Coordinated sample preparation with cryo-FIB-SEM and X-ray CT: Applications to chemical analysis for fluid inclusions in minerals

*Yu Kodama[1], Naotaka Tomioka[2], Tatsuhiko Kawamoto[3], Junichiro Kuroda[4], Masafumi MURAYAMA[5], Motoo Ito[2]

1. Marine Works Japan Ltd., 2. Kochi Institute for Core Sample Research, JAMSTEC, 3. Department of Geoscience, Faculty of Science, Shizuoka University, 4. Atmosphere and Ocean Research Institute, the University of Tokyo, 5. Faculty of Agriculture and Marine Science, Kochi University

Hydrothermal fluids and seawater are often entrapped in minerals as fluid inclusions in crustal/mantle materials, marine halites and even in rare meteorites [1, 2, 3, 4]. The chemical compositions of fluid inclusions and mineral inclusion therein are keys to decipher their origins and evolution of the geofulids. A laser-ablation inductively-coupled-plasma mass-spectrometry (LA-ICP-MS), a Raman spectrometry and an environmental SEM (cryo-SEM with EDS) are methods for chemical analysis of fluid inclusions, entrapped tiny minerals and gasses. For example, Kawamoto and coworkers recently determined Na/K ratio of fluid inclusions in olivine crystals from mantle xenoliths from the Pinatubo volcano by LA-ICP-MS in 20-μm spatial resolution [1]. The attempt successfully provided new insight on the fate of seawater subducting into the mantle in a plate convergent region. Lowenstein et al. investigated major chemistry of fluid inclusions from marine halites by a cryo-SEM with EDS system, and found systematic and oscillating changes in Phanerozoic seawater chemistry (Na^+ vs. Cl^- and Mg^{2+} vs. Cl^-) [3].

As unique sample preparation and analytical approach to a fluid inclusion, combined instrument of focused ion beam system and scanning electron microscope with a cryo sample stage (cryo-FIB-SEM) is recently utilized (e.g., Yoshida et al., 2018 [2]). Fluid inclusions can be exposed as ices in the vacuum sample chamber of cryo-FIB-SEM, and then the frozen fluids can be directly analyzed by EDS attached to SEM. Advantages of this approach are a direct access to fluid inclusions by precise sample milling with FIB, and simultaneous analysis for major elements with a relatively high spatial resolution (~5 μm). The method has been first applied to fluid inclusions in quartz [2].

A cryo-FIB-SEM approach is, however, time-consuming: entire processing and analysis with cryo-FIB-SEM requires several to few tens of hours for a several tens micrometer-sized sample. Descriptions of exact locations and spatial distribution of each fluid inclusion in host minerals will help to minimize a processing time with FIB. We, therefore, used a laboratory-based micro-focus XCT apparatus (μXCT) at the Center for Advanced Marine Core Research, Kochi University for acquiring an accurate three-dimensional characterization of fluid inclusions in minerals with a spatial resolution of 0.5 to 2.5 μm depending on sample size. In the present study, we aim to improve the throughput of the fluid inclusion analysis using cryo-FIB-SEM and establish a reasonable analytical flow. We have obtained spatial distributions of fluids in olivine from the Pinatubo xenoliths and those in halite in Sicilian rock by μXCT. Pretreatments for the samples have been carried out by micro-sampling system, and then microfabrication and chemical analysis of frozen fluids in the samples have been carried

out by cryo-FIB-SEM.

In this talk, we will present the effectiveness of coordinated sample preparation utilizing cryo-FIB-SEM and μXCT, and a comprehensive microanalysis of fluid inclusions in addition to cryo-FIB-SEM. For instance, residues of fluid inclusions and associated minute minerals in FIB-processed samples can be further characterized in a higher spatial resolution by an analytical transmission electron microscope (TEM) and a secondary ion mass spectrometer (SIMS).

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