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In situ reinvestigation of reaction phase A plus high-pressure clinoenstatite to forsterite plus water in the system MgO-SiO_2-H_2O (MSH)

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Supplementary materials:

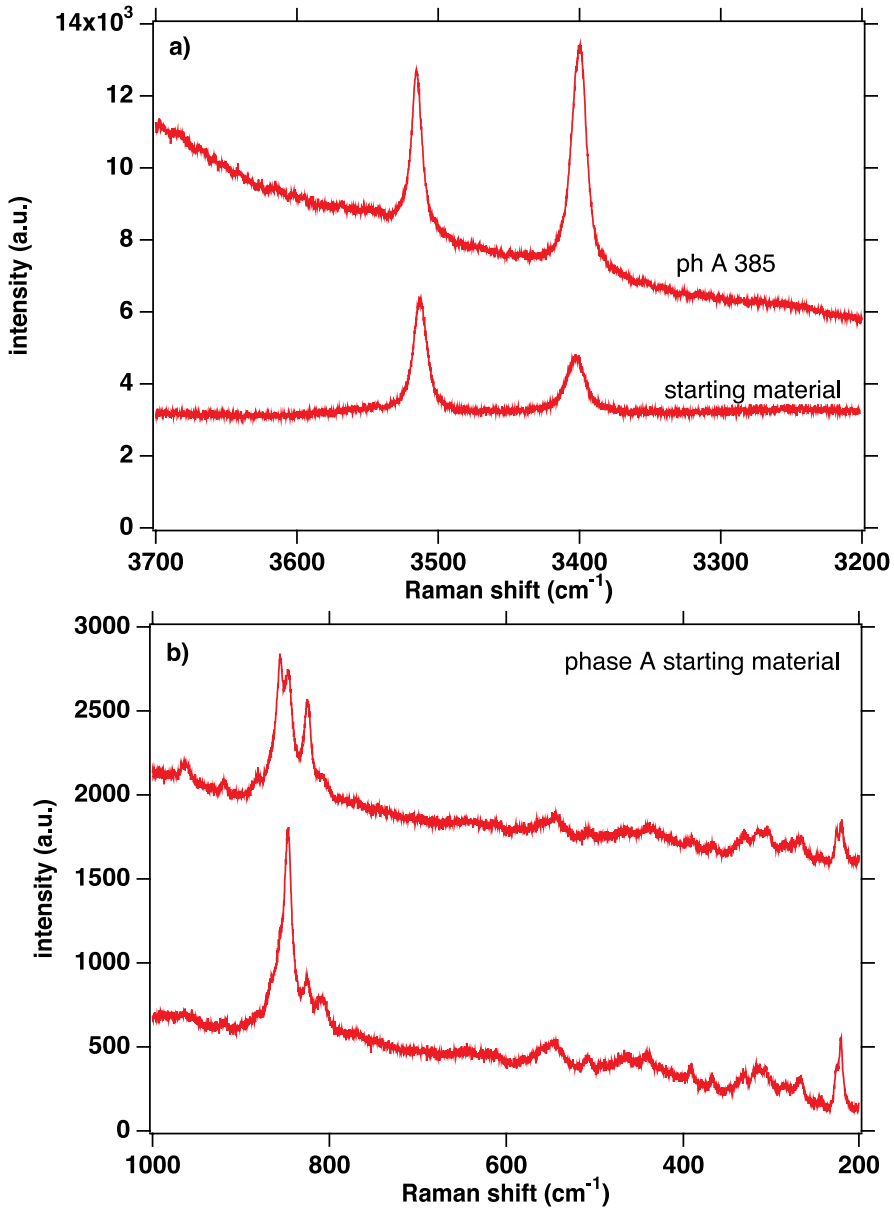

Fig. S1: Raman spectra of phase A a) from the starting material and product of run 385 in the OH stretching region; b) in the lattice vibration region; the spectra are different due to different orientations.

