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The Center for Integrated Nanotechnologies

An Office of Science user facility jointly operated by Los Alamos and Sandia National Laboratories.

The Center for Integrated Nanotechnologies (CINT) offers world-leading scientific expertise and specialized capabilities to create, characterize, and integrate nanostructured materials at a range of length scales, from nano- to meso-scale. CINT helps scientists perform cutting-edge research in the areas of nanoscience and nanotechnology, providing FREE ACCESS for nonproprietary research. CINT facilities are open to the international research community, including government, academic, and industry researchers.

One of CINT's core research areas is in-situ S/TEM experiments. Early capabilities focused on electrical and electrochemical property measurements of materials during high-resolution imaging. More recently, the staff has expanded the available techniques to include gaseous environments (ETEM), liquid-cell, mechanics, ion irradiation, laser ablation, cryogenic, heating, and air-free transfer. In-situ investigations have focused on coupled structure-property measurements on real materials, with the CINT user community expanding many of the in-situ method developments.

Publication Highlights

CINT CAPABILITY HIGHLIGHTS

Energy Storage: Solid-liquid interfacial reactions control the performance of many of today's portable energy storage technologies. Understanding the mechanisms of ion transfer across these interfaces provides critical insight for engineering solutions to improve performance, such as artificial solid-electrolyte interphases. CINT can analyze these materials and systems using a variety of techniques.

- ***Open-Cell In-Situ Electrochemistry:*** Individual nanowires or nanoparticles may be observed during reaction with charge-transfer ions (lithium, sodium, potassium, etc.).

  This requires the use of a Nanofactory STM-TEM holder.

- ***Closed-Cell In-Situ Electrochemistry:*** CINT's Electrochemical Discovery Platform is a custom-designed microelectromechanical system platform

  Its capabilities include customization of electrodes while providing low current electrochemistry (1 pA), thin-fluid path lengths (~150 nm), high-chemical compatibility, and the ability to perform multiple experiments within one platform (10-electrode configuration). The platform is controlled with a custom-built Nanofactory 16-electrical lead TEM holder.

- ***Cryogenic FIB Lift-Out and Cryo-S/TEM:*** Intact investigations of electrode/electrolyte interfaces may be achieved by preparing an electrode-surface cross-section under inert transfer and cryogenic conditions in a focused ion beam (FIB)

  The sample may be cryo-transferred to an S/TEM for high-resolution structural and compositional mapping. CINT has a Gatan single-tilt or double-tilt cryo-TEM holder for this work. The cryo-transfer is enabled with the Leica workflows

Material Science
Natural Resources
Electronics

- ***Cryo-TEM:*** Investigations of electrolytes may be studied using plunge-freezing methods to rapidly cool a thin electrolyte layer, then image any structural features using TEM
- ***Vacuum-Transfer S/TEM Holder:*** Allows for loading of a TEM grid in an Ar-filled glovebox for direct transfer into the S/TEM or ETEM

Soft Matter and Beam Sensitive Materials: CINT recently established a cryo-TEM suite dedicated to minimizing the electron dose for imaging materials and interfaces in their native hydrated (or solvated state). This provides an optimized characterization of sensitive materials across multiple length scales. Instruments included are a Talos dedicated cryo-TEM, a Scios 2 Dual-Beam SEM/FIB equipped with a Leica cryogenically-cooled stage, and a Vitrobot for reproducible and controlled vitrification of hydrated samples. Uniquely, CINT also provides Leica cryogenic-transfer equipment including a sample-prep station (VCM), high-vacuum transfer shuttle (VCT500), and a cryogenically-cooled sputter coater (ACE600). This robust and complete workflow allows for the preparation of frozen, electron-transparent lamellae that can be transferred to the TEM without ever leaving a cryogenic environment.

