SUPPLEMENTARY MATERIAL

Design and construction schemes of the new hydrothermal moissanite cell apparatus

Masotta M.[1], and Keppler H.[2]

[1]*Dipartimento di Scienze della Terra, Università degli Studi di Pisa - Via S. Maria 53, 56126 Pisa, Italy*
[2]*Bayerisches Geoinstitut, Universität Bayreuth, D-95440 Bayreuth, Germany*

This supplementary material provides information on the new hydrothermal moissanite cell and its operation. Figure A1 gives an overview of the components that have been designed specifically to perform experiments at high pressure and high temperature. A detailed description of these components and of the operation of the cell is given below.

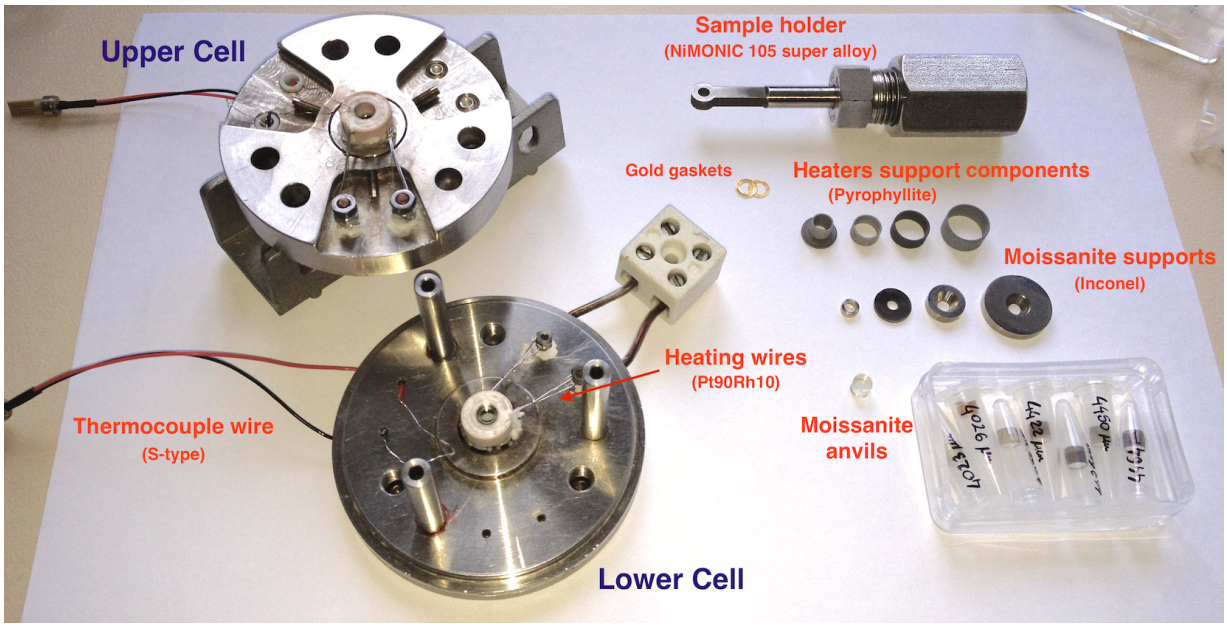

Figure A1. Overview of the components designed for the hydrothermal moissanite cell. The outer steel ring enclosing the entire cell is not shown.

Sample holder

The sample holder was machined from a single piece of the NIMONIC 105 super alloy, to resist high fluid pressure (up to 2000 bar) and high temperature (up to 950 °C). To make this part, a 100 µm diameter capillary hole was first spark-eroded into a block of the super alloy. Then the sample chamber (diameter of 3.5 mm and height of 4.0 mm) was made by drilling into the piece, such that the hole intersected the capillary. Finally, the block was machined to the final shape, with a truncation and thread on the side opposite to the sample chamber, in order to fit into a capillary connector (Figure A2).

Support parts for heating wires and moissanite anvils

Heating is provided by three concentric coils of $Pt_{90}Rh_{10}$ heating wires per anvil. Heating wires of 0.30 mm diameter are wrapped around three concentric pyrophyllite parts (Figure A3). In order to improve the durability of the pyrophyllite parts during the experiment, they are fired by slowly heating to 1100 °C at a rate of 100 °C/hour. After firing, the pyrophyllite parts are cemented on top

of two Inconel plates. These plates support the pressure applied to the cell. However, the metal increases heat dissipation during the experiments. The moissanite anvils are placed directly on the Inconel plates; a smaller Inconel ring fitting in the inner pyrophyllite part is used as spacer to raise the moissanite at about one millimeter above the inner pyrophyllite part. The thermocouple is attached with some cement in this space, as close as possible to the top of the moissanite. Gold gaskets rings (150 µm thick) are placed on top and bottom of the moissanite anvils in order to prevent the anvil from breaking and to improve sealing of the sample holder at high pressure.

