

FORMATION TIME OF SYNTHETIC INCLUSIONS: EVIDENCE AND INCIDENCES

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The objective of the fluid inclusion study is to determine the P-T-X conditions of fluid trapping. However, the time of inclusion formation is never clearly known. Petrography with the help of cathodoluminescence can allow us to describe the paragenetic succession and then the different generation of fluids responsible for mineral precipitation. But inclusions in crystal growth zones can be trapped later and cannot be always considered as synchronous to crystallogenesis. The question about the relation between inclusion and mineral formation is important, as exemplified by the problematic timing of oil migration versus diagenesis in petroleum geology.

One way to investigate the trapping time of fluid in minerals is to develop an experimental approach. Aqueous fluid inclusions have been synthesized within fluorite microfractures ($T=200^{\circ}\text{C}$, $P=P_{\text{sat}}$, $t<30$ days), quartz microfractures and quartz overgrowths ($T=400$ to 300°C , $P=400$ bars, $t<20$ days).

Fluorite crystals and a $\text{NH}_4\text{Cl-H}_2\text{O}$ solution (18 g/l NH_4) were enclosed in a gas-pressure autoclave. The temperature of the vessel attained a regulated temperature of 200°C within a few hours. The duration of the runs was 720 hours. The autoclave was purposely allowed to leak slowly, which results in a linear increase in NH_4Cl content of the solution with time.

About 1-3 ml of solution was collected during each sampling period (1, 8, 15 and 30 days). All samples were extracted from the sample port (liquid phase) and trapped in an inox tube and freeze into a liquid nitrogen dewar. Valves and tubes were heated at 200°C before using the N_2 dewar. The solutions were analyzed at room temperature by ion chromatography to determine NH_4 concentration. Significantly due to the small sampled volume of the solutions, sampling does not affect the composition of the bulk solution. Indeed, blank experiments have shown that the composition of the sample is equivalent to the composition of the remaining solution in the autoclave. Pressure and temperature were readjusted after each extraction.

Quartz experiments were conducted in a fluid pressure autoclave ([®]Autoclave Engineer), utilizing gold capsules a 20 day runtime (Landais et al., 1989). Each sample was prepared with a quartz crystal, an $\text{H}_2\text{O-NaCl}$ (1M) solution and silica gel. Temperature and pressure were adjusted at 400°C and 400 bar respectively. After stabilization, pressure was maintained at 400 bar during all the experiments time while temperature was gradually reduced from 400 to 300°C to enhance quartz precipitation and fluid inclusion formation. The cooling is isochoric and occurs on one p-T path.

NaCl solutions were used to increase the solubility of quartz and thus enhance the fluid inclusion formation (Xie and Walther, 1993). The maximum trapping temperatures of the fluid inclusions are thus lower than the critical temperature (425°C) of the NaCl (1M)- H_2O system.

The experiments were designed to evaluate how much time was necessary to form the inclusions - to the minute for fluorite experiments and to the day for quartz experiments. The time for fluorite experiments is estimated applying a microleakage to the autoclave inducing an increase of the fluid salinity with time. For quartz experiment the temperature variation of the autoclave with time induces variations of T_h of the inclusions, which can then be used as markers of time. The time needed to heal microfractures or create overgrowths is estimated by measuring the melting temperature (T_m) of the inclusions in fluorite and the homogenization temperature (T_h) of the inclusions in quartz.

For the fluorite experiments, healing does not appear to be a regular process along the microfracture. Fluorite experiments mimic boiling processes occurring in hydrothermal or epithermal natural systems and show how a boiling fluid may be progressively salted- during vaporization and trapped as fluid inclusions.

For quartz experiments, the quartz/ $\text{H}_2\text{O-NaCl}$ solution equilibrium is reached for the synthetic quartz in the 1st day and for natural quartz in the 6th day. This difference is linked to the hydration state of the two types of quartz. The decrease in T_h of synthetic fluid inclusions from the core-overgrowth boundary to the external part of the quartz overgrowth shows that the growth of quartz is a progressive and protracted process, which involves a silica supersaturated solution.

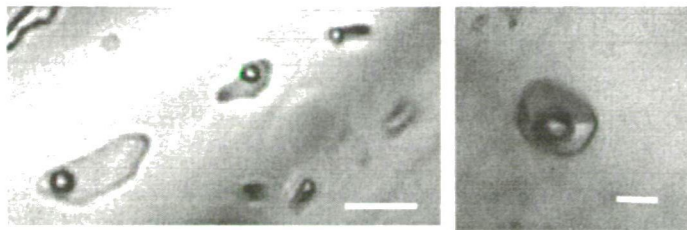


Figure 1: Synthetic inclusions created in fluorite (left) and quartz (right). Bar scale : 10 μm .

Experiments with a two-phase fluid (oil + water) have produced petroleum inclusions in quartz overgrowths. The objective of this set of experiments is to decipher the geochemical messages of fluid inclusion and isotope analyses of quartz. Experiments were carried out in a fluid-pressure autoclave up to 350°C and 400 bar. Petroleum and aqueous inclusions in quartz overgrowths have been created with a water/oil proportion of 50%. SIMS measurements show an enrichment in $\delta^{18}\text{O}$ of the quartz precipitating in the presence of petroleum. Overgrowths created in a water-salt system from a water solution with a $\delta^{18}\text{O}$ of -7.8‰ have an average $\delta^{18}\text{O}$ of -2‰ whereas overgrowths created in a water-salt-oil system have a $\delta^{18}\text{O}$ of +9‰. This experimental approach shows that fluid inclusions can be representative of quartz precipitation and the oil invasion of a reservoir can be marked in the isotope signature of the quartz.

Such study has implications for how we undertake microthermometric analysis and how fluid inclusions should be selected for analysis with respect to the fluid inclusion assemblage (FIA) principle. Synthetic fluid inclusions within healed microfractures are representative of the experimental conditions. The compositions of the fluid inclusions (T_m) and the isotope signature of quartz are representative to the parent fluid composition and its evolution during the experiments. The measured T_m range within a same FIA has thus a signification because each inclusions of the FIA have recorded a fluid composition at one time.