American Mineralogist, Volume 82, pages 337–344, 1997

Redox conditions in piston-cylinder apparatus: The different behavior of boron nitride and unfired pyrophyllite assemblies

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Abstract

Piston-cylinder experiments with C-H-O fluids in equilibrium with graphite as redox sensor were performed at 900 °C and 10 kbar. With this technique, it is possible to investigate the hydrogen permeability of the material surrounding the samples and to determine the hydrogen fugacity. The furnace assembly consisted of boron nitride or unfired pyrophyllite.

Solid organic compounds ($C_4H_4O_4$, $C_9H_{10}O_2$, and $C_{14}H_{22}O$) were used as starting materials. The different H/O ratios of these compounds (1:1, 5:1, 22:1) result in different initial hydrogen fugacities. The fluid formed during the experiments was analyzed by gas chromatography.

The experimental data demonstrate that boron nitride is nearly impermeable to hydrogen at the investigated conditions. The hydrogen fugacity observed in the gold capsules is controlled by the composition of the C-H-O starting compound. After approximately 1 h it is 340 bar for experiments with $C_4H_4O_4$, 2400 bar for experiments with $C_9H_{10}O_2$, and 3200 bar for experiments with $C_{14}H_{22}O$ and remains constant for at least 8 d.

The situation is different with unfired pyrophyllite as the pressure-transmitting material. In these experiments, the same hydrogen fugacity was determined irrespective of the compositions of the organic compounds. The hydrogen fugacity seems to be controlled only by the furnace assembly. It is approximately 470 bar after 3 d.

The hydrogen fugacity in two samples placed side-by-side in the boron nitride assembly and containing different starting compounds (H/O = 1 and 5) becomes identical during experimental durations of less than 2 d. This is because of the exchange of hydrogen through the adjacent capsule walls. This observation can be used to measure the hydrogen fugacity of any piston-cylinder experiment. In boron nitride assemblies, a second capsule with a C-H-O fluid and graphite can also be applied to control the hydrogen fugacity within given limits.