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Quantitative Control and Measurement of Hydrogen Pressure in Hydrothermal Experiments: Insights from Five Decades of Research

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The precise control of redox conditions in hydrothermal experiments, whether through oxygen buffer or Shaw membrane techniques in cold-sealed pressure vessels, hinges critically on the quantitative regulation of hydrogen pressure (P_{H2}) within the sample chamber. This regulation is achieved via the effective permeation of H_2 through enclosed precious metal capsules, typically made of Pt or Ag-Pd alloys [1]. However, this approach has been largely confined to experiments conducted at temperatures above ~450 ºC, as these conventional hydrogen-permeable membranes lose their efficacy at lower temperatures.

To extend the scope of redox control in hydrothermal experiments to lower temperatures, two methods have been successfully developed:

1. **Fused Silica as a Hydrogen-Permeable Membrane**: The use of fused silica capillary capsules (FSCCs) allows for continuous control and variation of P_{H2} within the sample. This method enables in situ observation and monitoring of the sample through quantitative Raman spectroscopic analysis (Fig. 1) [2, 3].
2. **The Isopiestic Method in Ti-Pressure Vessels**: This technique is effective below 350 ºC with relatively low P_{H2}'s. FSCC P_{H2} sensor measurements after quench show that P_{H2}'s higher than those defined by the Ni-NiO-H_2O buffer cannot be maintained (Fig. 2) [4, 5].

Currently, both methods are limited to vapor-saturated pressures. Efforts to achieve effective control and measurement of P_{H2}'s in hydrothermal diamond-anvil cells are ongoing, though further advancements are still required [6].

References

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Figure 1. In situ Raman spectra illustrating temporal changes in Sn-Cl-H_2O species within a sample solution ($SnCl_4$ + HCl) enclosed in an FSCC. Two P_{H2} levels were fixed externally at 300 ºC. For details, refer to [3].

Figure 2. Schematic representation of the "isopiestic method" setup, as described in [4, 5].