2D and 3D EELS Analyses on the Low-Energy Core-Loss Edges in Beam-sensitive Ryugu Asteroid Samples

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Background
The Hayabusa2 space mission from the Japan Aerospace Exploration Agency returned materials from the Ryugu C-type asteroid to Earth in 2020. Due to its lack of atmosphere and low mass, Ryugu is less altered than other space bodies in the Solar System, preserving information about its primordial stages. One of its most interesting features is its phyllosilicate fibrous matrix, which can feature absorbed water and organic matter, as evidenced by remote infrared measurements[1] and laboratory analyses[2].
This matrix contains two families of phyllosilicates with different, albeit similar, chemical compositions and crystallographic structures: saponites (with a higher Si stoichiometry) and serpentines (with the presence of Al), intimately mixed at the nanoscale in a fibrous matrix and surrounded by various other systems such as sulfurs, and organic matter.
Until recently, the high sensitivity of the material to the electron beam[3] made it very difficult to perform electron microscopy analysis at the resolution required to address some intriguing questions, such as the presence of organic matter in the interlayers and the precise morphology of the intermixing of the different phyllosilicate families. Fortunately, the advent of direct electron detection, along with other instrumental and data analysis advances, has begun to expand the scope of analyses that can be performed in such beam-sensitive systems. Exciting recent developments include vibrational EELS[4] and 4D-STEM [5] analyses on Ryugu samples.

Here, we bring together several state-of-the-art techniques to achieve EELS analysis of FIB lamellas at unprecedented spatial resolution and EELS analytical tomography of nanopillars of these beam-sensitive samples. In particular, we utilize a hybrid-pixel direct electron detector for EELS acquisition, which offers almost perfect DQE, high-speed acquisition, and a wide dynamic range. This enables the development of a highly sensitive EELS analysis approach for all elements of interest in the sample. In particular, we acquire EELS low-loss spectrum images (SIs), instead of the usual core-loss and corresponding low-loss SIs. Since most of the elements present in these samples (Fe, Al, Mg, Si and S) showcase a major EELS edge in the 25- 200 eV spectral range, we can perform EELS quantification with a single, fast SI acquisition. This approach offers the potential of a higher signal-to-noise ratio in comparison to the more conventional ionization edges at higher energies, at the expense of a more convoluted spectral analysis.

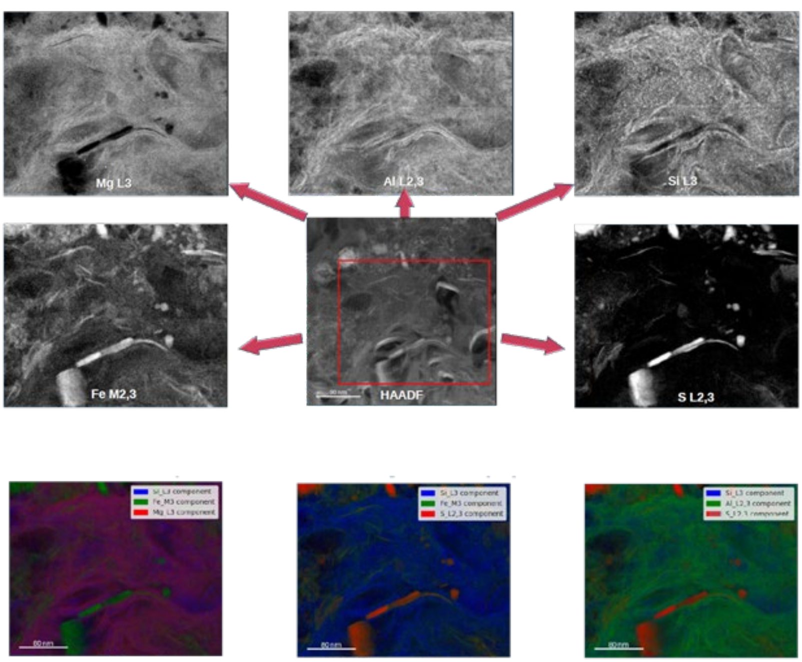
Methods
FIB lamellas from the Ryugu asteroid were fabricated from Ryugu grains and, subsequently, nanopillars for tomography were created also using FIB. EELS spectrum images were acquired on an FEI TITAN Themis 300 S/TEM, in muti-frame mode (developed in-house), with a dwell time varying between 0.5 and 2 ms and a pixel size ranging from 1 to 2nm. For the tomographic tilt series, EELS spectrum images were acquired at 9 angles ranging from -70 to 70 degrees.

The resulting spectrum images were then analyzed using SVD decomposition of the three- and four-dimensional datasets, blind source separation and curve-fitting EELS quantification.

Results
Figure 1 illustrates the successful EELS elemental mapping of a FIB lamella using curve fitting with enough spatial resolution that allows us to discern the different types of fibers in the phyllosilicate matrix and the characterization of Fe-rich minerals at the nanoscale. This allows to separate the two main phases in the phyllosilicate matrix (saponite and serpentine) by means of EELS analytical tomography.

Conclusion
We present a new method for the EELS analysis of astromineral samples at the nanoscale that, thanks to its dose efficiency, enables unprecedented spatial resolution and EELS analytical tomography on beam-sensitive samples. The method can be extended to other samples presenting EELS ionization edges at low energies. Results show that this technique can provide at least as much information as other microscopy-based techniques like EDX, but with a higher spatial resolution and a fraction of the dose.
Our findings not only provide deeper insights into the structure and chemistry of samples from Ryugu but also show the potential of this methodology for broader application to beam-sensitive astrominerals and geomaterials, such as biominerals.

Graphic:



Keywords:

Ryugu, EELS, Tomography,

Reference:

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