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A Novel Large Area Imaging NEXAFS Spectrometer for Combinatorial Chemical and Structural Analysis

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We show simultaneous molecular chemistry and bond orientation information over a large area (~100mm2) obtained with high transmission using a novel parallel process full field secondary electron spectrometer. This unique spectrometer, the LARIAT MKI (Large Area Rapid Imaging Analytical Tool) incorporates: a full field soft x-ray source (NSLS beamline U7A); electrostatic and magnetostatic electron optical elements to discriminate energy and depth of the secondary electron distribution and; a highly parallel electron detector. The rapid parallel process produces a series of two dimensional images as the incident soft X-ray energy is scanned above a K or L absorption edge. Synthesis of the image stack produces spatially resolved NEXAF spectra containing information about the chemistry including bond concentration and orientation of the surface-bound molecules with better than 100-micron lateral resolution and sub-monolayer molecular sensitivity. Rapid image-based full field capture of NEXAFS spectra over large areas permits new combinatorial approaches to data acquisition and analysis. The power of the combinatorial imaging NEXAFS method is described through a variety of examples including: i) simultaneously probing concentration and molecular orientation of single-strand DNA micro array sensors; ii) fluorinated molecular gradients; iii) and organic electronic combinatorial device arrays. Other possible applications described include the surface orientation and chemistry of continuously graded polymer films and graded or patterned self-assembled monolayers. We also envision combinatorial imaging NEXAFS as an in-situ probe for catalyst discovery using micro arrays to directly image catalytic chemical activity of thousands of catalysts simultaneously under reaction conditions. We will also briefly describe improvements of the second generation system (LARIAT MKII), currently in the design phase. The MKII will be installed on new Brookhaven National Laboratory synchrotron source, NSLS II to be completed in 2013. The key hardware differences in the LARIAT MKII configuration as compared to the MK I model is the use of superconducting magnet components and additional electrostatic elements in the electron optics assembly. The use of superconducting magnets is necessary to attain the significantly higher magnetic fields required to achieve ~600X increase in areal resolution. This overall resolution improvement derives from a factor of four increase in imaging area while simultaneously improving the lateral resolution by a factor of up to twenty to approximately 5um. The second generation spectrometer will also be able to selectively image electrons according to the emission angle to provide nanoscale depth resolution as well as thickness and uniformity data across a 400mm2 field of view.

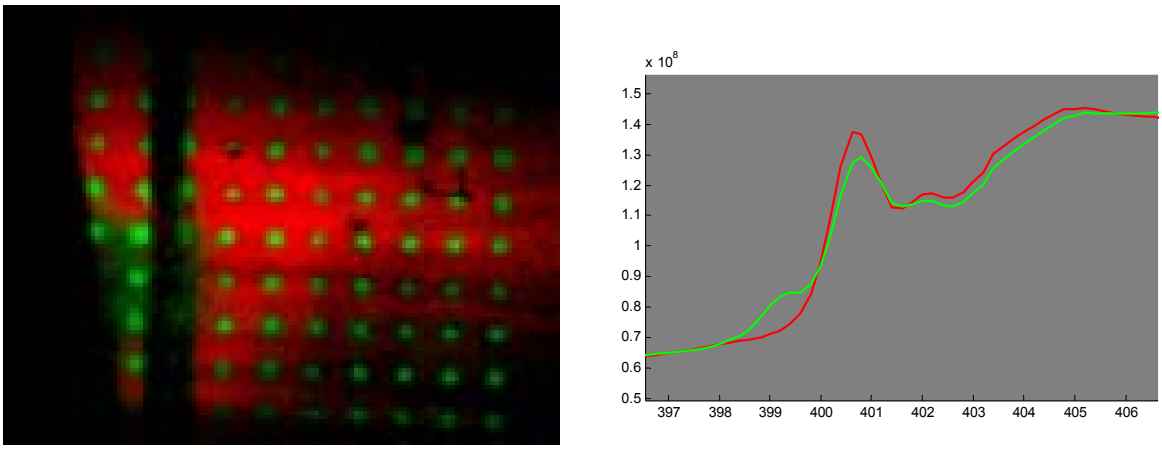

Figure 1. Nitrogen K edge NEXAFS hyperspectral image. A 300 NEXAFS image stack containing 500,000 (100mm^2 area) spectra reduced to a single image using AXSIA Multivariate Analysis. Shown is identification of nitrogen chemistry in single-stranded DNA arrays (green) distinguished from nitrogen chemistry in substrate (red).