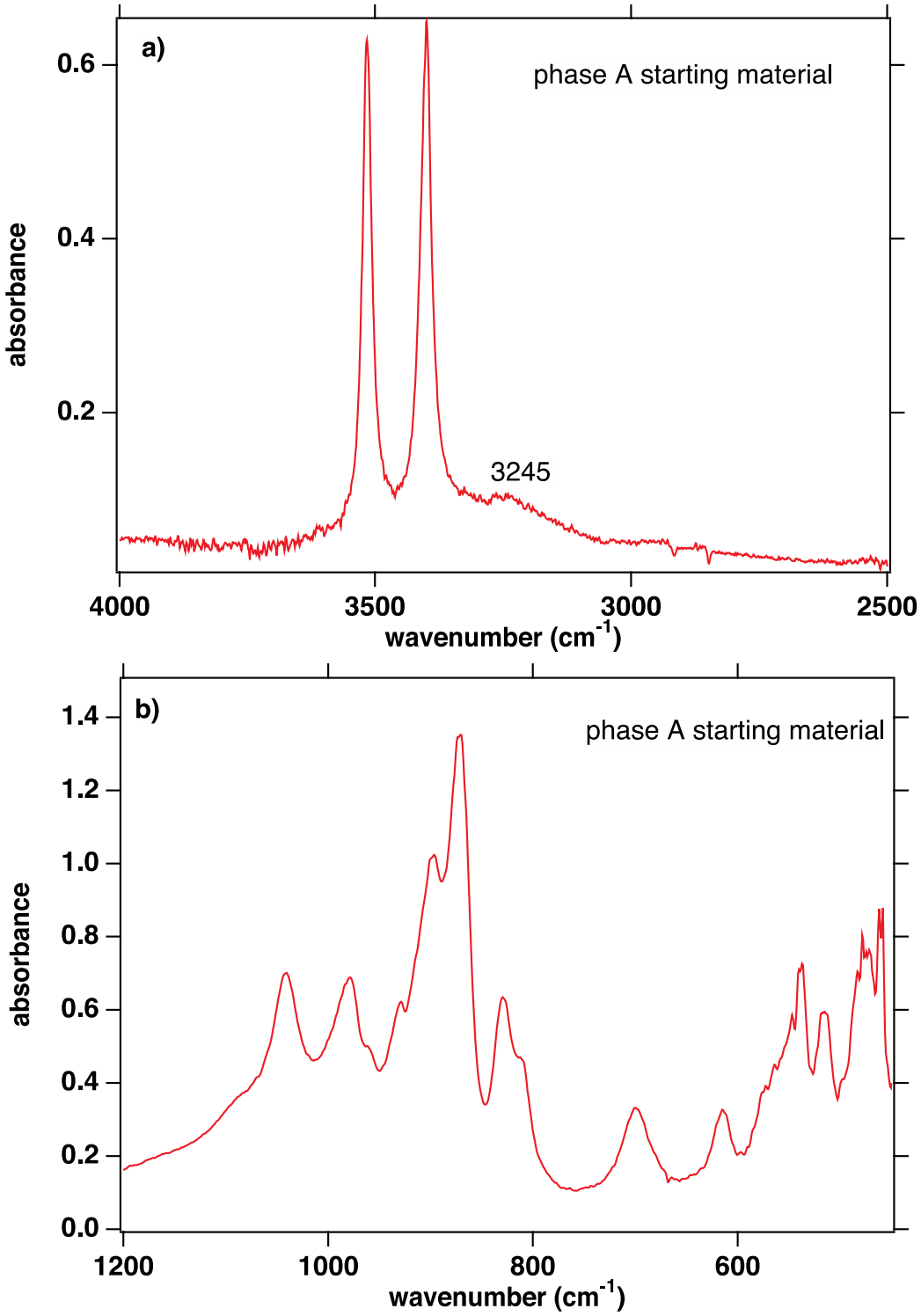

Fig. S2: FTIR spectra taken on a thin film of starting material phase A, a) in the OH region; b) in the MIR.

