Experiment Brief

K3 IS Camera and 626 Liquid Nitrogen Cryo-Transfer Holder

Title
Imaging discrete ions at a liquid-solid interface using low-dose cryo-electron microscopy (cryo-EM) and electron counting.

Gatan Instrument Used
The K3® IS camera delivers simultaneous low-dose imaging via real-time electron counting, fast continuous data capture, and a large field of view. The Model 626 Liquid Nitrogen Cryo-Transfer Holder enables frost-free low-temperature specimen transfer and subsequent imaging of radiation-sensitive frozen samples at high resolution with precise temperature measurement.

Background
Cryo-EM imaging has revolutionized structural biology by enabling imaging of proteins and other biomolecules at near-atomic resolution. Now, the same low temperatures and electron dose rates are being applied to other aqueous materials. In this case, the liquid-solid interface between a positively charged gold nanorod and an aqueous solution of phosphotungstic acid was studied to provide insight into the individual and collective behavior of ions at that interface. Interactions between a solid surface, ions, and the solvent govern the behaviors of a wide variety of technologically relevant materials, including batteries, catalysts, colloids, membranes, and supercapacitors, so understanding those interactions is essential.

Materials and Methods
The sample was prepared by plunge-freezing a thin liquid film on a lacey carbon TEM grid. The temperature was maintained at 100 K as the sample was inserted into an image-corrected Titan ETEM using a model 626 cryo-holder from Gatan. Images were collected with a K3 IS camera in counted mode with a dose rate of 500 e/Å²/s and a total dose of 100 e/Å².

Summary
The combination of low-dose imaging using the counted mode of the K3 camera and low temperature achieved using the model 626 holder enabled imaging of the ion distribution around a liquid-solid interface, with individual ions clearly resolved. Linear profiles and their FFTs were used to analyze the images, and this analysis found discrete layering of ions adjacent to the surface, as well as ordering along the layers. These were additionally compared with molecular dynamics and image simulations to aid in the experimental image interpretation.

Credit(s)
A special thanks to Stanford University, including Hao-Kun Li, Ze Zhang, Joel Martis, and Arun Majumdar.

Gatan, Inc. is the world’s leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.

This work was supported as part of the Center for Enhanced Nanofluidic Transport (CENT), an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Basic Energy Sciences under Award no. DESC0019112. Z.Z. and J.M. acknowledge financial support by the Air Force Office Scientific Research under grant no. FA9550-19-1-0309.

www.gatan.com