The FIB milling of LHDAC sample for TEM, EMPA and SEM analyses

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In order to generate ultrahigh-pressure (100 GPa) and temperature (3,000 K) conditions corresponding to the Earth's interior, we have carried out laser-heated diamond anvil cell (LHDAC) experiments. LHDAC experiments allow us to produce ultrahighpressure and -temperature conditions, the synthesized samples recovered from these experiments are in the form of thin films (the applicable area for analysis is 50 micro meter in diameter and 7 micro meter in thickness). TEM, EMPA or SEM is necessary for analyzing the samples recovered from the LHDAC experiments. Focused Ion Beam (FIB) system was applied to investigate these samples synthesized by LHDAC experiments. The TEM, EMPA and SEM analyses of cross-sectional surface of the thin film samples became possible by using FIB system. In the case of TEM analysis, a block piece (20-7-1 micro meter) of the sample was obtained by forming box-shaped grooves in the sample using a gallium ion beam in the vicinity of the area of interest for TEM first. It was removed on to a molybdenum-grid using a glass pipette under a dedicated optical microscope. The block piece adhering to the molybdenum-grid was thinned by gallium ion beam to form a TEM foil. On the other hand, in the case of EMPA and SEM analysis, a block piece (40-20-15 micro meter) of the sample was obtained by forming grooves around the block piece using a gallium ion beam. The block piece was removed from the sample and mounted on a molybdenum-grid using a glass pipette under a dedicated optical microscope. The cross-sectional surface of the block piece was cleaned by a gallium ion beam again and became EMPA and SEM sample. Larger areas could be analyzed by using the samples compared with the TEM foil. The application of FIB thus allows for the preparation of film samples recovered from LHDAC experiments expeditiously and effectively.