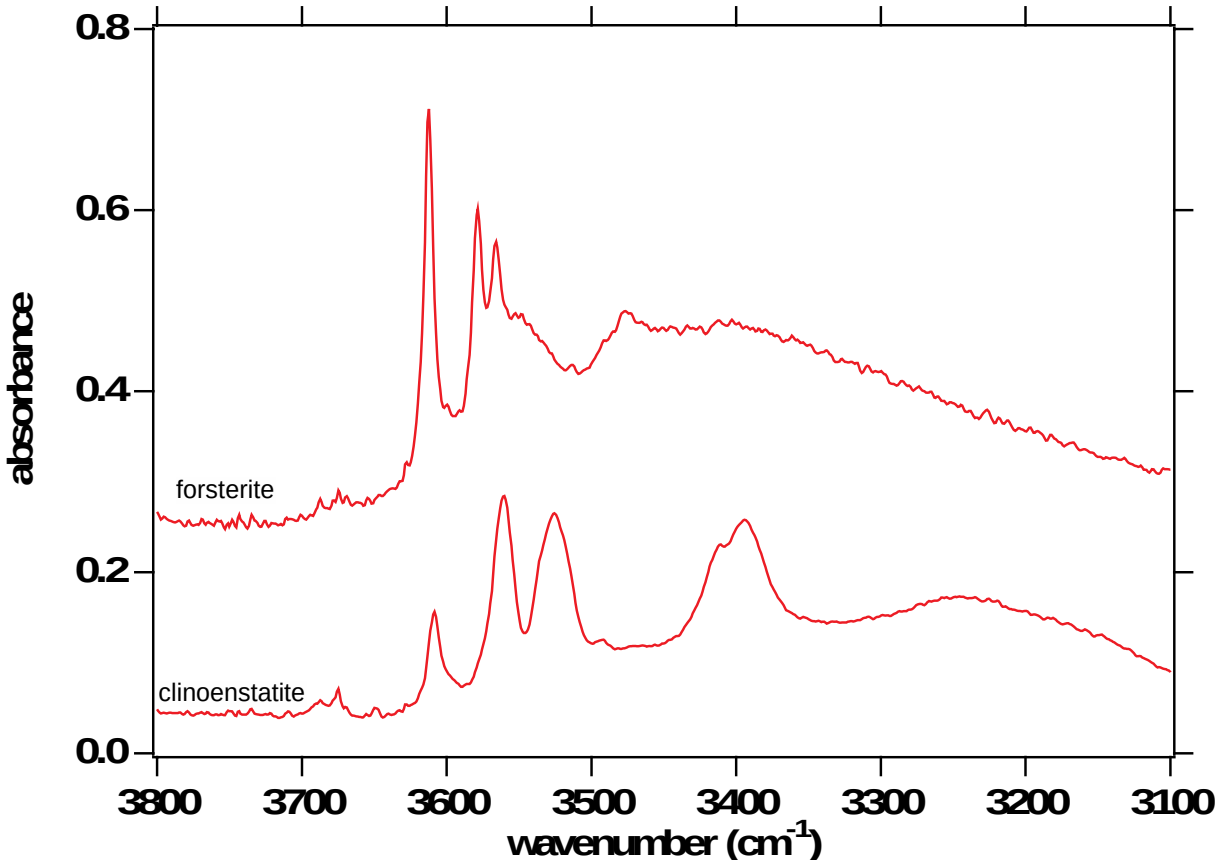

Fig. S3: FTIR spectra in the OH stretching region taken of recovered forsterite and clinoenstatite single crystals from run 501.

