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Micro-excavation and direct chemical analysis of frozen fluid inclusion using cryo-FIB-SEM-EDS: a novel tool for petrographic description

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We are showing the result of direct chemical analysis of a fluid inclusion using micro-excavation under cryo-temperatures (Yoshida et al., 2018). Since fluid inclusions in metamorphic rocks are small and sparsely distributed, previous approach using a scanning electron microscope (SEM) and a cold stage (e.g., Ayora and Fontarnau, 1990) has difficulties to conduct. A focused ion beam (FIB) is a novel tool for micro-processing of samples. A combination of an FIB, a SEM, an energy dispersive X-ray spectrometer (EDS), and a cold stage, can be a powerful tool to determine the chemical composition of small fluid inclusions trapped in metamorphic rocks.
We applied a new analytical method for the ultrahigh pressure (UHP) talc-garnet-chloritoid schist collected from the Makbal metamorphic Complex, Kyrgyz (Orozbaev et al., 2015). Fluid inclusions were found in pressure shadows surrounding large garnet porphyloblasts (ca. 1cm). Conventional techniques such as microthermometry and cryo- and room temperature (RT)-Raman detected NaCl and CaCl2 as solute species in the fluid inclusions. Salinity of fluid inclusions were estimated as 20.5 mass% CaCl2 and 1.7 mass% NaCl under the assumption of a NaCl-CaCl2-H_2O ternary system. One typical inclusion was further studied by cryo-FIB-SEM-EDS. Small chip of the sample was cooled to -130 degree C in a cold stage and the frozen fluid inclusion was excavated by FIB. Excavated ice has weak heterogeneity possibly derived from slow cooling rate although the scale of heterogeneity was finer than the EDS spot analysis. EDS qualitative chemical analysis revealed the existence of KCl as an additional solute species, which is hardly identified by conventional techniques. Quantitative analysis was not conducted.
Petrographic description of the fluid inclusions indicated that the studied inclusions have been trapped during the exhumation of the UHP talc-garnet-chloritoid schist. These fluid inclusions may be originated from the decompression-breakdown of lawsonite that described as polyphase mineral aggregates (PMA) by Orozbaev et al. (2015). Reports on CaCl2-bearing aqueous fluid have been known from the HP and UHP metamorphic terranes in the eastern Asia (e.g., Gao and Klemd, 2001). Since mica minerals are common in subduction-related metamorphic rocks, KCl can be easily accompanied with dehydrated fluid from such kind of rocks. Our data may question the existence of complex chemical system of the aqueous fluids reported in low-temperature type metamorphic rocks. Misidentification of solute species essentially brings errors in salinity estimates that will propagate inaccuracy in other quantitative analytical methods such as LA-ICP-MS. A combination of FIB-based sample-preparation (Miyake et al., 2014) and SEM-EDS analysis will enable to perform accurate quantitative analysis of individual fluid inclusions.