The Chang'e-5 Lunar Samples Stimulate the Development of Microanalysis Techniques

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People dreamed of touching the moon for a long time, but it was not until the 1950-1960s that this was attempted. One of the major milestones towards this dream was the Apollo 11 mission, which was the first crewed lunar landing whose mission objective was to collect samples and return to earth. In the next seven years, five subsequent Apollo missions and three Luna missions took place. Approximately 382 kg of lunar samples were collected during these missions, which has significantly enhanced our understanding of the chemical features, physical processes, and geological evolution of the moon. There is no disputing the fact that the first extraterrestrial samples brought to earth through the Apollo 11 mission ushered in a new era in planetary science.

Forty-four years after the Apollo and Luna missions returned with the lunar samples, Chang'e-5 (CE-5), China's first lunar sample return mission, returned with 1,731 g of lunar samples from Mons Rümker in northeastern Oceanus Procellarum.^{1,2} This landing site was located at an appreciable distance away from the Apollo-Luna sampling sites in low-latitude regions of the moon. The first wave of studies revealed that the samples brought by the CE-5 mission contained a new type of differentiated lunar basaltic rock with a Pb-Pb isochron age of approximately 2.0 Ga and originated from a non-KREEP dry mantle source.³⁻⁶ KREEP is a chemical signature of many Apollo 12 and 14 lunar samples, relatively enriched in potassium (K), rare earth elements (REE),

phosphor (P), and other incompatible elements. These CE-5 lunar samples provide a unique opportunity to study the evolution of the moon and the surface processes on airless bodies.^{1,7}

Moreover, the CE-5 mission is just the beginning. China is gearing up to launch a series of new space missions in the next ten years with varied mission objectives including returning to earth with samples from the South Pole-Aitken Basin of the Moon, asteroids, and Mars.⁸ From a global perspective, the National Aeronautics and Space Administration (NASA) has also announced the ARTEMIS program, intending to land on the moon again in this decade, with the mission objective of returning with new lunar samples. Furthermore, the NASA and the European Space Agency (ESA) plan to return with the first Martian sample as early as 2031. Because of continual efforts and deepening international cooperation on sample return missions, we are entering a new golden era of planetary science.⁹ New extraterrestrial samples would significantly promote the development of advanced curation and analytical techniques as expensive missions would only return with a highly limited number of samples.^{10,11}

Confucius said, “If a craftsman wants to do good work, he must first sharpen his tools.” Because extraterrestrial samples such as the CE-5 lunar samples are precious, non-destructive analyses and microanalyses are always preferred. For this reason, we organized two special issues, *Microanalytical Techniques for Extraterrestrial Samples - Part I and Part II*, to present some advanced and powerful microscopic and spectromicroscopic techniques to study the CE-5 lunar samples and other extraterrestrial samples. Contributions to the current special issue include (1) a non-destructive study of natural color and the 3D morphology of a lunar meteorite and lunar soil simulant using a high dynamic range, color structured illumination microscope (HDR-C-SIM);¹² (2) improvement of atomic force microscopy (AFM) analytical technique for the accurate measurement of adhesion force in micro-sized lunar dust grains;¹³ (3) development of Micro-X-ray diffraction (μ XRD) methods for *in situ* identification of glass and minerals in extraterrestrial samples;¹⁴ (4) development of analytical methods for the precise measurement of major and trace elements and $\text{Fe}^{3+}/\Sigma\text{Fe}$ in geological samples using electron probe microanalysis (EPMA);^{15,16} (5) study of the valence states of iron-bearing phases in CE-5 lunar soil using advanced transmission electron microscope (TEM) techniques;¹⁷ (6) *in situ* Rb-Sr dating of lunar meteorites using laser ablation multicollector-inductively coupled plasma mass spectrometer (MC-ICP-MS);¹⁸ (7) development of analytical techniques for the determination of water content in geological samples using secondary ion mass spectrometry (SIMS) and nanoscale SIMS (NanoSIMS);¹⁹ (8) development of the analytical method to measure the water content of a zircon hydrogen isotope using SIMS and the optimization of reference standards;²⁰ (9) a dedicated review of recent progress and selected applications of synchrotron-based

scanning transmission X-ray microscopy (STXM).²¹

Below, we summarize each of the contributions to this special issue:

