Study of Carbon Bonding with XES using a TES Microcalorimeter Detector

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Transition-edge sensor (TES) microcalorimeter detectors are capable of high-resolution X-ray emission spectroscopy (XES) which rivals XANES spectroscopic probes found only at synchrotrons. Commercial microcalorimeters offer spectral resolution around 5-7 eV which rivals that of wavelength dispersive XRF instruments yet provide full spectra of the material of interest not merely a single element, thereby surpassing WDXRF capabilities.

The bonding of carbon is predicated on the sp^2/sp^3 orbitals. The position of the carbon Kα peak as well as the peak shape provides information on bonding. We report on the measurement of the carbon Kα peak around 282 eV using a high resolution microcalorimeter detector mounted on a scanning electron microscope. Amorphous carbon, nanodiamond, and graphite were measured since all three have different carbon forms and sp^2/sp^3 bonding ratios. A soot specimen from a contained detonation of explosive material was also measured to determine the carbon species formed. The powder samples were pressed into indium foil that was mounted onto a stub.

The carbon Kα peak position, based on a Gaussian fit, was slightly downshifted from the expected 282 eV for each material. The peaks had differences in position and shape indicating differences in the sp^2/sp^3 ratio for each material. Relative to amorphous carbon, nanodiamond showed a shift to higher energy and graphite showed a shift to lower energy. The soot appeared most similar to amorphous carbon with a small shift to higher energy indicating possible nanodiamond character. Preliminary theoretical simulations of the XES of these materials indicate similar trends in both the shape and position of the peaks and support the experimental measurements. X-ray photoelectron spectroscopy and Raman spectroscopy provide supporting data on sp^2/sp^3 hybridizations and on sp^2 allotropes for the materials of interest.