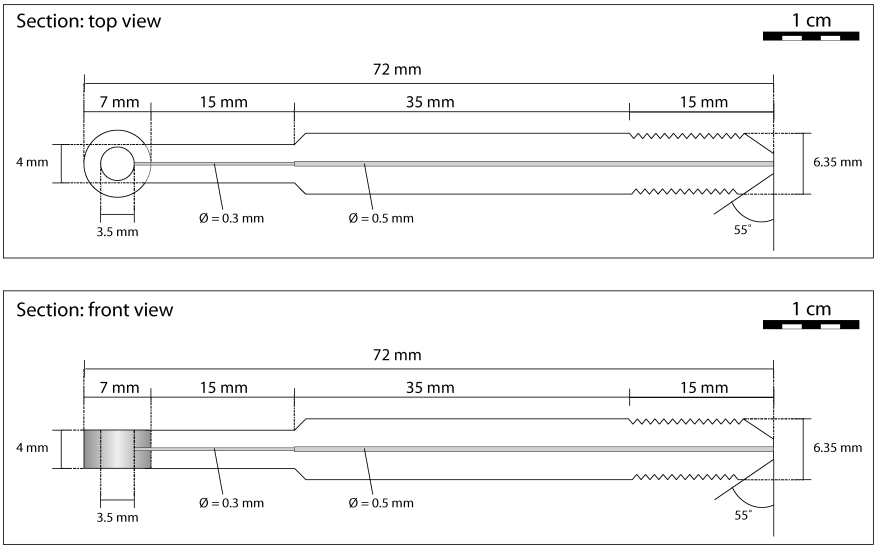

Figure A2. Design of the NiMONIC 105 super alloy sample holder.

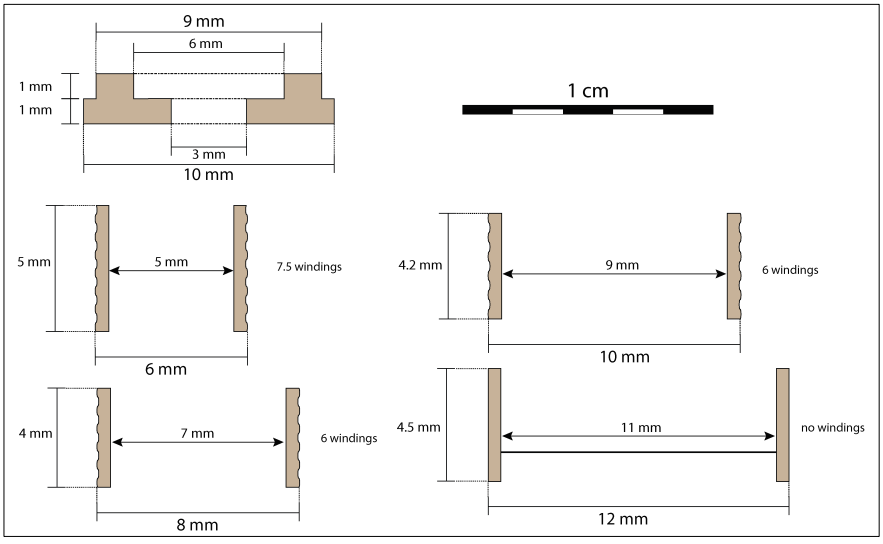

Figure A3. Design of the pyrophyllite parts supporting the heaters.

Experimental procedure

Similar to experiments in the regular moissanite cell, the sample is prepared in the form a glassy disk (100 µm thick, up to 3 mm diameter). In order to prevent deformation of the sample and to keep it at the same focal distance, the sample is placed between two quartz cylinders with polished surfaces that fill the volume of the holder. To prepare the cell for the experiment, the sample and the quartz crystals are placed on top of the lower moissanite and the sample holder is lowered down from above. The upper cell is then placed on top of the sample chamber and the entire cell tightened

by three screws (Figure A4). In the last step, zirconia wool is filled into the empty space around the heaters and close the cell is enclosed by a steel ring. The cell is finally placed under the microscope and connected to the external pressure line and the heating control system (Figure A5). Before heating, pressure is manually increased to the desired experimental pressure by activating the spindle motor and then hold for few minutes in order to check for water leaks.

