Development of cryogenic SIMS technique for isotope analysis of fluid inclusions

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Introduction: Fluid inclusions in halite crystals from meteorites provide direct information of extraterrestrial liquid water [1]. In-situ isotope analysis of the fluid inclusions was realized by SIMS equipped with a cold-stage [2]. However, the analytical precision is limited to ±14 ‰(1 σ) for $\delta^{18}O$ due to deep sputter crater to excavate fluid inclusions. One of the methods to improve the analytical precision would be expose fluid inclusions on the sample surface. We froze the fluid inclusions and polish under cryogenic condition in nitrogen atmosphere [3, 4]. In order to measure frozen polished fluid inclusions by SIMS, the surface should be coated by conductive thin layer such as Au. However, the Au thin layer was not enough conducted because condensation of frost particles occurred before introduce the polished sample into Au coater. The sample temperature was −100 ˚C while the dew point in the glove box (−60˚C) was higher than the sample temperature [4]. Therefore we changed the sample temperature.

Changes of sample preparation procedures: The sample preparation system for cryogenic SIMS is composed of a cryo-polisher and Au ion-coater in a glove box [3]. The temperature of polishing set to −35 ˚C. Thus frost condensation on the polished surface was prevented. After polishing, the sample was settled on the bottom of deep well of new designed cryo-coating stage. The cryo-coating stage was precooled to −196 ˚C by liquid nitrogen and the deep well was filled with cool nitrogen gas evaporated from liquid nitrogen in order to prevent frost formation on sample surface. This cooling process is necessary because sublimation temperature of ice is −50 ˚C during Au coating. The sample temperature was estimated to −196 ˚C before taking into the Au coater and warmed up −130 ˚C at taking from the Au coater after Au coating for 4 minutes.
The polished ice embedded in an epoxy disk was set to the cryo-holder and transferred to cryo-stage (−190 ˚C) of Cameca ims-1270 instrument using cryo-transfer system [3]. The liquid nitrogen was supplied by a roots pump to reduce the sample stage vibration. The measurement methods for oxygen isotopes were similar to those of [2].
We obtained mass spectra of oxygen isotopes from H_2O-ice. Intensity of ^{16}O peak was about 2.8 $\times 10^5$ cps and a tale was observed symmetrically at both side of the peak below 3 cps. The interference of tail from $^{16}OH^-$ to $^{17}O^-$ was estimated to be about 10 ‰because the intensity of $^{16}OH^-$ is comparable to ^{16}O peak. Oxygen isotope analysis using $^{16}OH^-$ and $^{17,18}OH^-$ signal would be effective for further high precision oxygen isotope analysis for H_2O ice as well as the tail correction method used in [2].
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