Interdisciplinary X-Ray Microanalysis: From Planets and Comets to Artifacts and Fine Art

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A cryogenic microcalorimeter x-ray spectrometer is coupled to an FEI XL30 environmental scanning electron microscope (ESEM). The microcalorimeter is designed to obtain high resolution broadband x-ray spectra in the energy range of 150 eV to 10 keV. An x-ray optic enhances the solid angle for x-ray energies between 150 eV and 2300 eV. This enables precise microanalysis with relatively low energy (< 5 keV) electrons. The detected x-ray photons are time-tagged and synchronized with the electron beam scan to generate high resolution x-ray maps.

The combined capability of the microcalorimeter and ESEM provides an important tool for precise microchemical analysis of complex/fragile materials as diverse as extraterrestrial specimens (e.g. cometary particles, Martian meteorites, interstellar and lunar dust returned to Earth by NASA space missions), archeological artifacts (e.g. painted organic relics found at Mayan burial sites) and fine art objects (e.g. coatings used to preserve sculptures such as those by Auguste Rodin). In addition to elemental identification and abundance determined from characteristic x-ray emission lines, we are able to differentiate the oxidation state of chemically bound elements by studying the peak shifts and variation in the relative intensities of the normal and satellite lines. Spectra and maps from a variety of these samples (see Figure 1) will be discussed.

The cometary particles and interstellar dust retrieved by NASA's STARDUST spacecraft present a challenge for microanalysis because they are embedded in silica aerogel. We are evaluating electron beam-induced etching (EBIE) as a method for effectively removing the aerogel in situ without damaging the cometary samples. This technique eliminates handling the particles and minimizes contamination from the terrestrial environment. With the aerogel overburden removed by EBIE, major and minor elemental abundances in particles can be characterized by the microcalorimeter. In preparation for analyzing STARDUST samples, NKT-1G basaltic glass was experimentally shot into aerogel at 6 km/sec, thus encapsulating them. Spectra obtained immediately before and after etching this sample will be presented.



Figure 1. Spectroscopic images of a small section of the Nantan meteorite. The top left spectrum is from the entire iron-rich region shown in the top middle image. The top right spectrum is produced from the nickel-rich area within the two dotted yellow ovals in the image labeled nickel, and the spectrum at the bottom right is produced from the nickel-phosphorus rich area within the central dotted white circle in the image labeled nickel.