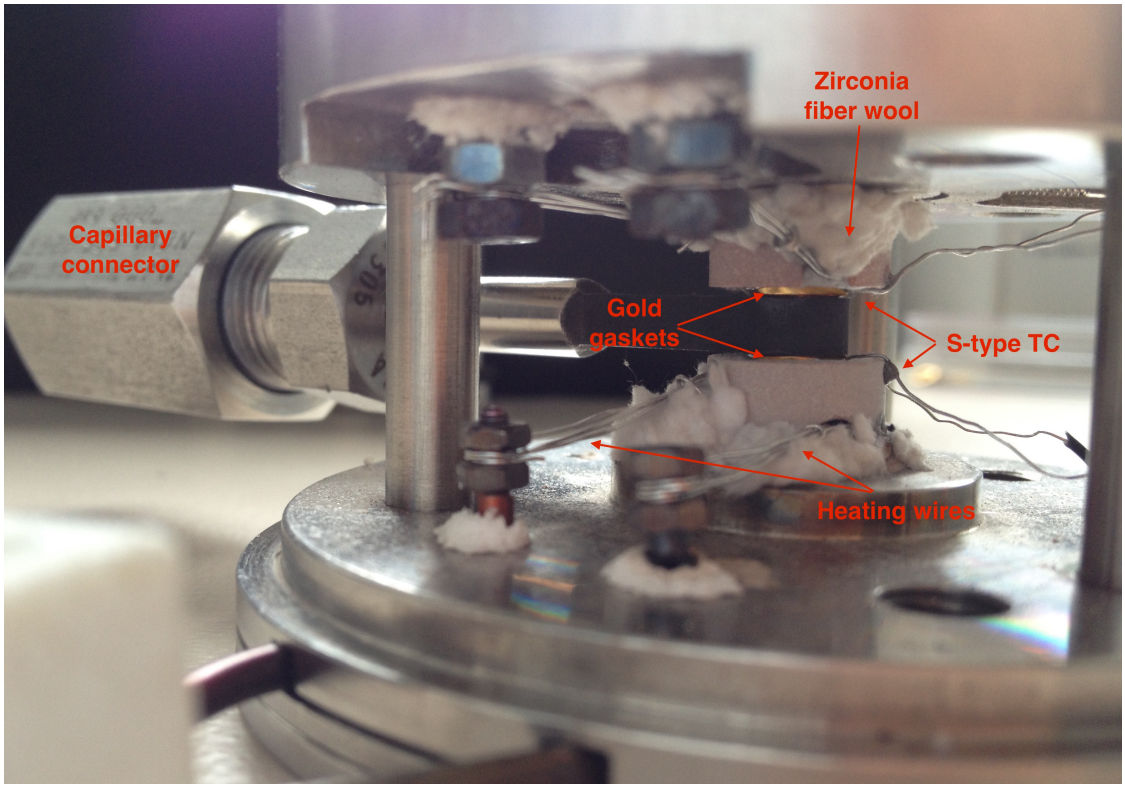

Figure A4. Image of the hydrothermal moissanite cell after loading with the sample and closing. The steel ring around the entire cell is not yet installed.

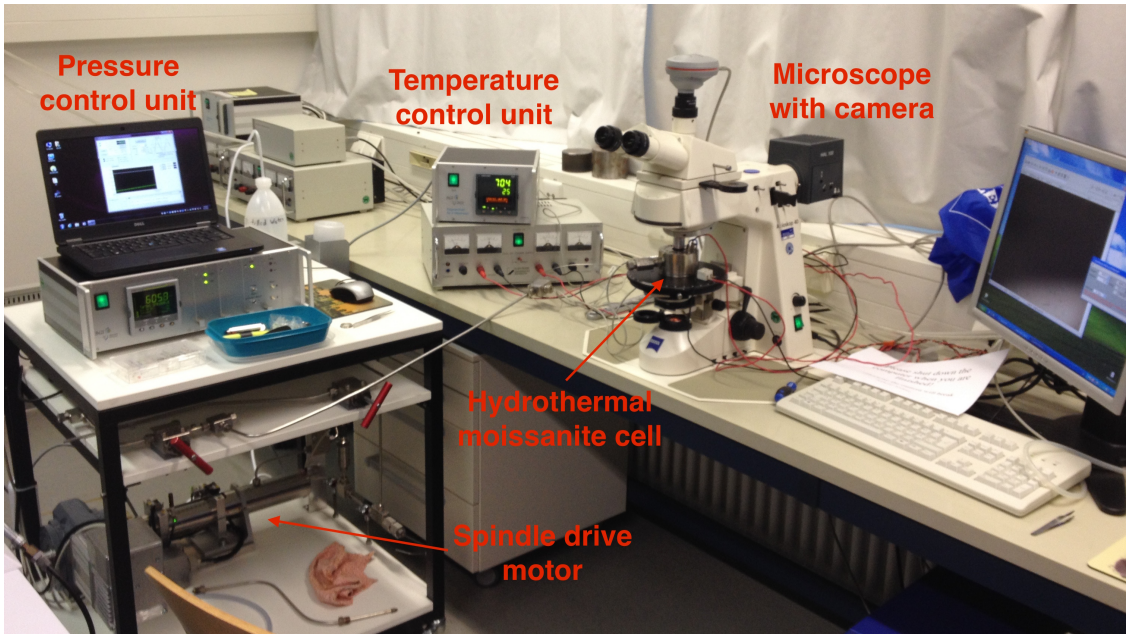

Figure A5. Image of the experimental setup.

