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science under pressure

TECHNICAL NOTE

High-pressure high-temperature Fourier Transform Infrared Measurements at BL 1.4.3 of the Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA USA

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Diamond anvil cell samples are on a scale of tens of microns. When studying chemical reactions in inhomogeneous systems, the high spatial resolution of synchrotron source FTIR is extremely useful for pinpointing where in the sample reactions take place. The short working distance of most infrared microscopes makes in-situ measurements in the DAC extremely challenging.

While the **Diacell® µScopeDAC-HT(G)** has been designed to fit into most available FTIR microscopes, the need to monitor the pressure of the sample while continuing to make measurements at high pressures and high temperatures remains a difficulty. After all, moving a DAC with 8 water tubes, electrical leads, the tube carrying gas to the membrane, a grounding wire and a fragile thermocouple from the IR microscope to a fluorescence system is not an option.

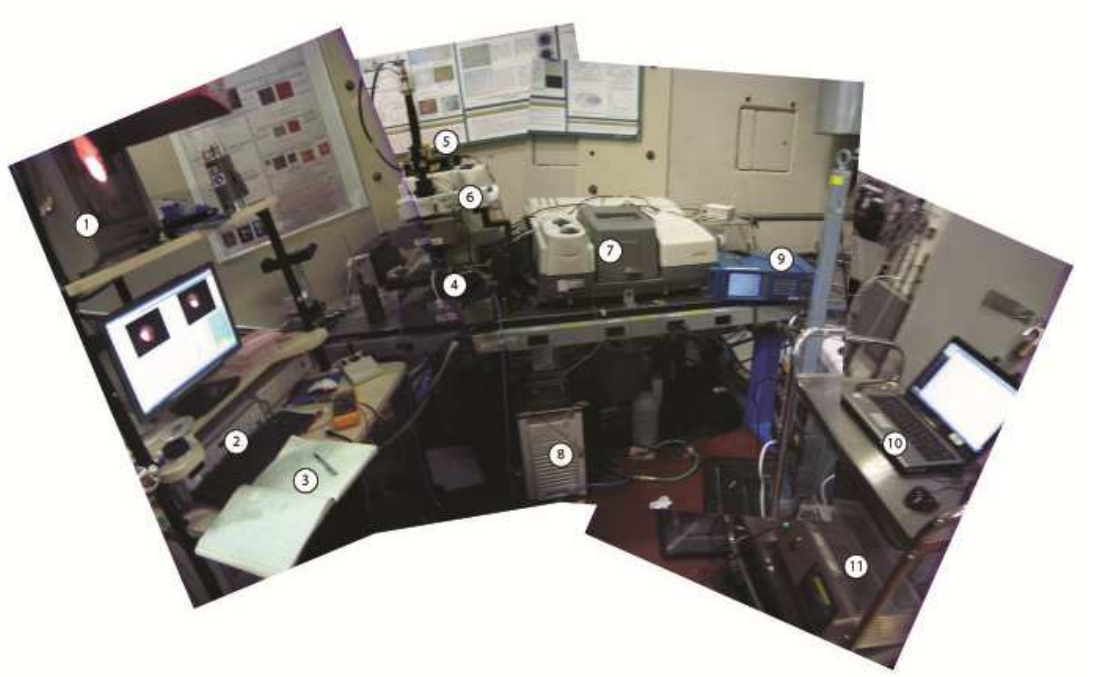

Figure 1: From upper left to lower right, the parts of a synchrotron-source high-pressure, high-temperature experiment: 1) television screen for in-situ monitoring of the sample. 2) computer controlling the IR microscope (note joystick controller to right. 3) Labbook. 4) **Diacell® μScopeDAC - HT(G)** 5) custom **Optiprexx™ PLS** (on loan from Daresbury Laboratory and supplied by **Almax easyLab**) 6) Nicolet Continuum XL IR Microscope. 7) Synchrotron-source Thermo Nicolet Nexus 870. 8) Chiller. 9) DAC pressure controller. 10) Ruby fluorescence readout. 11) DAC Heater controller.