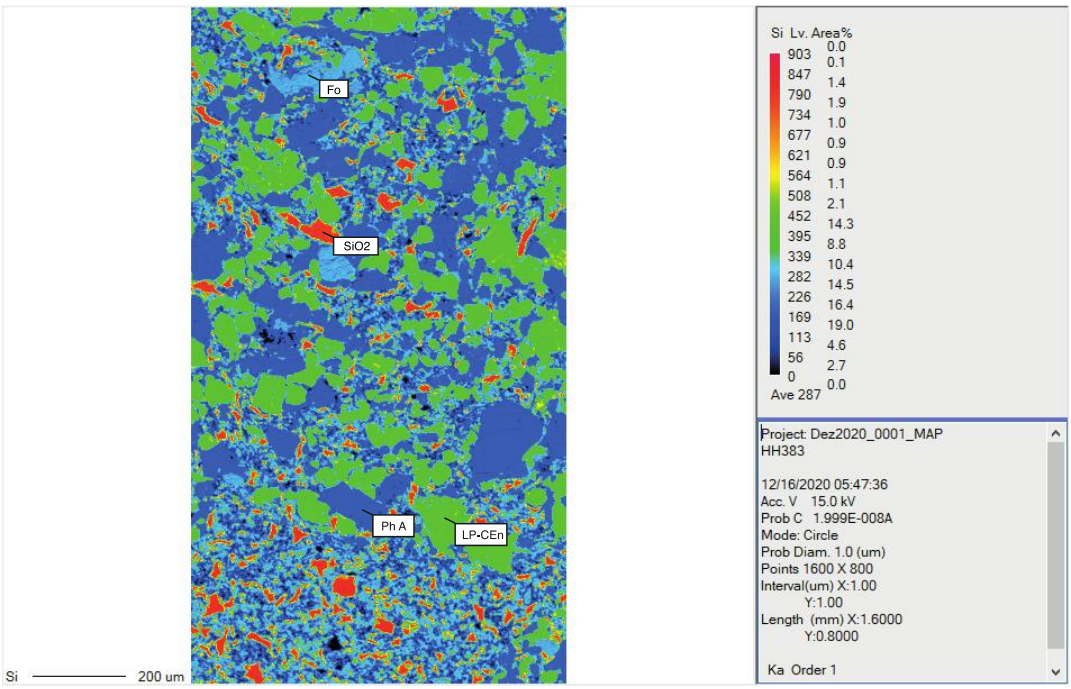

Fig. S4: Si element mapping of the recovered sample of run 383 (Table 1). The thermocouple broke and the temperature was estimated with T versus power calibrations obtained from the later performed experiments. The temperature was estimated to be below about 450 °C. A strong grain size-growth of phase A and Cen is visible, even the experiment lasted only about 40 minutes at 6.7 GPa. Forsterite is very fine-grained. Dark blue phase is phase A, light green is Cen (now LP-Cen), light blue forsterite, red is some SiO_2 quench. For some unknown reasons, only in this run the amount of SiO_2 quench is that high (see Fig S5, which only shows traces of SiO_2 quench phase). The reaction progress (growth of phase A and Cen) was estimated quantitatively to about 15%.

