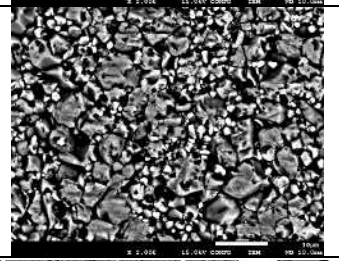
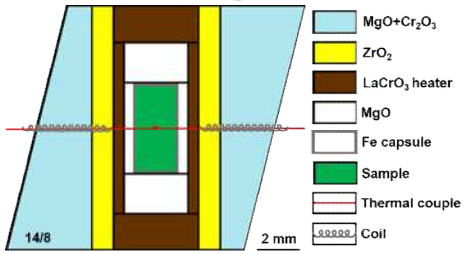
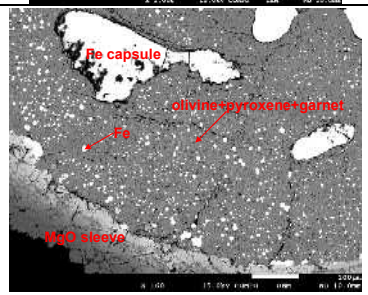
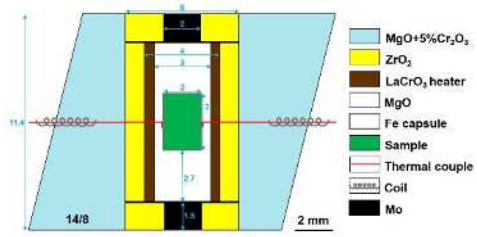
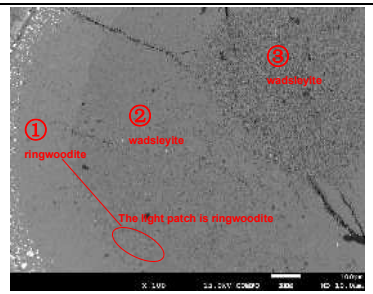
Wadsleyite has a grain size of 10 μm or more. Sample polishing was always suboptimal due to poor sintering even with binders. Therefore, we reground the hydrous and dry KLB-1 powder in an agate mortar with alcohol until the grain size was less than 3 μm.

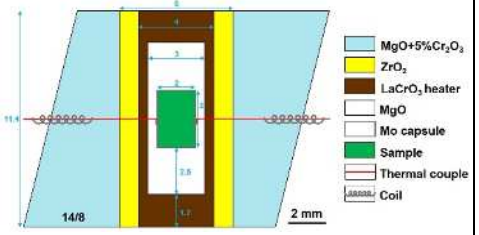
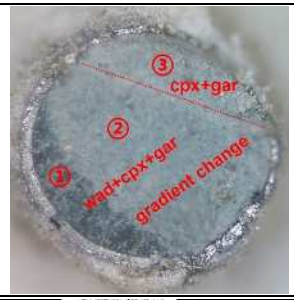
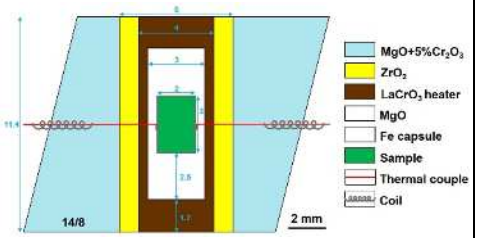
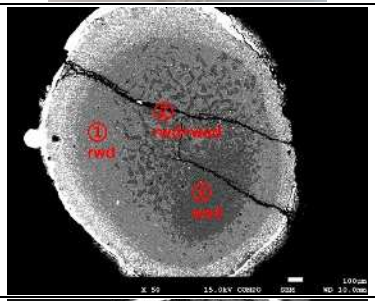
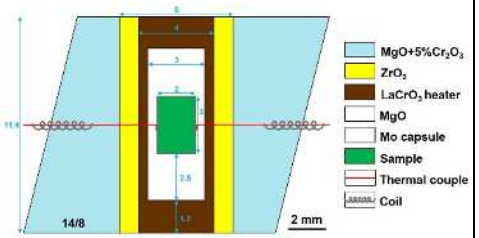
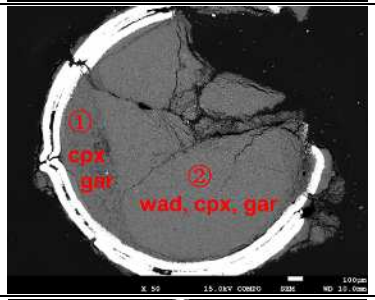
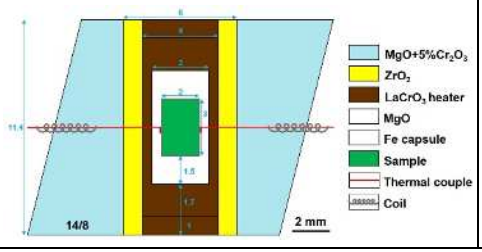
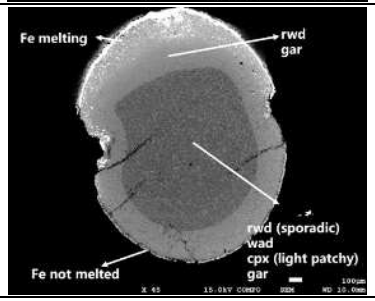
We resynthesized dry KLB-1 samples using Fe capsule in order to absorb excess water. After reaching the target temperature, the temperature drops due to unknown reasons and the temperature cannot be stabilized. The recovered samples were powdery or brittle due to short duration or low temperature that did not sinter well. On the other hand, the sample melted or the iron diffused and aggregated to form ringwoodite due to the high temperature. In the last experiment, we changed the length of the $LaCrO_3$ heater and the temperature seemed to become stable and a well sintered sample was obtained, and the polishing was also good, but there were still ringwoodite rings near the Fe capsule due to the high iron content. Therefore, we replaced it with Mo capsules in order to keep the same redox buffer.

