CRYOGENIC SCANNING ELECRON MICROSCOPY: Preparation & Characterization

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Structure-property relationship is important for the optimization of materials application. To understand this relationship, it is important to analyze basic building block of the materials and dynamics of the interactions at molecular scale. However, labile and disorder status of soft materials challenges material scientists to characterize through conventional method.

The goal of microscopy is to improve resolution and minimize specimen alternation during observation and specimen preparation. However, direct observation of microstructure has been challenging because they can be disturbed as the specimen is prepared. Cryogenic electron microscopy (cryoEM) technique is among the most powerful to characterize soft materials such as colloids, polymers, and emulsions at the nanometer scale. This characterization method does not require implicit models or assumptions about the structure. We can also visualize structure under dynamic conditions, capturing each stage of development.

Cryogenic scanning electron microscopy (cryoSEM) is complimentary method along with cryogenic transmission electron microscopy and freeze fracture electron microscopy and allows us to elucidate the structure at all length scale.

In this work, we discuss the preparation of the cryoSEM specimen (Figure 1) and the characterization of soft matter. CryoSEM can helps to reveal formation and development of nanoemulsions from microemulsions [1], analyze the pore size of mechanically tunable, nonswellable polymer hydrogels in hydrated status [2], observe drying process of paint coating as function of the time [3], and visualize oil-into-water spontaneous emulsification process [4] shown in Figure 2.

References:

[1] H Lee *et al*, Langmuir **30** (2014), p. 10826.
[2] J Zheng *et al*, ACS Macro Letters **6** (2017), p. 145
[3] Y Wu *et al*, J. Coat. Technol. Res **14** (2017), p. 455
[4] D Riehm *et al*, J. Colloid Interface Sci. **487**(2017), p. 52

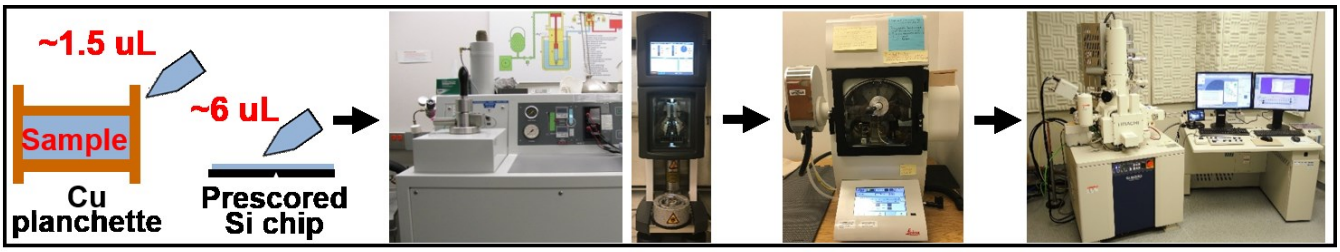

Figure 1. Ilustration of cryogenic scanning electron microscopy specimen preparation method: Sample application, freezing, etching, and observation.

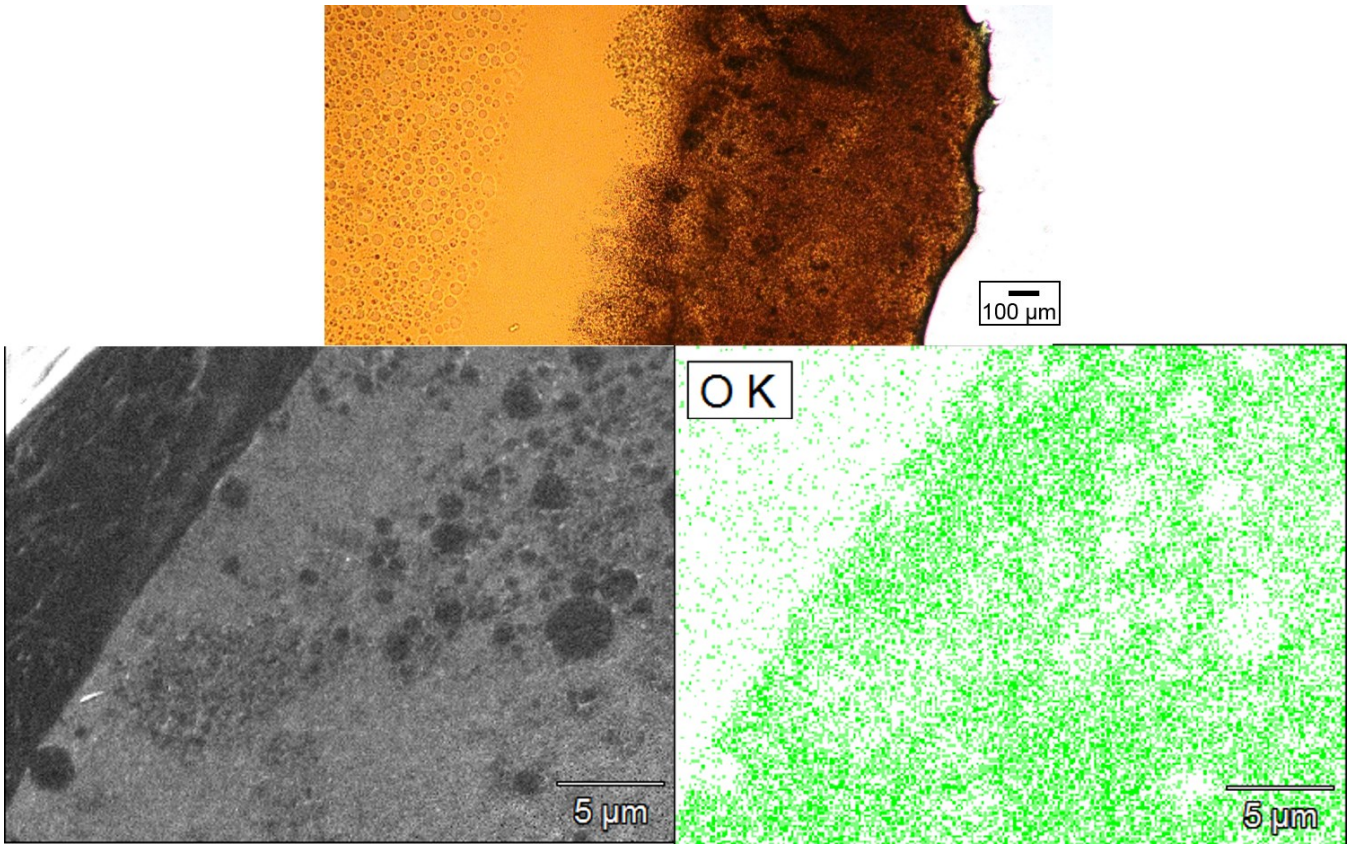

Figure 2. Example of self-emulsification with crude oil, sea water, and dispersant (TWEEN 80, lecithin) mixture. Top: Image by light microscopy. Bottom left: Image by cryogenic scanning electron microscopy (cryo-SEM) Dark contrast is oil regime and light grey is sea water regime. Bottom right: Oxygen elemental mapping by cryo-SEM and electron dispersive X-ray spectroscopy.