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## Geochemical and cosmochemical application of microanalysis



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The Special Collection of papers presented in this volume features the proceedings of the Korea-Japan Joint Conference on the Geochemical and Cosmochemical Applications of Microanalysis that was hosted by the Korea Basic Science Institute, and was held in Ochang, South Korea, in June 2023.

Precision of chemical and isotopic analysis in geosciences is constrained by the number of atoms and molecules available for measurements and also by the inherent properties of the analytical techniques. The interplay between these factors determines the areas of micro- and macroanalyses. Here we define microanalysis in geochemistry and cosmochemistry as any chemical, isotopic or structural analysis that could have been carried out with higher precision or accuracy if the sample size (i.e., the number of atoms) available for analysis was larger. And conversely, the analyses with precision that is primarily limited by the techniques and instrumentation, and cannot be directly improved by increasing sample size, are defined as macroanalyses. The techniques of microanalyses and their advancement and application to earth and planetary sciences are the subject of this Special Collection.

More often than not, samples of geological and extraterrestrial materials are available only in limited quantities. Specimens of extraterrestrial rocks delivered by sample return space missions, mineral inclusions in other mineral grains, individual mineral grains, fluid inclusions, and components of rare meteorites are just a few examples among the innumerable materials that could have been analyzed more precisely if they were larger. Increasing sample sizes for these materials is either impossible due to limited availability, or is prevented by the need to resolve the internal heterogeneity of the samples. At the same time, the requirements to precision of isotope analysis are becoming more demanding because of the need to resolve smaller natural isotopic variations (in isotope geochemistry) and shorter time intervals (in geochronology). Improvement of sensitivity of analytical techniques without losing precision or resolution is thus of great importance for the progress of earth and planetary sciences.

Many institutions and research groups in both Korea and Japan have long-standing traditions of developing novel microanalytical techniques for earth and planetary sciences. This collection of papers provides a glimpse of some of most recent of these developments and their applications.

Jeong et al. (2024) present the U–Th and U–Th–Pb dating of Quaternary zircons from Jeju Island, Korea, utilizing a femtosecond laser-connected multi-collector ICP-MS. They determined five  $^{238}$ U– $^{230}$ Th ages from 28.7 to 117.6 ka and two  $^{238}$ U– $^{206}$ Pb ages of 743 and 785 ka. The data in this study provide chronological evidence of trachyte magmatism occurring in Jeju Island during the transitional period between the Early and Middle Pleistocene and the Late Pleistocene. The zircon samples analyzed in this study can serve as a reference age for Quaternary geochronology research.

Choi et al. (2024) report the development of microstructural analysis of zircons using a combination of electron backscattered diffraction (EBSD) and electron microprobe (EMP) mapping, in addition to commonly used cathodoluminescence (CL) and backscattered



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electron (BSE) imaging techniques. The authors found evidence for subsolidus recrystallization of zircons within the Ulleung syenite, suggesting either coupled dissolution and reprecipitation or thermoactivated particle and defect volume diffusion due to inherent lattice strain. They conclude that the subsequent deformation observed in the zircons might be a result of increased stress within the magma system after the recrystallization.

Jeong (2024) reviews the recent findings of Asian and Saharan dust particles by mineralogical and microanalytical observations using scanning electron microscopy (SEM), transmission electron microscopy (TEM), electron diffraction, X-ray diffraction (XRD) and energydispersive X-ray spectroscopy (EDS). Dust particles are heterogeneous mixtures of clay and nonclay minerals. The author describes the importance of the mineralogy and microstructures of individual dust particles for better understanding the interactions between mineral dust and Earth environments. The constituent mineralogy of dust particles is discussed in an environmental context with a brief introduction of the geological backgrounds of the minerals in their source areas.

Hidaka (2024) presents a review of isotopic variations of Sm, Gd, Er and Yb in planetary materials caused by neutron—capture reactions, and a new, highly efficient procedure of separation of rare earth elements, including Yb, for such analyses. The isotopic shifts of <sup>149</sup>Sm–<sup>150</sup>Sm and <sup>157</sup>Gd–<sup>158</sup>Gd are established tools for studying the history of neutron irradiation in meteorites and lunar surface materials. Extending the range of measured isotopic variations to Er and Yb allows studying the balance of the fluences between thermal and epithermal neutrons. For better understanding the neutron fluence and its energy distribution, the use of Yb isotopic variation is discussed with application to two different materials: lunar regolith and rocks from the Oklo natural reactors.