The composition of the recovered samples was as expected using Mo capsules, but the texture was extremely heterogeneous. Gradient changes in composition and grain size occurred in both experiments, and the reason is still unknown.

All recovered samples of conforming composition were used for coring, but they were broken due to not being well sintered. Therefore, the conductivity measurement experiment was not performed.

Table 2. Experiment summary

	Assembly	Pressure	Temperature*	Time	XRD	SEM+EDS**	Typical image
1K3567 dry 1.24	Au capsule 	7 MN	1400 °C power + 88	156 min	wadsleyite	wad cpx gar	
5K3756 hydrous 1.28		7 MN	1200 °C power + 15	20 min	wadsleyite	wad cpx gar	
5K3765 dry 2.14	Fe capsule 	7 MN	1400-1240-no °C linear decrease TC broke at 20 min power + 146	30 min	olivine	ol cpx gar melt	
1K3573 dry 2.17		7 MN	1350-1213 °C linear decrease power + 26	24 min	wadsleyite	—	
5K3772 dry 2.25	Fe capsule 	7 MN	1350-1271 °C sawtooth decrease power + 30	30 min	ringwoodite wadsleyite	rwd wad cpx gar	

	Assembly	Pressure	Temperature*	Time	XRD	SEM+EDS**	Typical image
5K3776 dry 3.6	Mo capsule 	7 MN	1350-1210 ℃ decrease power + 27	60 min	wadsleyite	wad cpx gar	
1K3582 dry 3.11	Fe capsule 	7 MN	1350-1130 ℃ P control 40 min T decrease T control 28 min (power is stable at 527, similarly power + 122)	68 min	ringwoodite wadsleyite	rwd wad gar	
5K3785 dry 3.20	Mo capsule 	7 MN	1350-1225 ℃ T control 45 min power + 38 P control 10min T decrease	55 min	wadsleyite	wad cpx gar	
5K3790 dry 3.27	Fe capsule 	7 MN	1100-1300 ℃ T control 1100 keep 10 min 1200 keep 30 min 1300 keep 10 min decrease T keep 30	80 min	ringwoodite wadsleyite	rwd wad cpx gar	

* power + refers to the highest power minus the power when the target temperature is initially reached

** — refers to no measurement