Temperature calibration

The temperature inside the sample chamber was calibrated against the temperature measured with the two thermocouples attached to the moissanite crystals, using an external thermocouple inserted through a hole drilled in the upper moissanite crystal (Figure A6).

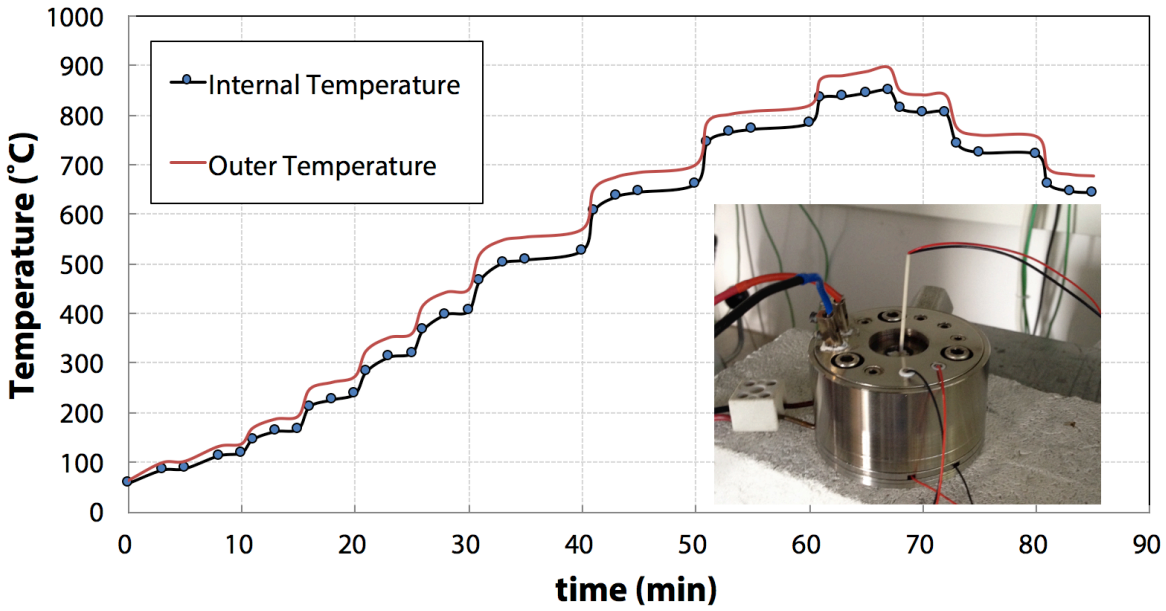

Figure A6. Chart of internal temperature calibration and configuration of the hydrothermal moissanite cell during temperature calibration (inset).

Videos of the experiment HGT-2

Two videos of the experiment HGT-2 are attached as supplementary material. Video 1 shows the effects on bubbles of the sequence of pressure oscillations (200 bar amplitude and period of 30 seconds; the video is accelerated to 30x). Video 2 shows the two stage of compression (to 800 bar)-decompression (to 200 bar) at the two different decompression rates of 50 and 200 bar/min (the video is accelerated to 60x). Detailed discussion of the experiment are given in the text.

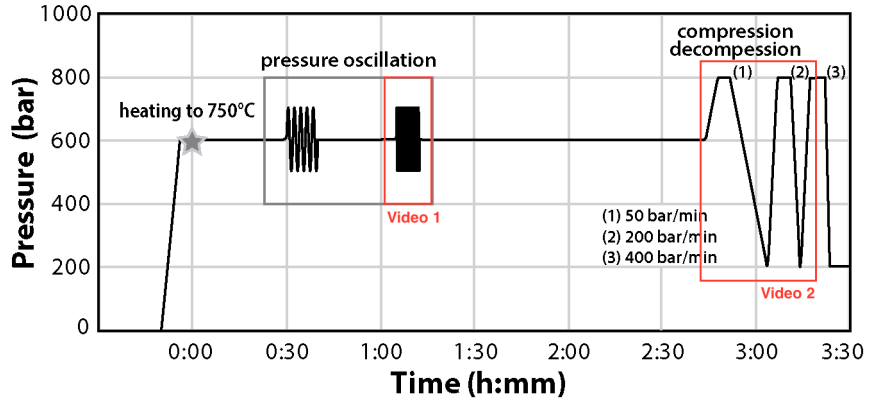

Figure A7. P-t path of the experiment HGT-2 showing the two cycles and the three decompression paths. The red boxes indicate the time intervals of the two videos.