

Light element quantification using electron microprobe and Os surface coating

OHFUJI, Hiroaki^{1*} ; YAMAMOTO, Masashi¹ ; KOJIMA, Yohei¹

¹Geodynamics Research Center, Ehime University

Electron microprobe analysis is a non-destructive method widely used for determining the chemical composition of solid materials such as not only minerals and rocks but also industrial and biological materials. Recent advances of solid-state detectors for energy dispersive spectroscopy (EDS) analysis allow us to readily collect precise quantitative data. For SEM and EDS analysis of non-conductive (insulating) materials such as minerals and rocks, surface coating of a thin conductive layer is a prerequisite for sample preparation. For this purpose, carbon and gold are most commonly used; the former with low atomic (*Z*) number is suitable for microprobe chemical analysis, while the latter is preferable for textural observation of samples with rough, uneven surfaces and/or with high porosity. Recently, osmium coating prepared by chemical vapor deposition (CVD) has been a focus of attention and found to be effective for high-resolution SEM observation of samples with uneven surfaces. In the present study, we applied the sample preparation technique using very thin osmium surface coating for chemical quantification of various mineral samples by EDS.

The SEM-EDS analysis was performed by using FE-SEM (JEOL, JSM-7000F) equipped with a silicon-drift-type EDS detector (Oxford Instruments, X-Max 20). Accelerating voltage and probe current were 15 kV and 1 nA, respectively. Osmium coating of 5 nm thick was carefully made by using a Neoc-ST osmium coater (Meiwafosis). Quantification analyses were conducted on a variety of mineral samples, silicate (including hydrous silicates), carbonate and oxide minerals.

The results showed that the quantification data obtained from samples with osmium coating are as accurate as those from samples with conventional carbon coating for principle elements such as Na, Mg, Al, Si, K, Ca and Fe. With respect to the quantification of light elements such as C, N and O, the results from osmium-coated samples are found to be closer to their stoichiometric values than those from carbon-coated samples. This is likely caused by the absorption correction of the X-rays passing through each coating layer. The thickness of the surface coating layer can be precisely controlled in the case of osmium coating prepared by the CVD technique, but not readily adjusted in carbon coating. As the result, the deviation of the estimated thickness of the coating layer from the actual thickness over/underestimates the effect of X-ray absorption correction, in which low-energy X-rays from light elements are more significantly influenced by the correction. We found that the precise quantification of oxygen as a separate element (not as oxide forms of cations) using osmium surface coating might be helpful and effective in estimation of the valence state of iron in iron-bearing minerals and water (hydrogen) content in hydrous minerals. We also confirmed that the present technique is also useful for precise quantification of carbonate minerals such as CaCO₃.