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Soft Matter X-ray Microanalysis in the Analytical Electron Microscope

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The recent developments in large solid angle x-ray detectors [1] has not only increased the ability of microanalysts today to perform high spatial resolution spectroscopy in the Analytical Electron Microscope (AEM) during materials science investigations, but it has also opened up opportunities to begin studying more challenging systems involving soft-matter and/or biological systems including experiments involving cryo-EM. In order to assess the viability of the new detector geometries, experiments have been performed using an FEI Titan AEM, equipped with the most recent generation of Dual X SDD systems developed by Thermo Fischer Scientific and Bruker. Two different and challenging system were chozen to test the veracity as detailed below.

The first, is a soft matter system studied consisting of a polyamide film ~ 100 nm in thickness, made via interfacial polymerization of *m*-phenylenediamine and trimesoyl chloride which was subsequently incubated in heavy metal ion solutions. After removal from aqueous solution and rinsing of residual salts, the film was supported on a gold mesh Quantafoil carbon film, dried and then studied in-vacuuo. High beam currents, with long analysis times were all unsuccessful due to beam damage of the polyamide. Multi-frame hyperspectral image measurements were conducted at 200 kV at < 200 pA beam current, dwell times of 100 usec/pixel, using drift correction and the Dual X system in a high visibility low background Be holder. Shown in figure 1, is a hyperspectral result of a region of interest from the film suspended over a hole in the carbon film. Comparison of images at the beginning and end of the analysis confirmed only minor microstructural damage was detectable. Site specific localization of the heavy metal species are clearly identifiable on microstructural folds in the polyamide. Temporally resolved hyperspectral images confirm no significant mass loss and/or translocation during the measurements. Of importance is the ability to now identify, without significant irradiation effects and mass loss [2] the microstructure and elemental distributions in this polyamide system. The affinity of various elements to the microstructural features, in this case folding in the film is of key importance and understanding the interaction of this elemental localization with the convoluted microstructure and it's applications for technological use is the subject of ongoing research in this system.

The second area studied was soft matter requiring cryo-EM technology [3]. Magnetotactic bacteria are organisms which contain intracellular organelles, composed of linear chains of nano-scale magnetic minerals and are of interest by a diverse community that ranges from palentologists and environmentalists, to biological and physical scientists. Numerous imaging methodologies have been developed to studies these bacteria ranging from simple bright field/dark field imaging to holography to conduct localized magnetic field studies, some being more successful than others [4,5]. For our purposes we chose to investigate whole Magnetospirillium Gryphiswaldensea (courtesy of Prof. D. Schüler, University of Bayreuth, Germany). These were drop cast onto hydrophilic Quantafoils which were then blotted and plunge frozen using a Vitrobot MarkVI (Thermo Fisher Scientific). The frozen grids were transferred to Fischione tomography cryo holder (model 2550) to maintain the sample at

liquid nitrogen temperature inside the Titan AEM instrument for our experimental measurements. A hyperspectral image set is shown in Figure 2. Low beam currents of 5 pA at 200 kV were used to insure minimal damage of the bacteria. Readily visible are Fe rich magnetozomes, embedded in the carbon cellular matrix, while the widespread Oxygen signal is due in large measure to the ice encapsulation of the bacteria. The Fe hyperspectral images provide unequivocal identification of the magnetozomes and in many instances provide identification that for some magnetozomes where the conventional HAADF imaging fails. Optimization of the hyperspectral/cryo-EM capabilities is in progress to further improve these capabilities for soft matter research [6].

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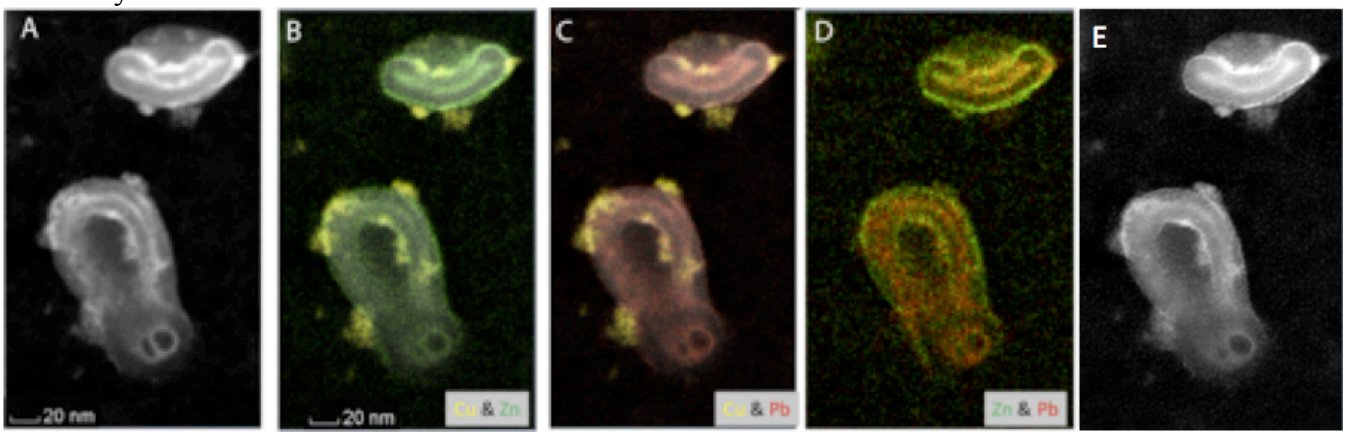

Figure 1.) Hyperspectral images of site specific heavy metal localization to folds of polyamide film.
A) HAADF preXEDS, B) Cu & Zn, C) Cu & Pb, D) Zn & Pb, E) HAADF postXEDS measurement (333 Frames)

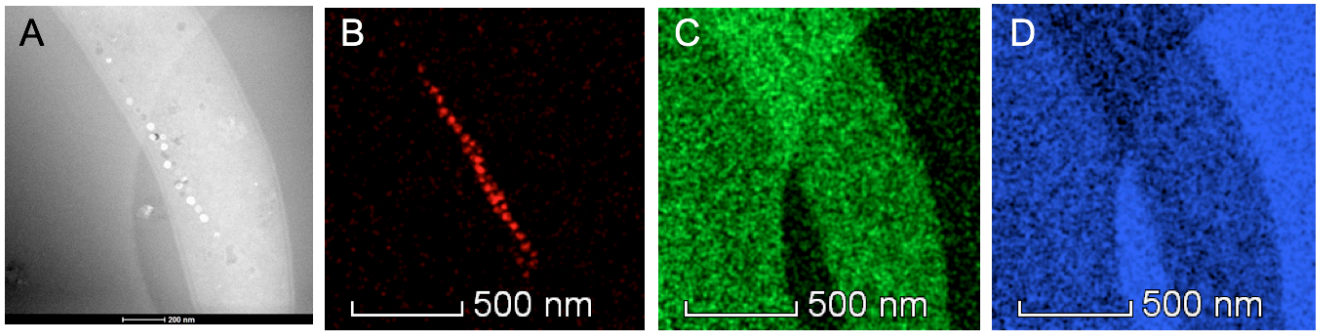

Figure 2.) Hyperspectral images of frozen magnetotactic bacteria, Eo:200 kV, 5 pA, Dual X detector.
A) HAADF image, B) Fe , C) Carbon, D) Oxygen elemental distributions.

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