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Organic chemistry within submicron regions of Earth and planetary materials using Scanning Transmission X-ray Microscopy

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Scanning Transmission X-ray Microscopy (STXM) is a powerful analytical tool for detecting and quantifying organic functional groups within submicron regions of polymer samples at high spatial resolution less than 30 nm. In a STXM, micron-sized samples are raster-scanned relative to a focused X-ray obtained by a zone plate, and the transmitted photon flux through the sample is detected to acquire an X-ray transmission image. STXM is located at beam line 5.3.2.2 (Polymer STXM), Advanced Light Source, Lawrence Berkeley National Laboratory (Kilcoyne et al. 2003). The beam line employs a bending magnet providing a photon range spanning ~250 to 700 eV (10^7 photons/s) that includes carbon-, nitrogen-, and oxygen- X-ray Absorption Near Edge Structure (XANES) regions. Samples are thin-sectioned (~100 nm of thickness) so that a soft x-ray is well transmitted to the sample. The acquired C-, N-, and O-XANES spectra and STXM images provide a quantitative assessment of the types of the organic functionality present (Cody et al. 2008).

The advanced performance of STXM is that positioning of zone plate with continuously changing energy and scanning of sample are controlled by laser interferometer (Kilcoyne et al. 2003). This eliminates energy-to-energy image position errors from vibrational or other environmental noise and stabilizes microscope. The diffraction limited, high spatial resolution less is achieved by a Fresznel zone plate. Ordering selection aperture (OSA), which is placed between zone plate and sample, allows only the first diffraction order to pass, in order to increase the signal-to-noise ratio. The transmitted photons are detected by photomultiplier systems. A X-ray absorption spectrum is obtained through converting the transmitted X-ray to optical density (OD) as expressed by Lambert Beers' Law; $OD = -\ln(I/I_0)$, where I is the X-ray intensity transmitted from the sample and I_0 is that recorded without the sample.

These days, micro-XANES using STXM has been increasingly applied to geo- and cosmochemistry, as well as polymer material science, environmental chemistry and biology. As one of the well-known achievements, a STXM has been applied in order to obtain the organic functional group distributions on organic solids from comet 81P/Wild 2 dust particles collected by NASA's Stardust comet sample return mission (Sandford et al. 2006; Cody et al. 2008). In biogeochemistry researches using STXM, C and Fe STXM mappings of particulate organic carbon in hydrothermal plume at the mid-ocean ridge (Toner et al. 2009) and structure analyses of young poorly-crystalline graphite in the older than 3.8-Gyr-old banded iron formation (Papineau et al. 2011) have been reported. Furthermore, combination use of focused ion beam (FIB) extraction, STXM, and isotope microscope has enabled the comprehensive investigation of molecular, isotopic, and structural compositions within a very small region of particular interest from sample (Yabuta et al. 2012, JPGU abstract). Also, 3-D chemical imaging using angle-scan nanotomography in a STXM has been developed (Hitchcock et al. 2008). Thus, the requirement for STXM, the highly sensitive technique without preprocessing of samples, e.g., extraction, is becoming greater in geo- and cosmochemistry, regarding the advantages for understanding the chemical heterogeneity and diversity of organic/carbonaceous compounds in nature.

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