
Some recent developments in electron microscopy analysis of samples recovered from high pressure experiments

Hiroaki Ohfuji*¹

¹Geodynamics Research Center, Ehime University (GRC) – Japan

Abstract

Together with in-situ/ex-situ X-ray/neutron diffraction and optical spectroscopy observations, electron microscopy analysis of samples quenched from high pressure and high temperature plays an important role in the advancement and development of high pressure science. Over the last decade, various new methods and novel techniques for sample preparation have been introduced to our community along with the further technical development of electron microscopes. Here, we show practical examples of some new techniques/methods to examine the microtexture and chemical characteristics of recovered samples from multianvil and DAC experiments by SEM and TEM.

Cross-sectioning using ion beam provides quick and effective solutions for direct observation of recovered samples by electron microscopy. We recently introduced a JEOL cross-section polisher (CP) to prepare large-area sections for SEM-EDS, EPMA and EBSD analysis. CP uses an argon beam and gives smooth and mechanical damage- and contamination-free sections of up to 4 mm in width. We found that this technique is useful and powerful to prepare a whole cross-section across the sample chamber in a Re gasket of LHDAC samples. It is also convenient to analyze microtexture and chemical composition of recovered samples that have not been sintered well, but are porous like the case of fluid-saturated system.

Another technique we have recently introduced is osmium surface coating, which is found to be effective for SEM-EDS quantitative analysis, particularly for light elements such as oxygen, carbon and nitrogen. The Osmium coating prepared by chemical vapor deposition provides an extremely thin and uniform layer whose thickness can be controlled simply by coating time. Because of the high reproducibility and reliability of the coating process, users have no difficulty in evaluating the actual coating thickness, which enables strict and precise adsorption corrections (for the coating layer), even for low-energy characteristic X-rays. Our study shows that oxygen concentrations in silicate and oxide can be quantified correctly when using the osmium coating. The ability to accurately quantify oxygen may stimulate new applications such as the estimation of Fe²⁺/Fe³⁺ concentrations and water content in minerals.

*Speaker