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Oxygen fugacity control and measurement in hydrothermal diamond anvil cell

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Hydrothermal diamond-anvil cell (HDAC) has been applied in many experimental studies[1, 2]. However, despite of previous attempts[i.e., 3, 4], techniques for rigorous redox control and measurements in HDAC have not been developed. In this study, based on previous experiences[5], we first developed a technique for in situ quantitative measurement of H_2 in HDAC, such that new techniques for redox control in HDAC can be tested.

We used several different metal gaskets, including Re, Ir, Ni, Co, Mo, W, for pure H_2O samples in HDAC experiments, to see whether, at a fixed pressure-temperature condition, they can provide a steady-state H_2 pressure. Experiments, with and without H_2 in the flushing gas outside of the HDAC sample chamber, were performed to see whether the external H_2 has any influence on the redox state of the sample. In some experiments, a small quartz chip was added in the sample chamber as a pressure sensor[6].

Results show that dissolved H_2 in supercritical fluid at 500 ^{o}C, as well as quenched H_2 gas, was detected in W- and Mo-gasket chambers. But in Co- and Ni-gasket chambers, $H^{Â}_2$ can only be detected in the quenched vapor phase. In addition, the Raman peak area ratios of dissolved H_2 to water and the internal pressure in the Mo-gasket chamber decreased with the increase of reaction time; these trends did not seem to be affected by the flushing gas, with or without H_2. It indicated that Mo-gasket chamber cannot hold a steady-state H_2 pressure at 500°C. On the other hand, W gasket might be a possible choice for HDAC experiments when an extremely low oxygen fugacity condition is needed. The specific permeability of hydrogen through the gasket or diamond will be resolved in future work.

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