Jinnouchi et al. (2024) report a development of a new compact magnetic separator for on-site screening of geological materials. The existing compact magnetic circuits allow separation of a mixture of ferro- and ferrimagnetic minerals, but its resolution was not sufficiently high to analyze various heterogeneous particles studied in geological research. The new separator design has greatly improved separation efficiency of particles due to magnetic translation increased by a factor of ~ 2.5. The authors also established the orbit simulation program in magnetic and gravitational field, which accurately predicts the trajectory due to magnetic translation. The new device is compact and requires little electric power, allowing on-site material screening in various geological research.

Bajo and Yurimoto (2024) report the development of a nanoscale analysis technique for noble gases in solids.

Noble gases are valuable tracers in geochemistry, which are used to elucidate the origin and evolution of the solar system and planets. Noble gas analyses have been previously limited to bulk and spot analyses of solids without the possibility of two- and three-dimensional imaging. Recent developments in isotope imaging using secondary neutral mass spectrometry are reviewed. The images have been fully quantified, and the spatial resolution has reached the nanoscale. With this development, the concentration distribution of He in solids has been visualized as a map for the first time.

Park and Kim (2024) determined the age of lunar zirconolites by chemical analysis using electron microprobe. The age of the zirconolites found in a granitic clast of the lunar meteorite DEW 12007 is determined to be  $4333\pm14$  Ma, which is consistent with the U–Pb age ( $4340.9\pm7.5$  Ma) of zircon grains from the same clast. The precision and accuracy are significantly improved over previously reported chemical ages of lunar zirconolites. The authors remark the applicability of electron microprobe dating for microscopic U–Th–Pb-containing minerals, especially in extraterrestrial materials.

Amelin (2024) discusses the analytical precision of Pb isotopic dating for meteorites and ancient rocks as a function of sample size and analytical performance. Considering the possible ways for minimization of sample size and the additional sources of uncertainty of isotopic ratios, the author evaluates the limits to precision. As little as 2.9 pg of radiogenic Pb with the age of 4555 Ma would be sufficient to achieve the precision of  $^{207}$ Pb/ $^{206}$ Pb ratio=0.007% (2 s) corresponding to the uncertainty of the age of 0.1 Ma in an analytical setup that is free from noises of signals, biases of isotopic ratios and losses of Pb, but larger quantities of Pb would be required to achieve similar precision with existing imperfect analytical methods.

Yi and Amelin (2024) develop a procedure for the determination of elemental abundances of U, Th and Pb and isotopic abundances of Pb in several accessory minerals by SHRIMP IIe for the purposes of understanding distribution behavior of these elements in various minerals, and interpretation of Pb isotopic ages of meteorites. The authors report the level of sensitivity below part per billion concentration and precision of ~ 20–30%, which is deemed adequate for measuring U, Th and Pb distributions in both rock-forming and accessory minerals in chondrites, achondrites and their components.

Sano et al. (2024) present a development of a new standard material for analysis of meteorite zircons, prepared by high-pressure sintering. Homogeneity of the new standard is verified with laser ablation and Nano-SIMS microanalyses. Doping with hafnium oxide and tungsten oxide produced sufficiently high concentrations of Hf and W for precise determination of relative sensitivity factors. The new synthetic standard was used for determination of  $^{182}\mathrm{Hf}-^{182}\mathrm{W}$  age of zircon from the mesosiderite Asuka 882023.

The paper by Terada et al. (2024) presents a development of non-destructive isotope analysis technique using negative muon beam, and natural galena (PbS) as a test material. In Earth and planetary science, Pb isotopic composition is usually measured by mass spectrometry a destructive technique. The authors report a development of the non-destructive isotopic measurement using an energy shift of muon-induced characteristic X-rays. The Pb isotope composition derived from characteristic X-ray spectra is consistent with mass spectrometry analyses, although cannot yet match the precision of the latter. With the further development of the high-energyresolution X-ray detectors, isotopic measurements of various natural samples (solid, liquid and gaseous) might be eventually performed non-destructively.

Analytical developments reported in this issue can be applied to a great variety of terrestrial and planetary materials, producing improvement in analytical data, and in some cases opening completely new opportunities. We are confident that these developments will make a significant contribution to the advancement and deeper comprehension of the realms of geochemistry and cosmochemistry.

Published online: 08 March 2024

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