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Reduction in piston-cylinder experiments: The detection of carbon infiltration into platinum capsules

RICHARD BROOKER,[1,*] JOHN R. HOLLOWAY,[1] AND RICK HERVIG[2]

[1]Department of Geology, Arizona State University, Tempe, Arizona 85287, U.S.A.
[2]Center for Solid State Science, Arizona State University, Tempe, Arizona 85287, U.S.A.

ABSTRACT

Problems associated with intermittent and variable degrees of sample blackening are often reported for studies involving the preparation of CO_2-bearing silicate glasses in piston-cylinder apparatus. This phenomenon is generally attributed to H infiltration, which leads to the reduction of CO_2 and the precipitation of graphite with the concomitant formation of water. In this study we demonstrate that carbon diffusion into platinum capsules may be a common cause of blackened glasses and this process may be detected using fourier transform infrared spectroscopy (FTIR) to identify the presence of CO without elevated H_2O contents. The simulated infiltration of ^{12}C from a graphite furnace into a ^{13}C-bearing sample is illustrated using secondary ion mass spectroscopy (SIMS) and micro-FTIR analysis.

Careful FTIR monitoring of variable sample reduction has helped to identify the precautions required to reduce C (and H) infiltration in solid media assemblies and it appears that physical barriers can be more important than the chemical buffers traditionally employed.