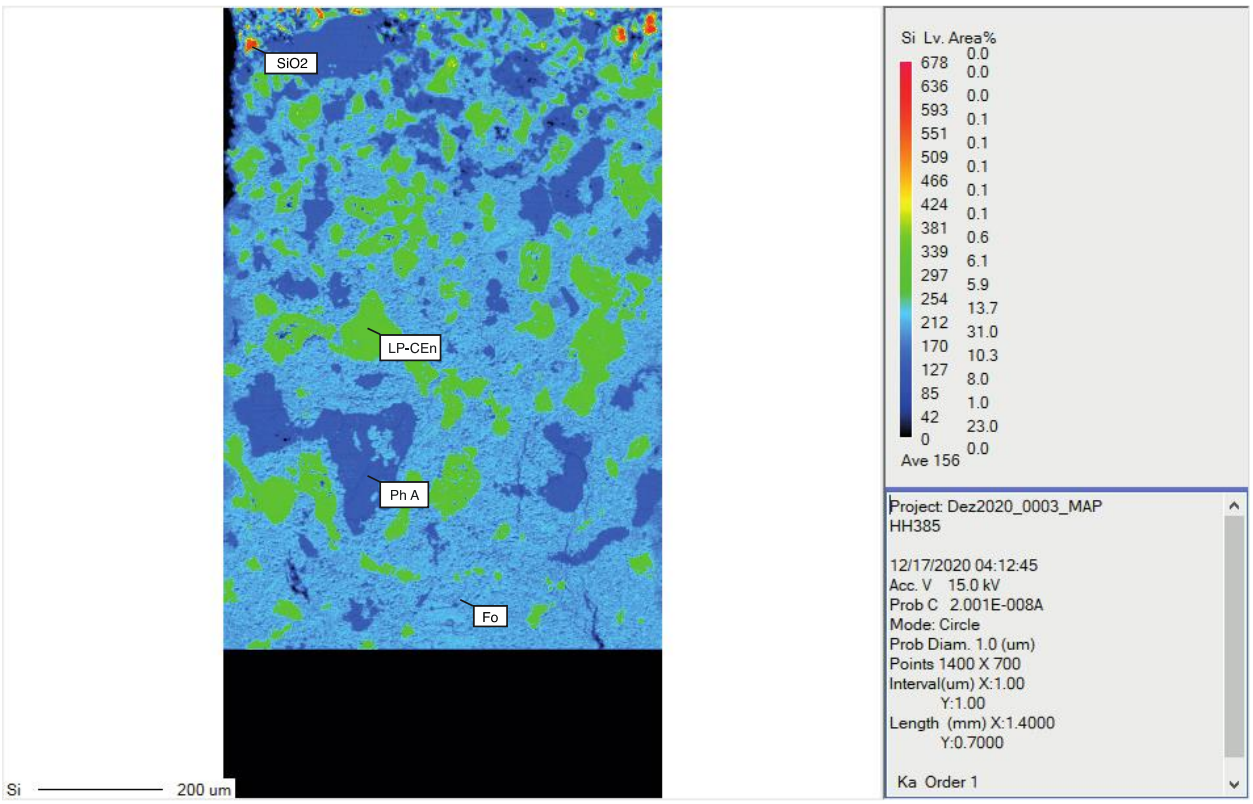

Figure S5: Si element mapping of the product phases of run 385 (Table 1). Dark blue is phase A, light blue is the newly formed forsterite, green corresponds to clinoenstatite and red is some quenched SiO_2. We interpret the observed grain-sizes as follows: in the first step, during T-increase to 700 °C and annealing for 40 minutes at this temperature strong grain-size coarsening of phase A and HP-clinoenstatite took place. In the second step, during annealing to 760 and 800 °C, nano-sized forsterite newly formed from phase A and HP-clinoenstatite.

