Fluid flow equipment for hydrothermal investigations: apparatus and preliminary results

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Introduction

During the last two decades, experimental dynamic systems have been developed to simulate geochemical mass transfer induced by fluid flow through various geological materials (Posey-Dowty *et al.*, 1986). Experiments using these fluid flow systems are conducted to investigate various environmental and geochemical problems. For several reasons, they are more attractive than batch experiments. For example, in fluid flow experiments the water/rock ratio is not affected by fluid sampling and the fluid chemistry can be monitored during the course of reaction without disturbing the temperature and pressure conditions. In addition, the control of fluid flow makes it easier to derive kinetic parameters.

The extraction of useful energy from the so called 'Hot Dry Rock' (HDR) requires circulation of fluids through fractured rocks between two or more wells. This will induce mass transfers. The Soultz-sous-Forêts (Alsace, France) project consists of testing the feasibility of developing a heat exchanger in the deep granite basement. As part of this project, we designed and built fluid flow experimental equipment.

Fluid flow equipment

All components of the fluid flow system consist of inert material (PEEK and Titanium). The reacting solution is circulated, using a HPLC pump type at flow rates up to 10 ml/min. The titanium reaction cell (I.D. 50 mm, L. 120 mm) containing the rock grains is placed within a furnace along with a reservoir where the fluid is stored after flowing out of the reaction cell until sampling. Before the experiment starts, the storage reservoir is closed by a piston maintained by N_2 gas under experimental pressure conditions. This allows to keep the fluids under stable temperature and pressure conditions during the course of the experiment. The fluid reaches experimental temperature before entering the rock containing cell. It can be cooled before sampling. A device allowing pH measurement before degassing is installed on line.

Because of the low value of the dispersion parameter (studied by NaI tracer), longitudinal dispersion is insignificant (Levenspiel, 1971). Therefore, dissolved amounts of a mineral can be easily quantified as a function of time and flow rate.

Experiment

An experiment of reacting granite with distilled water was conducted at temperature of 180°C and pressure of 14 bar during 38 days. The starting material is a crushed granite from GPK1 borehole at Soultz-sous-Forêts (France) (sample K-20, at depth 1995 m) with a grain size ranging from 1 to 2 mm. It consists of a porphyritic K-feldspar granite, rich in Na-perthite plagioclase and Fe-Mg biotite with minor components such as amphiboles, sphene, apatite, zircon and allanite.

The solid was washed with distilled water (in order to eliminate ultrafine particles) and dried. The initial specific surface as measured by the N_2-B.E.T. method is around 0.6 m^2/g. Close to 293 g rock sample was used in this experiment and the instantaneous water/rock mass ratio was kept around 0.37. The fluid flow rate was maintained constant at 0.09 ml/min during the experiment. The fluid was sampled every 24 hours. To recognize sources and sinks of different elements, it is important to examine dissolving minerals and possibly precipitating minerals. Therefore, after experiments, the solid phase was consolidated in supercooled water (–20°C) in order to remove it from the cell without disturbing the spatial repartition of material. Seven sections (N1, ..., N7) was realized between the inlet and the outlet. Each section was studied using various techniques (Microscopy, Electronic Microprobe, Chemical analysis, SEM, etc ...) to examine spatial evolution of dissolution and precipitation reactions along the flow path in the core sample.
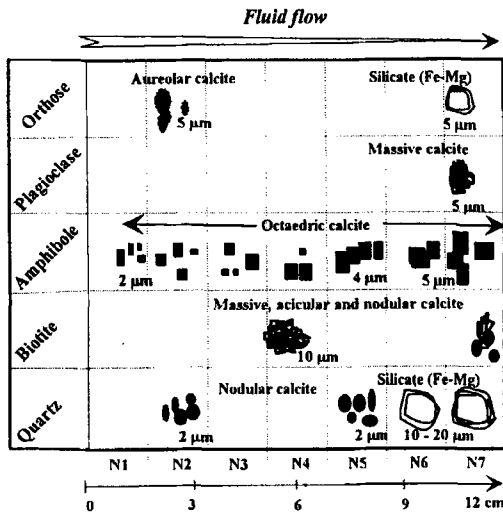
FIG. 1. Form, size and spatial repartition of precipitations on different primary minerals along the fluid flow in the core sample (12 cm).

Results

The examination of outlet fluid composition allow evidencing of dissolution reactions. The mobility order is: $Si > \Sigma CO_2 > F \sim Na > Ca > Al > K > Li > Cs > Mg > Ba > Rb > B > Sr$.

The dissolution processes are also observed on primary mineral surfaces. The feldspars seem to be the most reactive minerals. K-feldspar is very altered at N4 and N6 levels but plagioclase is altered especially at N2, N3 and N7 levels. Biotite and quartz show some dissolution figures at N4, N6 and N7.

Furthermore, figure 1 shows a description of the alteration product minerals. Calcite, the most abundant alteration product, was observed especially on hornblende grains. Five mineralogically different forms are distinguished; aureolar calcite on K-feldspar at N2 level, octaedric calcite on hornblende at all levels, acicular and nodular calcites on biotite at N7 level and on quartz at N2 and N5 levels and massive calcite precipitated on plagioclase at N7 level and on biotite at N4 level. The size of the secondary calcite minerals increases in the direction of fluid flow. Ferromagnesian silicate (saponite) was precipitated on K-feldspar at N7 level and on quartz at N6 and N7 levels. Thus, it is clear that the autigenic minerals are not uniformly distributed along the fluid flow.

Conclusion

Chemical reactions (dissolution and precipitation) affecting a sample of granite have been evidenced in a fluid flow experiment by studying both aqueous and solid phases. Examination of solid phases has shown zonalities in the distribution of dissolution and precipitation. Such experiments should be of great interest to predict chemical behavior of heat exchanger upon fluid circulation. The fluid flow equipment presented here should allow kinetics studies as discussed by Fouillac and Azaroual (this volume).

References

Levenspiel O. (1972) *Chemical reaction engineering.* John Wiley and Sons, New York, 578 pp.

Posey-Dowty J., Crerar D., Hellman R. and Chang C. D. (1986) *Amer. Mineral.* 71, 85−94.