Optical sectioning structured illumination microscopy (OS-SIM) is a state-of-the-art three-dimensional technique that uses fringe illumination patterns to modulate the in-focus and out-of-focus information of the specimens. Because of the sharp decrease in the modulation, in-focus information can be calculated. OS-SIM has the highest imaging speed among the above-mentioned methods, and it ensures an axial resolution which is as good as that of confocal laser scanning microscopy. Another advantage of OS-SIM is its ability to obtain natural color, which allows for high-magnification color micrographs. OS-SIM has been increasingly used to study various insectology, materialogy, and geology samples. In *Reconstructing the Color 3D Tomography of Lunar Samples*, Zhao *et al.*¹² present a high dynamic range, color structured illumination microscope (HDR-C-SIM) for recording the 3D tomography of lunar soil simulants and their natural color in a time-efficient manner. The system has the advantages of high sensitivity, low thermal damage, and high-speed three-dimensional imaging capabilities, which are demonstrated by the fine details and color 3D tomography of the lunar meteorite and the lunar soil simulant. This method is expected to provide a new and powerful tool for visually presenting the geological evolution of the moon.

Atomic force microscopy (AFM) is a powerful technique for measuring the microphysical properties of lunar grains. The lunar dust adhesion force is an essential factor that harms spacecrafts and astronauts, and is the key to understanding the migration of lunar dust and revealing the cause of lunar horizon glow (LHG) phenomena. As a microphysical property of the interface, the adhesion force is difficult to measure accurately because of the surface adsorption of gas molecules. To measure the adhesion force of CE-5 lunar sample grains accurately, Jin *et al.*¹³ proposed an improved AFM method that can eliminate the effect of surface adsorption based on experimental measurements of the adhesion force between a silica ball and silica wafer under different environmental pressures and temperatures. At a temperature of 200 °C and environmental pressure of less than 2.4×10^{-4} Pa, the adhesion force changes sufficiently, indicating that the effect of surface adsorption is eliminated effectively. In such cases, the adhesion force can be measured accurately using AFM. This improved method provides an effective technique for measuring CE-5 lunar samples.

Glass and amorphous state materials are vital components of lunar regolith and contain considerable geological evolution information. However, it is often challenging to distinguish glass from crystals based on morphology and elemental composition, especially in complex extraterrestrial samples that have been

subjected to significant impact and metamorphism. Micro-X-ray diffraction (μ XRD) techniques have been suggested for identifying crystalline minerals and glassy phases in CE-5 lunar breccia samples by Ma *et al.*¹⁴ It is an *in situ* analysis method for identifying the crystallization state and phases of samples in a non-destructive and intuitive manner. The samples were identified as amorphous materials, polycrystals, single crystals, and a mixture of polycrystals and single crystals based on diffraction patterns classified into dominant dispersion halos, concentric diffraction rings, independent diffraction spots, and coexisting rings and spots. The texture and structure of glassy and crystallized materials identified in the CE-5 lunar samples reveal a process of high-energy thermal motion resulting from meteorite impact on the moon.

Electron Probe Microanalysis (EPMA) can efficiently determine the major elements of various materials from earth and other planetary samples with a high spatial resolution (1–5 μ m), low cost, and high detection limits of up to a few hundred μ g/g. Recent advances in EPMA have enabled the measurement of trace elements. In *High-precision Measurement of Trace Level Na, K, P, S, Cr, and Ni in Lunar Glass Using Electron Probe Microanalysis*, Zhang *et al.*¹⁵ proposed an optimized method to determine trace-level elements (Na, K, P, S, Cr, and Ni) in lunar glass. The authors used a 20 kV accelerating voltage, 100 nA beam current, 10 μ m beam diameter, linear background mode, large-area analytical crystals, and an aggregate counting strategy to lower the detection limits and improve the accuracy and precision of these trace elements in lunar glass. The detection limits (3σ) for Na, K, P, S, Cr, and Ni were lowered to 17–96 ppm, and the accuracies of these elements were better than $\pm 10\%$ (2σ). This method can fully exploit the advantages of the non-destructive nature, high spatial resolution, high precision, and accuracy of EPMA, and opens a new perspective into the study of geochemical processes involving lunar glass.

