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Quantitative X-ray Spectrum Imaging of Extraterrestrial Materials with a Field-Emission STEM

Lindsay P. Keller

Robert M. Walker Laboratory for Space Science, Mail Code KR, NASA Johnson Space Center, Houston, TX 77058

The value of X-ray mapping is widely recognized because element distributions often provide a unique visual perspective on the genesis and evolution of extraterrestrial materials. This capability is available in the field-emission scanning and transmission electron microscope (FE-STEM) with nanometer-scale spatial resolution. Spatial resolution is critically important because of the sub-μm to nm-sized grains encountered in the analysis of cometary dust particles (the most appropriate analog for STARDUST mission samples). Conventional X-ray mapping provides mainly *qualitative* data on the spatial distribution of specific elements selected prior to the start of the mapping analysis. Ideally, one would like to obtain a *quantitative* elemental map of the sample, where each pixel of an image corresponds to a full EDX spectrum (spectrum imaging) with sufficient counts to be quantified using standard thin-film analysis techniques.

We are using a JEOL 2500SE STEM equipped with a thin window EDX detector (50 mm^2) to obtain spectrum images of ultramicrotome thin sections (50-70 nm thick) of cometary dust particles (Figure 1). The large solid angle of the microscope combined with a large active area detector results in spectrum images consisting of a high count-rate EDX spectrum in each pixel (Figure 2), enabling the determination of quantitative element abundances in addition to displaying the spatial distribution of major and minor elements. Spectrum images are acquired by rastoring a 2-D array of points with a 2 or 4 nm incident probe whose dwell time is minimized to avoid beam damage and element diffusion during mapping. Successive image layers of each sample are acquired and combined in order to achieve ~5% counting statistics for major elements). The ability to sum spectra within a defined grain allows the detection and quantification of minor and trace elements (Figure 2).

The spectrum images are used to determine 1) quantitative abundances, elemental composition, and size of individual phases (i.e. the abundance of enstatite in a thin section as well as its compositional range and the apparent grain size), 2) quantitative bulk elemental compositions of cometary dust thin sections, 3) the qualitative minor element associations among phases, and 4) the petrographic context for subsequent isotopic and spectroscopic measurements. We are also combining spectrum imaging with GIF images in order to include elements such as N that are poorly detected by EDX.

Our sample preparation methods (primarily ultramicrotomy) allow for multiple types of measurements to be made on the same sample. Spectrum images obtained in the FE-STEM are used in conjunction with NanoSIMS isotopic measurements (see Messenger, this volume) and synchrotron X-ray spectroscopic analyses of the same thin sections to determine the nature and abundance of presolar materials in cometary dust particles, and similarly, the nature and origin of the constituent grains in STARDUST mission samples.

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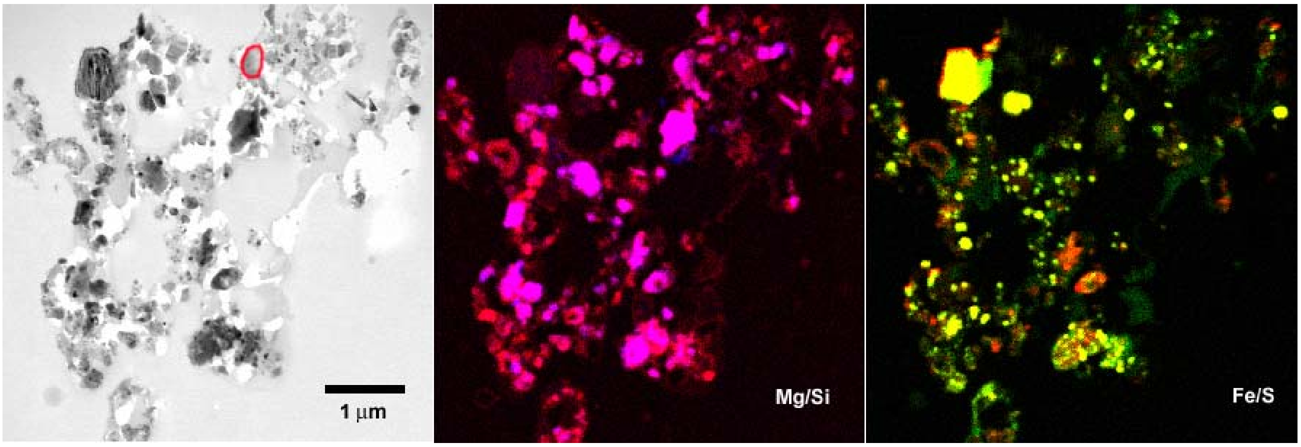

Figure 1. Brightfield STEM image from a microtome thin section of cometary dust particle L2011B10 (512x512 array), along with Mg/Si and Fe/S correlation images. Magenta-colored grains in the Mg/Si image are enstatite ($MgSiO_3$), while the yellow grains in the Fe/S image are pyrrhotite ($Fe_{1-x}S$) grains.

