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Exploring Electron Energy-Loss Spectroscopy for Characterization of Structured Fluids

Brittany Ford[1*] and David W. McComb[1, 2]

[1.] Department of Materials Science & Engineering, The Ohio State University, Columbus, OH, United States.
[2.] Center for Electron Microscopy and Analysis, The Ohio State University, Columbus, OH, United States.
*Corresponding author: ford.1027@osu.edu

Major developments in cryogenic electron microscopy (cryoEM) have been driven by the structural biology needs in life sciences. However, there are beam-sensitive samples in materials science that can benefit from cryogenic approaches as well. Structured fluids, materials that contain multiple phases and display a complex array of flow behaviors, are abundant in our everyday lives and present a great and unexplored opportunity for characterization using electron energy-loss spectroscopy (EELS) in scanning transmission electron microscopy (STEM) [1]. Due to the sensitive nature of these materials, they must be plunge frozen and imaged using cryogenic electron microscopy for their structure and chemistry to be investigated [2]. In this project, the aim is to use a cryogenic sample holder to stabilize plunge-frozen structured fluid samples and use STEM-EELS to investigate the chemical interactions between components. The mapping of chemical matter in structured fluid sample will help to understand how this material interacts with packaging products, skin and even microorganisms.

A Gatan cryo-transfer holder will keep the vitrified samples stable during analysis in the Titan 60-300 at the Center for Electron Microscopy and Analysis (CEMAS) facility using 300kV monochromated beam and an EELS system fitted with a K2 direct electron detector. Initially we will utilize TEM low-dose capabilities in order to find the best grid squares, obtain sample images, and save locations for STEM-EELS analysis. A gold standard sample will be used for the initial imaging alignments to prevent excess beam damage to the sample. Low-dose methods can prevent beam-induced changes, while the use of a direct electron detector can mitigate the signal-to-noise ratio (SNR) in the spectra[3].

In particular, the sample that is being explored is a surfactant mixture with and without an active salt ingredient. While imaging provides insight into the structure of the fluid, there is no information about the location of the salt. With the spatial resolution provided by STEM-EELS, the goal is to track the location of where the salt deposits within the sample's vesicle lamella or within the vesicle itself.

The Thermo Scientific Krios G3i Cryo-TEM at CEMAS will be utilized for energy filtered transmission electron microscopy (EFTEM) elemental mapping experiments. A small energy range will be used for the energy selecting slit and moved in increments to investigate the possibility of obtaining information pertaining to the chemical bonding environments of the research materials in this proposal. Using a larger field of view to spread out the electron dose, this could be a valuable tool for monitoring elemental locations. By inserting a slit at the dispersion plane and adding other lenses beyond that slit, very specific inelastic scattering events at characteristic energies in the EELS dataset can be used to form (EFTEM) images [4]. This is another way to achieve elemental mapping using STEM-EELS.

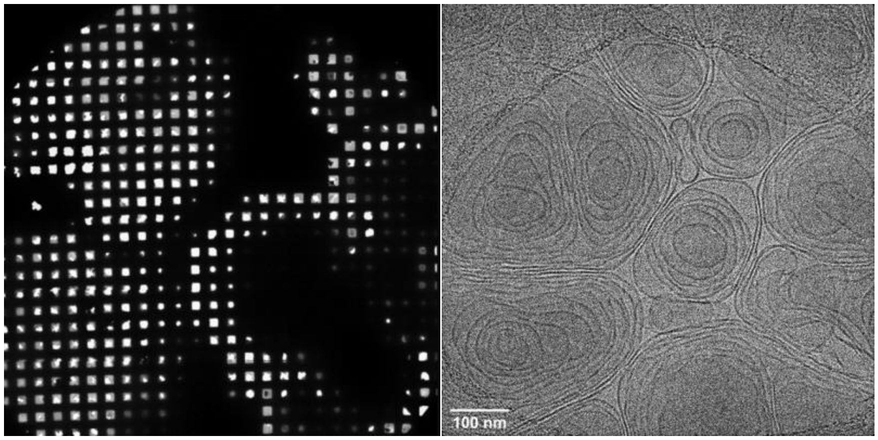

Figure 1. Structured fluid images taken using the Falcon direct electron detector in a Glacios microscope. An atlas image showing the entire sample grid is seen on the left. The sample's lamellae shown on the right.

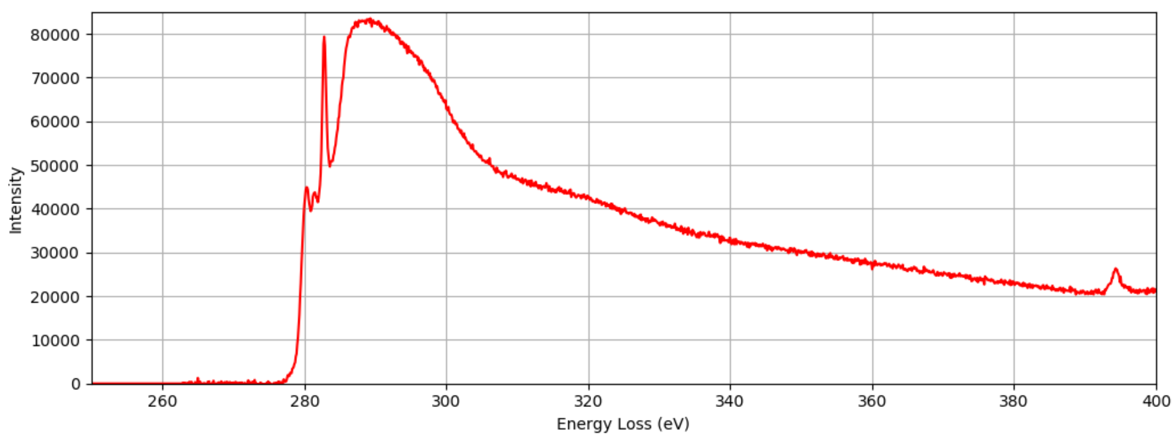

Figure 2. STEM-EELS spectrum collected from the active salt ingredient.

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