Another advantage of the EPMA technique is the $\text{Fe}^{3+}/\Sigma\text{Fe}$ ratio. However, for a long time, there was no optimized method for simultaneously determining the major and trace elements and the $\text{Fe}^{3+}/\Sigma\text{Fe}$ ratio within a single run. In *Simultaneous In-Situ Determination of Major, Trace Elements and $\text{Fe}^{3+}/\Sigma\text{Fe}$ in Spinel Using EPMA*, Jia *et al.*¹⁶ first developed a high-precision EPMA method that can simultaneously determine major and trace elements together with $\text{Fe}^{3+}/\Sigma\text{Fe}$ ratio in spinel, an important mineral sensitive to petrologic and geochemical processes and the redox state. The authors first set up a set of spinel standards for major and trace elements and $\text{Fe}^{3+}/\Sigma\text{Fe}$ analyses. Then, they constructed an optimized EPMA method under double beam conditions (200 nA for trace elements and 60 nA for major elements) with an acceleration voltage of 25 kV and used a secondary standard calibration method to determine the $\text{Fe}^{3+}/\Sigma\text{Fe}$ ratio of spinel. The detection limits for trace elements were 16–55 μ g/g (3σ), and the estimated accuracies for the major and trace

elements were within $\pm 2\%$ and $\pm 6\%$ (1σ), respectively. The accuracy of the $\text{Fe}^{3+}/\Sigma\text{Fe}$ ratio in spinel was within ± 0.04 (2σ). This method provides robust and precise data on spinel for small, precious, and rare terrestrial or extraterrestrial samples (e.g., spinel in lunar and martian samples), which can be used to understand the formation and evolution of rocky planets.

Nanophase iron (np- Fe^0), a major product of space weathering, is important for understanding the modification of iron during the formation and evolution of lunar soils and notably alters the reflectance, absorption intensity, and continuum slope of the UV-VIS-NIR spectra of lunar soils. In *In situ Investigation of the Valence States of Iron-bearing Phases in Chang'E-5 Lunar Soil using FIB, AES, and TEM-EELS Techniques*, Mo *et al.*¹⁷ conducted *in situ* nanoscale valence analysis of iron-bearing phases in Chang'e-5 lunar soil by combining focused ion beam (FIB) microscopy, Auger electron spectroscopy (AES), and transmission electron microscopy-electron energy loss spectroscopy (TEM-EELS) techniques. The results reveal both metallic and ferric iron in the amorphous matrix of Chang'e-5 lunar soil, which provides new insights into space weathering processes (e.g., disproportionation reaction of Fe^{2+}) at the landing site.

Geochronological investigation is fundamental to revealing the stories of extraterrestrial samples, commonly achieved by U-Pb dating. Tiny sizes or the lack of relevant accessory minerals (e.g., zircon) usually impede the *in situ* U-Pb dating of extraterrestrial samples. In *In situ Rb-Sr Dating of Lunar Meteorites Using Laser Ablation MC-ICP-MS*, Zhang *et al.*¹⁸ developed an analytical procedure on LA-MC-ICP-MS and a new data reduction strategy for *in situ* Rb-Sr dating on rock-forming phases (e.g., plagioclase, pyroxene and glass). The robustness of the method was demonstrated by the isochron ages obtained for two lunar meteorites, which are in good agreement with previously reported U-Pb dating results. The method presented should have broad applicability for the Rb-Sr isochron dating of extraterrestrial samples.

