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In situ determination of the spinel–post-spinel transition in Fe_3O_4 at high pressure and temperature by synchrotron X-ray diffraction

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Abstract

The position of the spinel–post-spinel phase transition in Fe_3O_4 has been determined in pressure-temperature space by in situ measurements using a multi-anvil press combined with white synchrotron radiation. Pressure measurement using the equation of state for MgO permitted pressure changes to be monitored at high temperature. The phase boundary was determined by the first appearance of diffraction peaks of the high-pressure polymorph (h-Fe_3O_4) during pressure increase and the disappearance of these peaks on pressure decrease along several isotherms. We intersected the phase boundary over the temperature interval of 700–1400 °C. The boundary is linear and nearly isobaric, with a slightly positive slope.

Post-experiment investigation by TEM confirms that the reverse reaction from h-Fe_3O_4 to magnetite during decompression leads to the formation of microtwins on the (311) plane in the newly formed magnetite. Observations made during the phase transition suggest that the transition has a pseudomartensitic character, explaining in part why magnetite persists at conditions well within the stability field of h-Fe_3O_4, even at high temperatures. This study emphasizes the utility of studying phase transitions in situ at simultaneously high temperatures and pressures since the reaction kinetics may not be favorable at room temperature.

Keywords: Magnetite, post-spinel, phase transition, synchrotron experiment