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Experimental technique for re-equilibration studies of water related species through quartz

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Due to the possibility of post-entrapment compositional and density changes of fluid inclusions, the analyses of fluid properties have to be performed with particular attention. Re-equilibration processes are not yet fully understood, therefore further investigations are required to characterize all aspects of post-entrapment changes in fluid inclusions. We design our recent experimental work to test the behaviour of aqueous fluid inclusions in Brazilian quartz crystals under conditions of different pressure (Δp) and different fugacity (Δf) of water related species at a variety of temperatures.

For our purpose fluid inclusions are synthesised according to the method of Bodnar and Sterner (1987) by healing fractures which are induced by thermal shock. Fracture healing experiments, to synthesize fluid inclusions, are conducted in annealed gold capsules having an inside diameter of 3 mm and a length of 40 mm to prevent chemical intersections with the autoclaves. In our hydrothermal laboratory there are 10 autoclaves installed, in a vertical position, which are specially designed for the synthesis of fluid inclusions. Starting materials (quartz-rods) are drilled in specific crystallographic orientations. To minimize the influence of birefringence of laser light in Raman spectroscopic analyses after the experiments, rods are orientated parallel to the c-axis, according to Baumgartner and Bakker (2009).

The *P*-*T*-path followed from room temperature to final run conditions may affect fluid inclusion properties, therefore we heat up and cool down the pressure vessel along the specific isochore such that the specific volume of the fluid doesn't change. This can only be achieved with the use of an internal thermo-couple that measures the temperature directly at the sample surface. An example is illustrated in Figure 1.

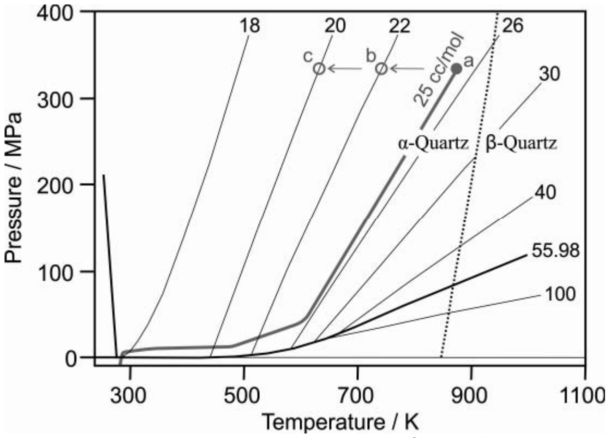

*Fig. 1. Isochore diagram (in cm^3 mol^{-1}) of H_2O. Point **a** is the synthesis of H_2O inclusions at 337 MPa and 873.15 K which correspond to a molar volume of 25 cm^3 mol^{-1}. The grey thick line illustrates the heating and cooling conditions of the experiment.*

During the experiment, both temperature and pressure are controlled by a computerized operating system that is able to stabilize the conditions within 1 °C and 2 MPa (Fig. 2). The maximum temperature and pressure of the hydrothermal experimentation are 700 °C and 1 GPa (Argon pressure).

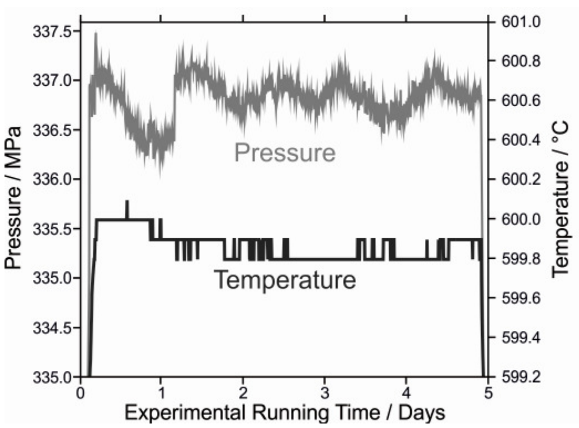

Fig. 2. Experimental run conditions with a running time of 5 days.

Fluid inclusions of known composition and density are synthesised in carefully selected inclusion-free Brazilian quartz crystals in cold seal pressure vessels under hydrothermal conditions. For performed re-equilibration studies see Doppler et al., this abstract volume. A collection of natural quartz samples with well-defined natural fluid inclusions (mixture of $NaCl-H_2O-CO_2$) is also available for experimental diffusion studies (see Baumgartner et al., this volume). After the initial experiment, the quartz cores are cut into disks of ~2750 µm in diameter and a thickness of ~500 µm, which are then polished on both sides for microscopic, microthermometric and Raman spectroscopic investigations. About 100 fluid inclusions of variable sizes, shapes and distances to the surface of each quartz disc are analyzed. We measure the T_h and T_m by using a Linkam heating-freezing stage (LINKAM MDS 600 stage and LINKAM THMSG 600 stage). The composition of the fluid inclusions is measured by an ISA JobinYvon LABRAM confocal Raman spectrometer. The morphological properties of each fluid inclusion are characterized by the total area and the perimeter. The area fractions of the vapour bubble are measured with the ImageJ software by tracing digitally around the outside edges of the shadows that define the perimeters of the bubble and the inclusion at room temperature (Bakker and Diamond, 2006).

The quartz disks with synthesized fluid inclusions are subsequently used for re-equilibration experiments. To prevent any damages during the re-equilibration experiment, we place the fragile sample in between two quartz spacers (Fig. 3).

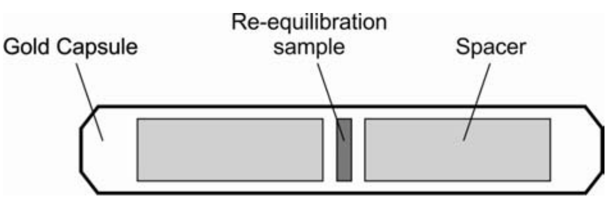

Fig. 3. Illustration of the setup of the filled gold capsule of a re-equilibration experiment.

Re-equilibration experiments are defined by four parameters: 1. fugacity of fluid components; 2. fluid pressure; 3. temperature; and 4. time. Re-equilibration conditions at the same temperature and pressure as the initial conditions (point **a** in Fig.1) can be performed with gradients in fugacities (or concentrations). Diffusion can be provoked without pressure differences between inclusions and the hydrothermal fluid in the capsule. Re-equilibration experiments can also be performed by varying temperature (point **b** and **c** in Fig. 1) and/or pressure, with and without a gradient in fugacities. Careful analyses of the same fluid inclusion assemblages that were selected after the synthesis, may reveal changes in shape, size, position, and fluid properties (V_m and x). The main goal of these experimental studies is to find a relationship between the parameters (including gradients in fugacity and pressure) and the fluid inclusion alterations, in order to predict the behaviour of natural fluid inclusions in specific rock exhumation P-T-t paths.

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