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Experiment Brief

Continuum with DualEELS

Title

Using EELS to reveal ferric iron content from a Chang'e-5 lunar surface sample.

Gatan instrument used

The Continuum S is an electron energy-loss spectroscopy (EELS) system that combines high-speed XCR™ sensor stack technology and advanced acquisition hardware to acquire shift-corrected, dual-energy range EELS (DualEELS™) data at ~8,000 spectra per second with near-zero dead time. Utilizing the advanced features of the DigitalMicrograph® software, 3D chemical state maps can be quickly and reliably generated.

Background

Using the presence of iron in lunar regolith, researchers can record and reveal the oxygen potential of our solar system. Previous research on Apollo samples showed that the lunar surface and interior are highly reducing and mainly contained ferrous (Fe^{2+}) or metallic iron (Fe^0), with only a tiny amount (<1 wt%) of ferric iron (Fe^{3+}). However, a much higher Fe^{3+} concentration in lunar glass beads has been recently revealed. The mechanisms for the creation and stability of Fe^{3+} in the highly reductive environment of the moon's surface are still under debate. Using EELS chemical state mapping and tomography, the authors propose an explanation.

Materials and Methods

Chemical oxidation states of iron in Chang'e-5 agglutinate glass (Sample ID: CE5C0400YJFM00408) will show differences under energy-loss near edge structure (ELNES) analysis of the Fe $L_{2,3}$ edge (Figure 1e). However, the identification and automated localization of these differences are only possible if the energy zero-loss peak (ZLP) position is accurately known for all points in the acquired data (Figure 1d). The authors used the DualEELS and ZLP-lock features to simultaneously collect spatially resolved low- and high-loss EELS information from lunar samples as a function of specimen tilt. Using multiple linear least-square (MLLS) based data fitting of these tilt-series ELNES data sets, the quantitative distribution of iron oxidation states was determined (Figure 1b and c for Fe^0/Fe^{2+} and Fe^{3+}, respectively). This was done for the entire tilt series, and chemical state tomograms were created. These findings and further analysis lead to a conclusion that the high Fe^{3+} concentration in glass beads originated from charge disproportionation during micrometeoroid impacts on the airless lunar surface.

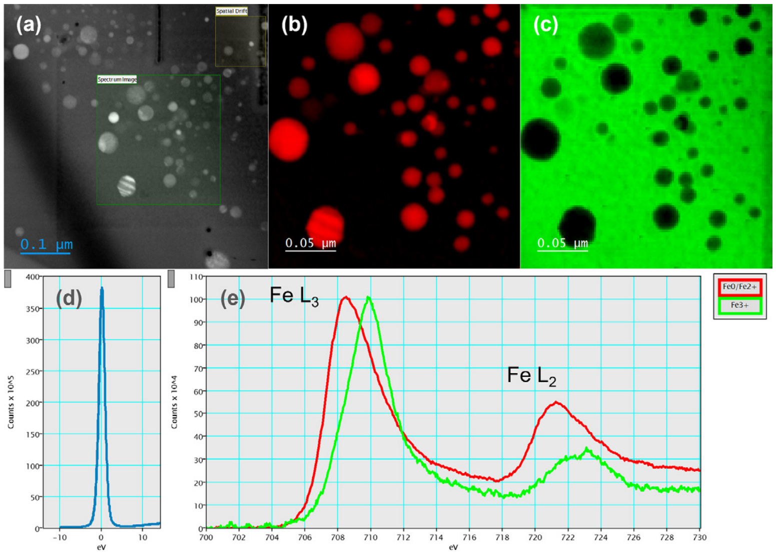

Figure 1. (a) An annular dark field (ADF) image for the region of interest and drift correction; Fe chemical oxidation states mapping using the MLLS function for (b) Fe^0/Fe^{2+} and (c) Fe^{3+}; (d) zero loss peak after using ZLP-lock, and (e) detailed Fe L ELNES shows different valence features.

Summary

A study of ferric iron content origin in a moon surface sample was possible with the help of DualEELS. This technique can be used to separate and analyze iron content based on its chemical oxidation states.

Credit(s)

A special thanks to Dr. Haiyang Xian from Guangzhou Institute of Geochemistry, Chinese Academy of Sciences.

[1] Xian, H., Zhu, J., Yang, Y. et al. Ubiquitous and progressively increasing ferric iron content on the lunar surfaces revealed by the Chang'e-5 sample. Nat Astron (2023). https://doi.org/10.1038/s41550-022-01855-0.

Gatan, Inc. is the world's leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.