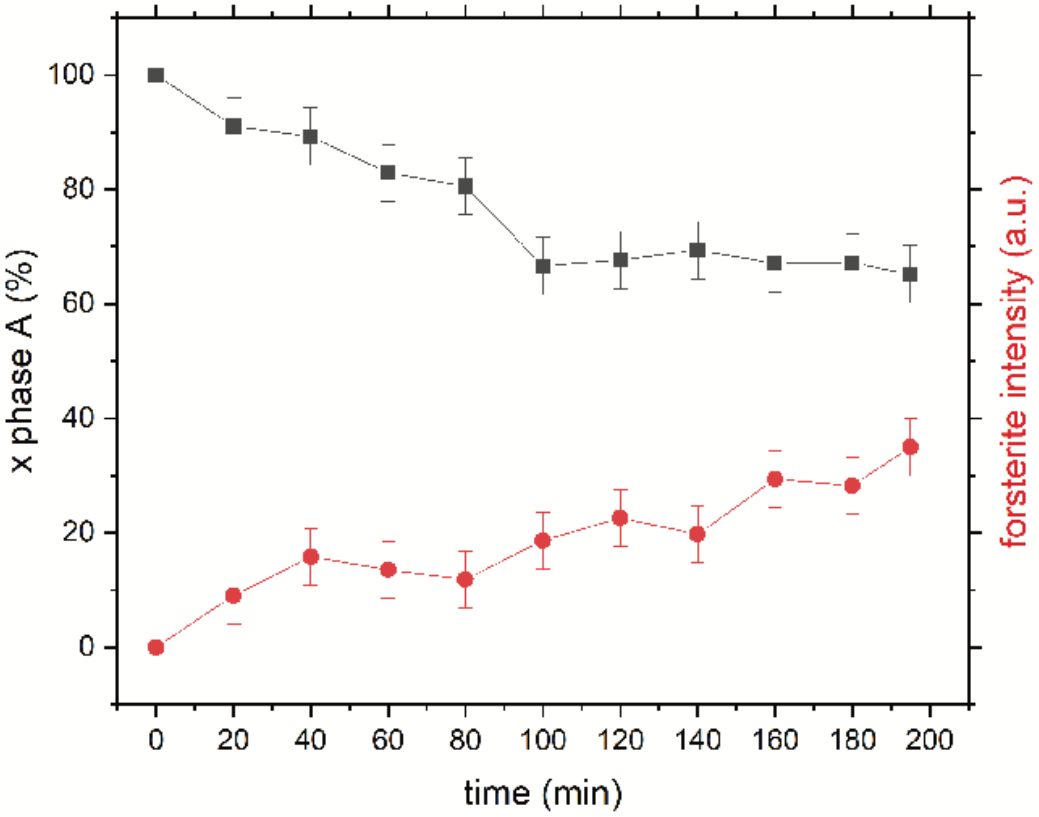

Fig. S6: Reaction progress in run 503 (Table 1) by comparing exemplarily the time-depending intensity change of phase A (reflection 110) shown as black squares and forsterite (reflection 021) shown as red circles. In this run, shortly after reaching 830 °C, 10.2 GPa, the synchrotron beam was shut down for 2 hours. During this duration, the temperature of the experiment was turned down to 200 °C (at constant P of 10.2 GPa) and increased to 830 °C, when the beam was available again. The reaction progress during the 2 hours at 200 °C is not shown here.

Table S1: Characterisation of the starting materials

Starting materials	*a* (Å)	*b* (Å)	*c* (Å)	*β* °	*V* (Å^3)	Space group
Clinoenstatite[1]	9.607(4)	8.817(3)	5.172(4)	108.35(5)	415.7(5)	$P2_1/c$.
Forsterite[2]	4.7549(1)	10.1958(2)	5.9810(1)	90.00	289.959(9)	*Pbnm*
Phase A[3]	7.8628(3)	7.8628(3)	9.5758(3)	90.00	512.70(3)	$P6_3$.

[1] Clinoenstatite was synthesized at 1 atm heating a $MgSiO_3$-glas at 1500 °C for 3 hours, followed by rapid quenching using the method described by Anderson and Browen (1914). The synthetic clinoenstatite was already described in Wunder and Schreyer (1997): Antigorite: High-pressure stability in the system MgO-SiO_2-H_2O. Lithos, 41, 213-227, 1997.

[2] Synthetic forsterite was provided by the Leibniz-Institut für Kristallzüchtung (IKZ). The sample contained 100 % forsterite.

[3] Phase A was synthesized at 8 GPa, 700 °C, 48 hours using a gel with the Mg/Si-ratio of 7/2 plus water in excess. The sample contained >0.99 wt % phase A and traces of periclase as determined by X-ray diffraction plus Rietveld refinement. Average of 16 electron microprobe analyses: $Mg_{6.89(4)}Si_{2.03(2)}O_{14}H_{6.1}$.