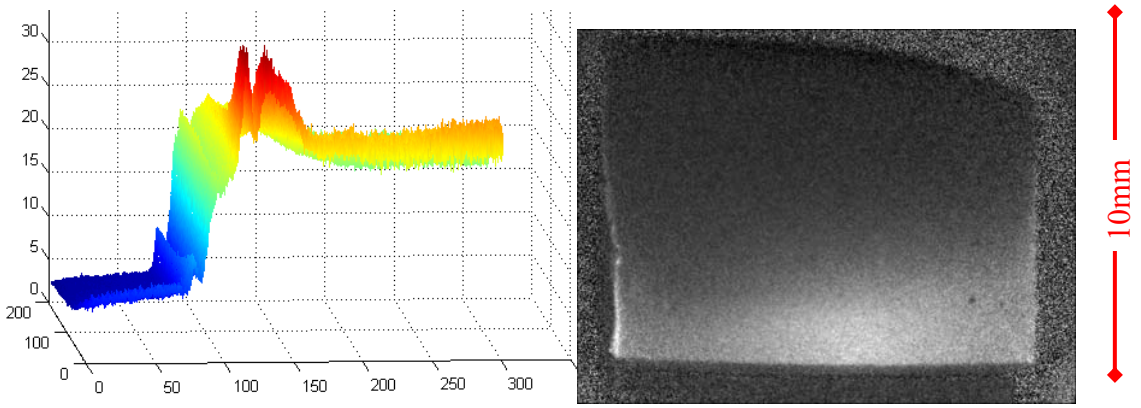

Figure 2. Identification of C-F bonds (left red peak) and C-C (right red peak) in a Self-Assembled Monolayer (SAM) gradient across ~100mm^2 area at 100um resolution.

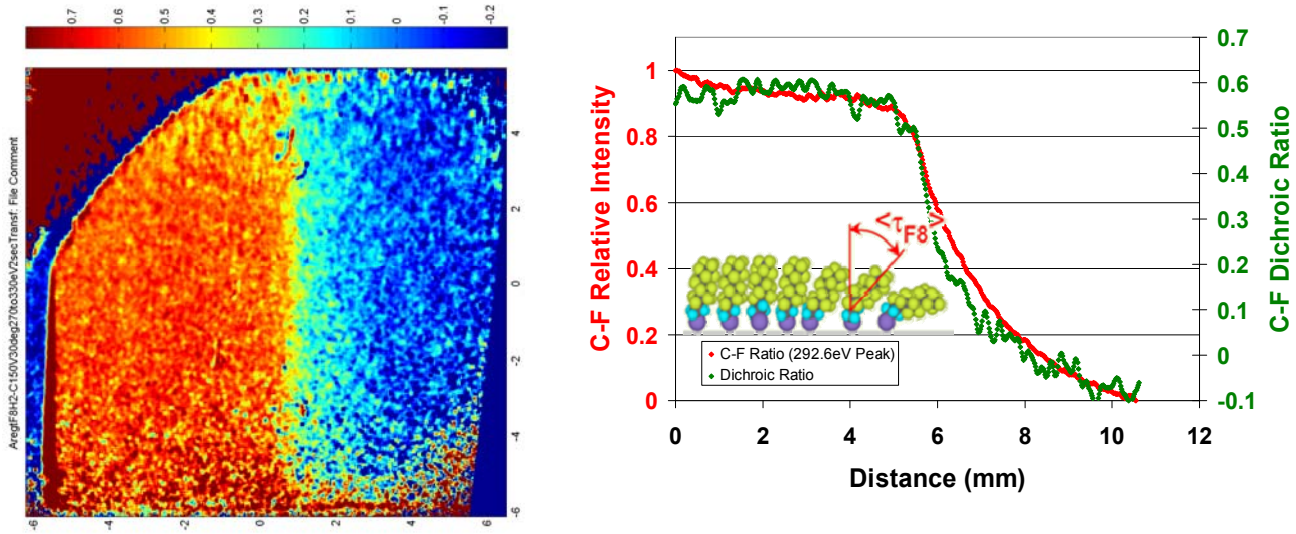

Figure 3. A **dichroic image** is shown at left and associated line plots (right) of a SAM gradient reflecting the orientation changes correlated with reduction in SAM coverage across the gradient in ~100mm^2 area.

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