Focused Ion Beam (FIB) processing of samples from Laser-heated DAC experiments for TEM observations

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Ultra-fine processing of materials by Focused Ion Beam (FIB) is a key technology in recent nano-sciences, where Ga ion is accelerated and focused onto a solid surface so that precise caving of the sample in a very small (~0.010 mm) target area can be made under the Secondary Ion Microscopic (SIM) observations. In Earth science community, this technique has been used to make thin films of minerals for TEM observations (e.g. Heaney et al., 2001), but its application to those synthesized in high-pressure apparatus has been very limited (Miyajima et al., 2003). We have purchased a FIB tool (JEM-9310FIB) at GRC, Ehime Univ., for sample preparation for TEM observations, and started to use it for processing of small samples available in multianvil and laser heated diamond anvil cell (LHDAC) experiments. Here we present some preliminary results on the TEM observations for magnesite and its new high pressure phase obtained in our recent in situ X-ray diffraction measurements at SPring-8 (Isshiki et al., 2004).

The samples were natural magnesite with a composition of (Mg0.995Ca0.004Fe0.001)CO3 (plus some Pt powder as a pressure marker) sandwiched by a pair of Al2O3 disks, which were heated in a LHDAC and retrieved with surrounding Re foil gasket. The disk sample with dimensions of ~0.050-0.100 mm in diameter and 0.010 mm thickness was processed using the FIB, and a rectangular thin film of 0.010 mm X 0.005 mm X 100 nm was picked up with a micro-manipulator under the microscope. The film was transferred to a carbon-coated copper mesh and was analyzed with a 200 kV analytical transmission electron microscope (JEM-2010) at GRC.

We recently reported that magnesite transforms to a new structure (magnesite II) at pressures greater than 115 GPa, at ~2000 K, using LHDAC combined with synchrotron radiation at SPring-8. This magnesite II was found to be unquenchable to the ambient conditions, and we concluded that it transformed back to magnestie on release of pressure on the basis of in situ X-ray diffraction measurements. In the present study, we examined those obtained from the stability fields of magneiste (MS-29; 30 GPa, 2300 K) and of magnesite II (MS-15; 115 GPa, 2200 K) using TEM after the FIB processing of the samples.

We found that the sample was indeed of the MgCO3 composition without any signs of dissociation to CO2 or C-bearing assemblages and of reaction with the surrounding Al2O3 thermal insulator, but noted the presence of some amounts of Ga, presumably implanted in the sample during the FIB processing. TEM observations demonstrated that the sample from MS-29 is of the magnesite structure in any portions, with notable grain growth in the middle of the sample. On the other hand, while the peripheral parts of the sample adjacent to the thermal insulator were made of polycrystalline magneiste, the middle part of the sample from MS-15 were found to be an amorphous on the basis of electron diffraction measurements. This suggests that there was significant temperature gradient in the axial direction of the disk sample in a distance of a few micrometers, and the magneiste II formed at the relatively high temperature portion of the sample is amorphisized upon decompression, whereas magnesite was untransformed on the lower temperature side and retrieved to the ambient condition. Such a decisive conclusion is hard to be obtained by the in situ X-ray diffraction study alone, as it is difficult to identify the crystal structure of the sample in such a small spacial resolution. Thus the FIB processing of the sample should be potentially important in gaining accurate knowledge on the nature of the phase transitions in such small samples as are available in high-pressure experiments.