Recently, we successfully set up and carried out high spatial-precision heated high pressure experiments at BL 1.4.3 of the Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA USA, with support from BL 12.2.2 of the ALS and Dr. Ray Jones of Daresbury Laboratory, UK (now STFC). Typically, we were able to reach pressures in excess of 12 GPa and temperatures higher than 350 degrees C.

By incorporating an **Optiprexx™** custom-made ruby florescence system (from **Almax easyLab**) into the optical path of the Nicolet Continuum XL IR microscope, we were able to monitor pressure changes without moving the diamond anvil cell, which enabled us to perform heated experiments. Once the entire system was in place, a single scientist could monitor the experiments alone - all critical controls were within an arm's reach. (Essential swivelling chair not pictured.) Using this system, we were able to begin the study of the interactions between organic acids and silicate minerals.

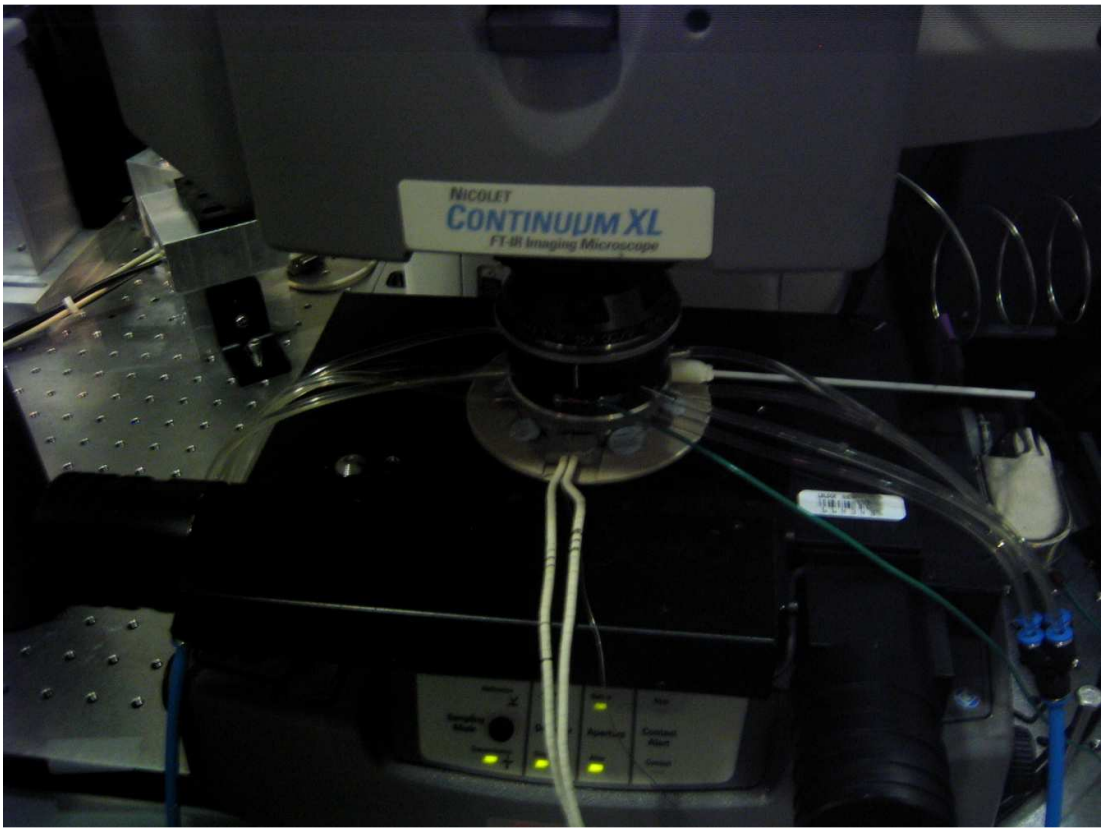

Figure 2: The **Diacell® μScopeDAC -HT(G)** is shown close up; because of the multiple connections for water, gas, heating and thermocouple, adhesive clay was used to hold the cell in place.