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X–Ray Imaging Spectroscopy Using an NTD Ge-Based Microcalorimeter: An Interdisciplinary Spin-Off from Astrophysics Research

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We have coupled our cryogenic microcalorimeter to a standard thermionic electron source ESEM to perform proof-of-concept x-ray spectroscopy and imaging experiments. Although originally developed for measuring x-ray spectra from astrophysical sources[1,2,3] the microcalorimeter's unprecedented power to simultaneously resolve x-ray emission lines over a broad energy band (0.2 keV - 10 keV) has provided tantalizing hints of unusually rich elemental information at micron dimensions. The spectral resolution (2.5 - 6 eV) and consequent signal-to-background ratio provides better element-to-element discrimination than present generation EDS systems.

In a microcalorimeter (operating at ~60 mK), x-ray photons are absorbed by a metal foil and converted into heat. The temperature of the foil will increase because the heat capacity of the super-cold foil is ~10^{-14} J/K. This temperature rise (~10 mK for a 6 keV x-ray) is proportional to the x-ray energy and is transformed by a thermistor into an electronic signal. The thermistor is fabricated by neutron transmutation doping (NTD) of germanium (Ge). The dopant concentration establishes a well-known relationship between resistivity and temperature [4]. Our instrument can incorporate a microcalorimeter array of 16 pixels, configured as a 4 x 4 or 2 x 8. Each pixel consists of an NTD Ge thermistor attached to a superconducting tin (Sn) absorbing foil. The Sn is 0.3 mm x 0.3 mm x 7 μm thick.

An x-ray optic is integrated into the beam line that connects the microcalorimeter to the ESEM. The optic is a symmetric, point-to-point focusing cylindrical spiral lens that is 1 inch in diameter. It reflects x-rays at grazing angles and was developed and patented by us to decouple the ESEM from the microcalorimeter to minimize vibrations in anticipation of replacing the liquid cryogens with a mechanical cryocooler [5]. Not only does the optic increase the solid angle of collection compared to using the detectors alone, it also behaves as a low energy bandpass filter. The focal length is variable; as it is decreased, more lower energy x-rays are reflected because the grazing angles increase. Therefore, the optic has a *cutoff* energy that is dependent on its focal length. When chosen judiciously, unwanted high energy bremsstrahlung x-rays produced by the ESEM electron beam are prevented from reaching the microcalorimeter detector (see Figure 1).

X-ray imaging in conjunction with an ESEM has historically been accomplished by scanning the electron beam slowly enough to acquire enough x-rays to produce a spectrum. We have developed a microcalorimeter spectroscopy system that synchronizes photon detection with rapid ESEM scanning. As the electron beam scans across the sample, each x-ray event is assigned the position of the electron beam at the time that it is detected. The event is also time-tagged so we can correct for the fixed time delay between the electron-induced x-ray event and the electronic registration of the photon. This approach also enables rapid, simultaneous acquisition of secondary electron images which can be analyzed in real time and used to modulate the scan signal to offset sample and beam drift occurring during x-ray mapping.

High resolution spectra from representative bio- and non-biomaterials will be shown to demonstrate how the microcalorimeter can resolve neighboring x-ray lines of low-Z and high-Z elements in inorganic sub-micron structures and cellular tissue that are not individually distinguishable by solid-state EDS detectors. In addition, we present spectroscopic x-ray images that were obtained in conjunction with the ESEM imaging process.

References

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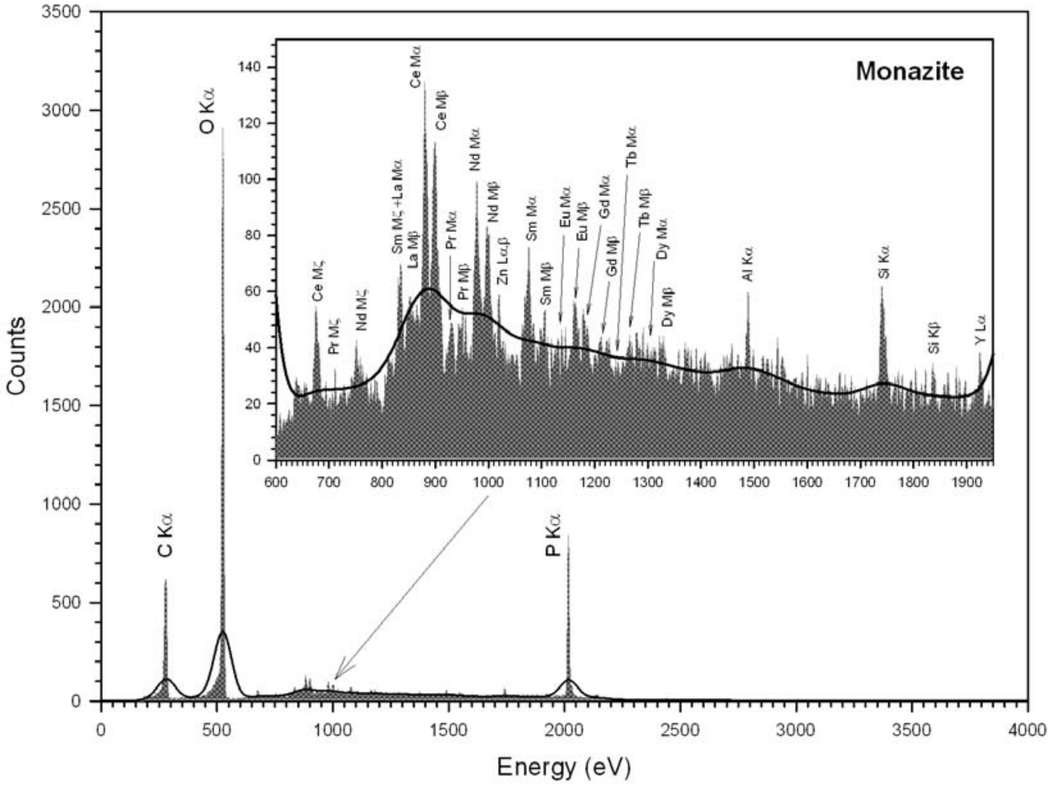

Figure 1. The x-ray spectrum of monazite, a rare earth phosphate mineral, obtained with the microcalorimeter, the x-ray optic and an ESEM. Extremely fine spectral detail in the high resolution microcalorimeter data is apparent, particularly regarding closely spaced rare earth element M x-ray lines, shown in dark gray. The smooth black curve is the spectrum that would be obtained with a traditional energy dispersive (EDS) x-ray detector.