## Re-equilibration studies of synthetic fluid inclusions in quartz

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Synthesised fluid inclusions in carefully selected inclusion-free Brazilian quartz crystals are used for re-equilibration experiments. Due to the possibility of post-entrapment changes of fluid inclusions, interpretations of fluid properties have to be done with particular attention. Therefore we perform re-equilibration experiments in cold seal pressure vessels under hydrothermal conditions (for applied experimental technique see Doppler et al., this abstract volume). The experiments are carried out at high experimental temperatures and pressures (max. 700 °C and 1 GPa). The experimental studies allow investigating the main factors that affect  $H_2O$  diffusion through nominally anhydrous minerals.

The present study illustrates the changes of primary synthesised  $H_2O$ -rich fluid inclusions which were exposed to  $D_2O$  during the reequilibration process at high experimental temperatures and pressures.

The presence of  $D_2O$  in  $H_2O$ -rich fluid inclusions is detected with microthermometry and Raman spectroscopy. The melting temperatures of pure  $H_2O$  and pure  $D_2O$  are significant different, 0.0 °C and +3.8 °C, respectively. Intermediate melting temperatures can be directly reflected to relative amounts of  $H_2O$  and  $D_2O$ . Furthermore, the Raman spectrum of  $H_2O$  and  $D_2O$  are similar in shape, however, they occur in completely separated wavenumber intersections: 3000 - 3800cm<sup>-1</sup> for  $H_2O$  and 2000 - 2800 cm<sup>-1</sup> for  $D_2O$ .

A variety of parameters that play an important role in diffusion experiments are examined: 1) concentration gradient (chemical potential) of  $H_2O$  (negative) and  $D_2O$  (positive); 2) distance of fluid inclusions to the quartz surface; 3) size of fluid inclusions; 4) experimentation time; (Fig. 1).



Fig. 1. Schematically illustrated quartz crystal with primary synthesised fluid inclusions with known composition at **a**. variable distances to the crystal surface and **b**. variable sizes. Re-equilibration experiments are performed with a different fluid phase at the same P-T conditions and different experimentation times.

The original synthesised inclusions have a large variety of shapes (Fig. 2, definitions according to Bakker & Diamond, 2006). After reequilibration, the original equant and regularly shaped inclusions do not change their shape, whereas elongated and irregularly shaped inclusions tend to become equant and regularly shaped during re-equilibration (arrow in Fig. 2).



Fig. 2. Shape classification diagram (modified after Bakker & Diamond, 2006).

Analyses of re-equilibrated inclusions at variable distances to the crystal surface allow us to obtain concentration profiles in quartz. Concentration profiles of the  $D_2O$  content of fluid inclusions in quartz allow us to develop new three-dimensional diffusion models.

An example of а re-equilibration experiment is shown in Figure 3. The primary fluid inclusions have been synthesised with pure H<sub>2</sub>O at 600 °C and 337 MPa, which corresponds to a molar volume of 25 cm<sup>3</sup> mol<sup>-1</sup>. The experimental running time for the first synthesis was nineteen days. The primary synthetic fluid inclusions were re-equilibrated with pure D<sub>2</sub>O at the same P-T conditions for one, five and nineteen days. An example of a concentration profile, as evidenced by the melting temperature of ice  $T_m(SV \rightarrow LV)$ , within a quartz crystal after five days is illustrated in Figure 3.



Fig. 3. Detected concentration profile as a function of distance from the surface of a quartz crystal (after an experimentation time of five days).

The performed re-equilibration experiments carried out at different running times illustrate a shift of the envelope to higher  $T_m$ values, i.e. D<sub>2</sub>O-richer, as it is shown in Figure 4.



*Fig. 4.* The increasing running time of reequilibration experiments illustrates the development of the concentration profile of the measured fluid phases.

Potential changes of the ambient fluid inclusions during re-equilibration correlate with the size (total volume) of the fluid inclusion. Figure 5 demonstrates again three envelopes of the detected melting temperatures of re-equilibrated fluid inclusions.



Fig. 5. Dependency of the size of the fluid inclusion to potential changes in composition during reequilibration experiments.

These experiments have illustrated that  $H_2O$  and  $D_2O$  can diffuse relatively fast trough quartz at 600 °C and 337 MPa. Variation in reequilibration is controlled by distance to the quartz surface, size of the fluid inclusion and experimentation time.

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