Soft X-ray transmission X-ray microscopy - fuel cell and magnetotactic bacteria applications

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Synchrotron based soft X-ray scanning transmission X-ray microscopy (STXM) provides chemical analysis and quantitative mapping of chemical species in 2D and 3D with a spatial resolution better than 30 nm [1]. I will give an overview of current capabilities of STXM and show how the spatial resolution is being extended into the sub-10 nm range using coherent diffraction imaging with ptychography [2,3,4]. Cartoons of STXM and ptychography are presented in **Fig. 1**. I will present examples where these techniques are used to address important problems in both fundamental science and applied technologies.

The world faces a crisis caused by fossil fuel combustion. Transportation causes over 20 % of global CO_2 emissions and is the most challenging to economically convert to non-polluting alternatives. Fuel cells provide similar performance to automotive internal combustion engines (ICE), including range and rapid refueling. However further improvements in durability and cost reduction are sought. STXM and ptychography studies of polymer electrolyte membrane fuel cell (PEM-FC) catalyst layers are providing valuable information to automotive fuel cell developers. In particular, quantitative mapping of the mission-critical proton conducting ionomer in the cathode is being used to optimize formulations and processing, thereby improving performance and reliability [5,6]. An example of quantitative mapping of the ionomer, carbon support and Pt catalysts components in a PEM-FC cathode is presented in **Fig. 2**. Species-specific C 1s and F 1s spectra are used to convert optical density images into thickness maps, which are then combined as a color composite to view the spatial relationships. Since ionomer is very radiation sensitive [7], STXM provides an attractive alternative to analytical transmission electron microscopy (TEM-EELS or TEM-EDX) [8]. While 2D projection mapping as in Fig. 2 can be done with negligible radiation damage [5-8], it is the 3D chemical structure and porosity that control performance and thus are the ultimate analytical target. Recent development of a compressed sensing tomographic reconstruction method [9] has allowed high quality 3D STXM mapping of ionomer with minimal radiation damage [10]. Progress is being made on using spectro-ptycho-tomography to get higher spatial resolution 4D mapping of PEM-FC cathodes.

Magnetotactic bacteria (MTB) biomineralize chains of high quality magnetic single crystals (magnetite or greigite magnetosomes) that provide an internal compass which orients cells along the earth's magnetic field. This effect, in combination with oxygen chemotaxis, allows MTB cells to find their preferred environment at the oxic-anoxic boundary. Fe L-edge spectroscopy and X-ray magnetic circular dichroism (XMCD) of MTB measured by STXM [11] and ptychography [12] are being used to study MTB and provide insights into magnetosome formation. **Fig. 3** is an example where spectro-ptychography is used to characterize the chemical states of cytoplasmic Fe, immature magnetosomes, and precursor structures, relative to the mature magnetosomes [12]. Early STXM results will be presented from a time course study in which MTB are grown in the absence of iron and then magnetosome formation is initiated by placed non-magnetic cells into Fe containing media, followed by regular sampling as the magnetosomes form. Such studies promise to provide new insights into one of the simplest biomineralization processes under well-defined genetic control [13].

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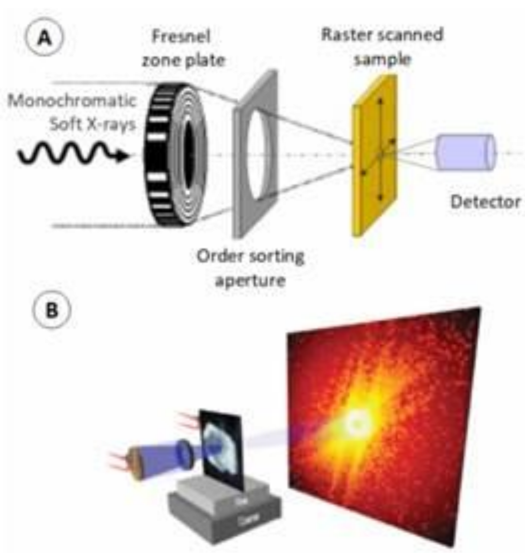

Fig. 1 Schematic of (a) STXM and (b) ptychography.

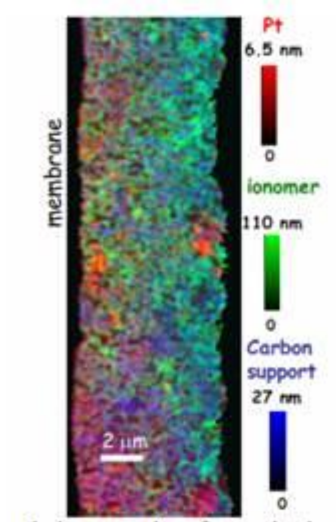

Fig. 2 Color coded composite of quantitative 2D projection maps of Pt, ionomer and carbon support in a PEM-FC cathode, derived from C 1s and F 1s STXM.

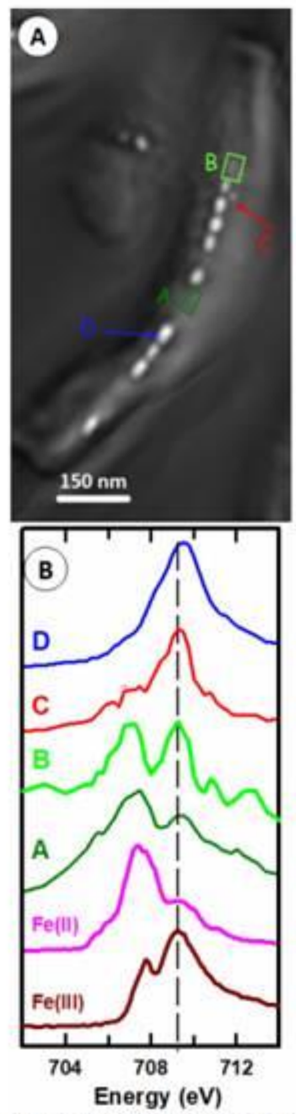

Fig. 3 (a) Ptychography image at 709.6 eV of an MTB cell. (b) Fe L_3 spectra of the color coded regions, compared to reference Fe(II) and Fe(III) spectra.