- ***Talos L120C:*** Dedicated low dose, low kV TEM (user switchable from 20-120 kV) for imaging beam sensitive materials, and analyzing buried sample features and interfaces

    This TEM is optimized for imaging of biomimetic materials, polymers, nanocomposites, and their interfaces, nanoparticles and solution dynamics, and low-Z systems and their interfaces. It also includes a dedicated cryogenic electron tomography (cryo-ET) holder with an increased acquisition angle (± 70°).

- ***Scios 2 Dual Beam cryo-SEM/FIB:*** Contains a cryo-cooled SEM stage for surface analysis, analysis of buried interfaces, and serial sectioned 3D tomography of soft matter and nanomaterials in their native, hydrated state

    An in-column T3 detector is optimized for low-kV, low-energy secondary electron detection. Additionally, an EDAX Team EBSD Analysis System with Velocity and EDAX Octane Elite Super EDS System offers microstructural and compositional maps. A cryo-cooled EasyLift Nanomanipulator enables the preparation of frozen TEM lamellae.

- ***Leica Transfer Equipment:*** Leica EM VCM enables users to easily put their cryogenic samples on holders under liquid nitrogen

    Features such as an adjustable LED illumination, an adequate magnifier, and a tool dryer enable convenient handling of samples. Users do not have to refill nitrogen since the Leica EM VCM has automatic refill functionality. Due to active cooling with the Leica EM VCT500 shuttle, samples can be transferred simply and safely through the complete workflow from preparation to analysis. Samples can be maintained under vacuum throughout the entire process. Vacuum and temperature are displayed, so users can monitor their samples closely. The EM ACE600 sputter coater is a versatile high vacuum film deposition instrument with a cryogenically-cooled stage installed. Soon a glovebox attachment for an Ar-filled glovebox will be installed to complete this workflow for air-sensitive samples.

Corrosion: Reactions at the surfaces of metals in contact with various solutions can cause massive structural deterioration, in the form of uniform, localized, and pitting corrosion. In-situ S/TEM has recently been proven as a method to investigate the initiation and propagation mechanisms of localized corrosion, with implications for progression mechanisms that cause

- ***S/TEM Experimental Methodology for Pre-, In-situ, and Post-Characterization:*** Using ACOM, energy-filtered TEM, EDS mapping, nanobeam diffraction, and high-resolution imaging, the metal sample may be mapped structurally and for the identification of compositional variations

    An *in-situ* investigation may be performed using a Hummingbird Scientific liquid-cell microfluidic holder or CINT's static Electrochemical Discovery Platform. Post characterization allows for measurement of the thickness change in the sample, corrosion product identification, and final structure analysis.

- ***Cryo-FIB/SEM/EDS/EBSD:*** Bulk corrosion samples with the corrosive medium intact can be studied using the Scios SEM with an attached Leica cryo-stage

    This equipment allows for nanoscale imaging combined with EDS and EBSD mapping of the chemistry, phase, and orientations of a 3D volume surrounding a localized corrosion site.

Gas-solid Reactions: CINT offers a 300kV Titan G2 Environmental TEM which is monochromated, image-corrected, and equipped with a Gatan K2-IS camera. The gas cart provides high-level control over the inlet gas composition while minimizing contamination throughout the system. Many different studies may be conducted with an array of TEM holders. We also offer Protochips' AXON software for minimizing image drift during in-situ reactions.

- ***Catalysis:*** Gasses fed directly to the objective lens in the TEM include oxygen, hydrogen, nitrous oxide, methane, carbon monoxide, carbon dioxide, nitrogen, argon, and helium

    Gasses compatible with the Protochips Aduro double-tilt heating holder can enable high-temperature reactions.

- ***Environmental Surface Reactions:*** Oxidation, reduction, annealing, and electrical break down mechanisms may be investigated
- ***Hydrogenation:*** Coupled mechanical studies may be performed using a Hysitron PI-95 TEM holder
- ***Vapor-phase corrosion:*** a new system has been installed to allow vapor introduction into the ETEM, including water for deliquescence corrosion studies

PUBLICATION HIGHLIGHTS

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