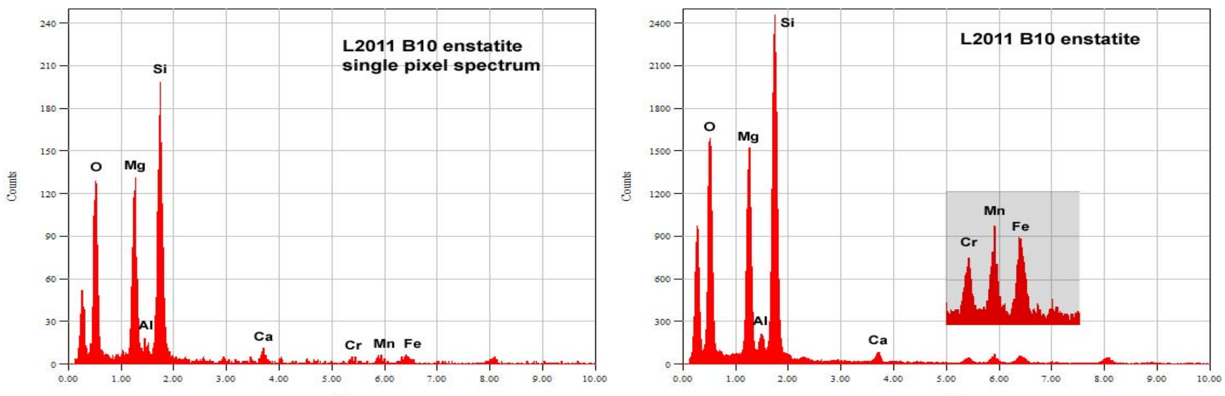

Figure 2. Extracted EDX spectra from the spectrum image in Figure 1. Single pixel spectrum (left) obtained with a 4 nm incident probe (250 counts full-scale). Spectrum from the entire grain (right) is obtained by summing all pixels that define the grain allowing for the quantification of trace elements Cr, Mn, and Fe (Kβ peaks not indicated), 2500 counts full-scale (grain is circled in red in Figure 1).

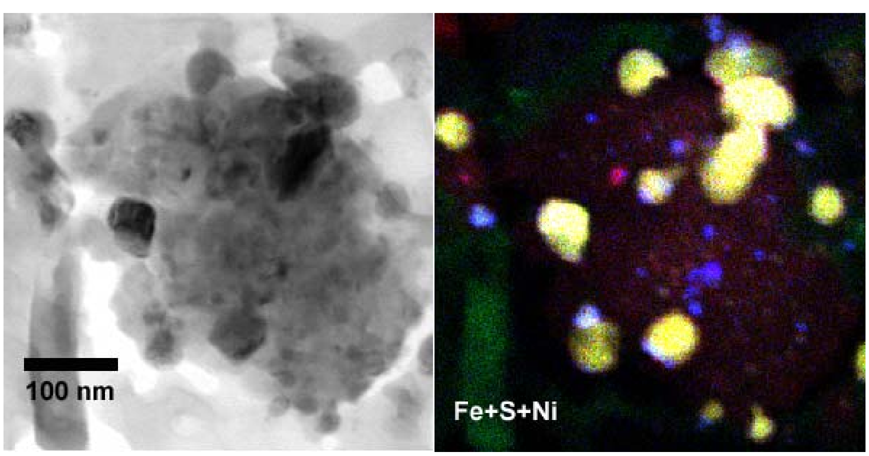

Figure 3. Brightfield STEM image (left) and Fe+S+Ni correlation image (right) of a glass grain with nm-sized inclusions. Nanophase (~10 nm) Ni-rich metal grains (blue) are dispersed throughout the glassy grain along with 10 nm Fe-. Larger Fe-sulfide grains are also apparent (yellow).

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