Geochemical investigation can provide important clues to the structures, nature, and evolution of earth and moon. High spatial resolution analytical methods with a low background are crucial for quantifying the water content in nominally anhydrous minerals such as olivine. For secondary ion mass spectrometry (SIMS) studies, primary beams with high currents are utilized to achieve a low background. However, a high current also results in a low spatial resolution. Even though NanoSIMS is designed to measure high spatial resolution, the spatial resolution is maintained at values greater than 10 μ m for water content analysis in order to maintain a low water background of less than 10 ppm. The work presented by Li *et al.*¹⁹ optimized the analytical settings for water content measurement in olivine using NanoSIMS and achieved a high spatial resolution of $\sim 6 \mu$ m (i.e., a factor of ~ 2 better than the

previous studies) while maintaining a low background of 6 ± 2 ppm. This improvement represents the highest spatial resolution for water content analysis of nominally anhydrous minerals. This analytical method can be applied to olivine and possibly other minerals with complex zoning in lunar samples brought by China's Chang'e-5 mission.

Water is crucial not only for life but also for promoting the melting process and consequently, other geological activities on planetary bodies. The estimation of water content with constraints from hydrogen isotopes is an ongoing hot topic in studies on extraterrestrial samples. In *SIMS Zircon Hydrogen Isotope and H₂O Content Analyses and Reference Material Development*, Cui *et al.*²⁰ first reported a combined procedure on SIMS to simultaneously measure hydrogen isotope composition and the water content of zircon grains. By applying this procedure, two zircon standards (D15395-3 and D15395-4) out of the three examined were demonstrated to be homogeneous at the micrometer sampling level. Combined with data calibrated using traditional continuous-flow methods, these two standards are recommended as the first reference materials for zircon hydrogen isotope analyses using SIMS; these results have the potential to encourage further studies into the water content and hydrogen isotope analyses of zircons from terrestrial and extraterrestrial samples.

Synchrotron-based STXM is a state-of-the-art spectromicroscopic technique that efficiently integrates X-ray microscopy and X-ray absorption spectroscopy (XAS) to provide quantitative, chemically specific imaging of elements, functional groups, bonding, and oxidation states in 2D and 3D modes at a high spatial resolution (sub-10 to 30 nm), high energy resolution, and low radiation doses. It has been increasingly used to study various materials and samples in life, earth, planetary, and environmental sciences. In the past decade, efforts have been taken toward developing the next generation of STXM and the beamlines to ensure that STXM technology remains innovative and at the cutting edge to meet the growing demands of material and device characterization. In *Scanning Transmission X-ray Microscopy at the Canadian Light Source: Progress and Selected Applications in Geosciences*, Wang and Li²¹ reviewed the STXM principle, the instrumentation of conventional STXM, and the latest STXM-ptychography at the Canadian Light Source, and subsequently provided two representative applications of STXM on geoscience-related samples including magnetotactic bacteria, soil microaggregates, and related systems to fully illustrate the strong capabilities and suitability of STXM to elucidate complex systems, processes, and associations in the natural sciences. Finally, the authors prospected the potential applications of STXM-related techniques in characterizing precious extraterrestrial samples (*e.g.*, lunar samples from China's Chang'e-5 mission).

It is notable that each contribution to these two special issues maintained a detailed focus on a particular technology or a specific field. Unlocking as much information as possible from each targeted sample is critical and essential because these extraterrestrial samples are precious and limited in quantity. Therefore, we call for a correlative characterization by linking existing laboratory-based instruments to establish rich multimodal and multiscale correlated datasets for mapping the local structure, mineralogy, chemistry, crystallography, and isotope geochemistry, as well as the local mechanical and magnetic performance across length scales.¹¹

AUTHOR INFORMATION

Jin-Hua Li is a full professor of Biogeomagnetism and Geobiology at the Institute of Geology and Geophysics, Chinese Academy of Sciences (IGG-CAS). He received his B.S. degree in Biology from Northwest University (NWU, Xi'an city) in 2001, M.S. degree in Microbiology from Shandong University (SDU, Jinan city) in 2006, and completed Ph.D. in Solid Earth Geophysics from the IGG-CAS in 2010. He

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Notes

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