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Redox control and measurement in low-temperature (<450 °C) hydrothermal experiments

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Abstract

Redox control in hydrothermal experiments is routinely achieved through double-capsule and Shaw membrane techniques. These techniques control oxygen fugacity (f_{O_2}) by imposing a defined hydrogen fugacity (f_{H_2}) on a studied sample enclosed, together with H_2O, in a hydrogen membrane capsule made of Pt or Ag-Pd alloys. However, due to the low permeability of these membranes to H_2 at low temperatures (T), these techniques do not work efficiently below 450 °C. Here, we tested fused silica as a new hydrogen membrane and successfully applied it to monitor and control the redox states of studied samples at T down to 200 °C in hydrothermal experiments. Our results showed that 3, 8, 16, 36, 96, and 216 h are sufficient for a fused silica capillary capsule (FSCC) to reach osmotic equilibrium with the externally imposed 1 bar of H_2 at 350, 300, 250, 200, 150, and 100 °C, respectively, and H_2 pressures inside a FSCC was very close to the externally imposed values after osmotic equilibrium. By using FSCC as a hydrogen fugacity sensor, equilibrium H_2 pressures for Ni-NiO-H_2O and Co-CoO-H_2O redox buffer assemblages at 250–400 °C and 1000 bar total pressure were measured. The equilibrated f_{O_2} calculated are consistent with those derived from previous literature. Besides, FSCC can be used as a sample container, where f_{H_2} and f_{O_2} of enclosed samples can be continuously controlled. Furthermore, FSCC is an ideal container for sulfur-bearing samples, and its transparency allows spectroscopic analyses of the sample. Our work extended the low-T limit of previously well-developed redox control techniques and may open up a new research avenue in low-T hydrothermal experiments.

Keywords: Redox buffer and control techniques, hydrothermal experiments, redox reactions, hydrogen fugacity sensor, fused silica capillary