University of Pittsburgh

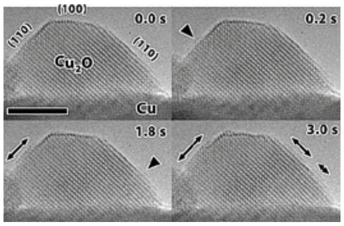
Environmental Transmission Electron Microscopy (ETEM) Catalysis Consortium

The ECC provides researchers with managed and comprehensive access to the cutting-edge *in situ* electron microscopy tools at Pitt. Dedicated technical staff aid in designing and carrying out experiments, project consultation, and can assist in data analysis.

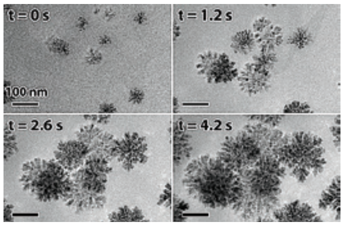
Dynamic *In situ* Environmental Characterization

Time-resolved visualization of a catalyst under meaningful reaction conditions and interfaces where one or more sides are not solid: e.g., solid/gas, solid/liquid, and liquid/gas. Acquisition rates up to 25 frames-per-second.

- Mixtures of up to two gases and one vapor, at pressures up to 10^{-2} Pa. Currently attached gas sources include H_2, O_2, CO, CH_3OH, and CH_4. This selection can be easily expanded. An *ex situ* reactor supports up to 1 atm.
- Heating (with or without gas) up to 1000 °C. The ECC has holders for both thin film ("bulk") and nanoparticle (on a membrane) specimens.
- A sealed-cell liquid flow holder enables TEM observation of specimens in a liquid environment.



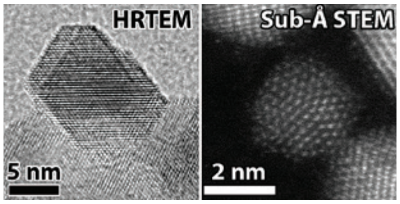
Layer-by-layer facet-preferenced nucleation and growth of Cu_2O island on Cu (100).



Growth of dendritic carbon nanoparticles in water/methanol solution.

Structure and Morphology Determination

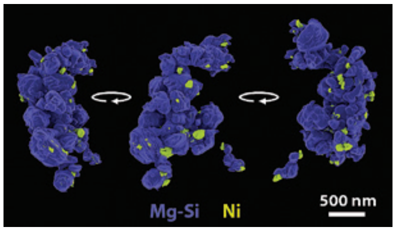
A powerful range of imaging and diffraction modes for determining the crystal structure, orientation, size, and morphology of materials from µm- to sub-Å-scale.



High-resolution TEM (HRTEM) and sub-Å ADF-STEM images of catalyst nanoparticles.

3D Structure and Composition (Electron Tomography)

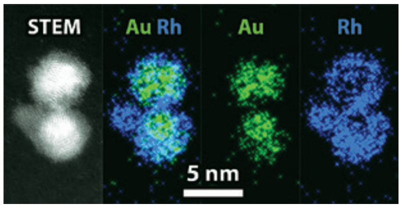
True, three-dimensional nm-resolution reconstructions of internal and external specimen structure and composition. Includes TEM, STEM, and EDXS tomography.



Tomographic reconstruction of Ni catalyst nanoparticles in Mg-Si.
Courtesy Stephen D. House.

Elemental Composition, Mapping, and Chemical State

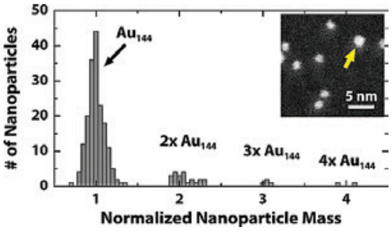
Energy-Dispersive X-ray Spectroscopy (EDXS) enables the quantification of elemental composition and even mapping of each element's spatial distribution, uniformity, or ordering. Electron Energy-Loss Spectroscopy (EELS) allows the direct investigation of nano-scale chemistry and electronic structure of a material, e.g., oxidation state.



Elemental distribution in core-shell 50:50 Rh/Au bimetallic nanoparticles via EDXS.

Quantitative Mass Measurement (Quantitative STEM)

A family of techniques where the image intensity is related to the number of atoms in the nanoparticle. Can be used to measure, e.g., mass dispersion of samples.



Quantitative STEM revealed the larger particles were coalesced integer multiples of Au_{144}.

(See: S.D. House, et al., Ultramicroscopy, 2017, 182, pp. 145-155.)

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Core Instrumentation

	HRTEM (Å)	STEM (Å)	Diffraction	Quant STEM	DPC STEM	EDS Res (≈ nm)	Area	Pt/Line/Map	EELS (eV)	Tomography	Gas	Heat	Liquid	SEM/BSE (Å)
Hitachi H-9500 ETEM	1.0		x			50	x				x	x	x	
FEI Themis G2 AC-S/TEM	1.0	0.7	x	x	x	< 1		x		x				
JEOL JEM-2100F S/TEM	1.0	2	x	x		< 5		x	1.0					
Zeiss Sigma 500 FE-SEM		x				x		x						15

ECC Pricing

	Rate ($/hr)		
	Hitachi H-9500 ETEM, JEOL 2100F S/TEM	**FEI Titan Themis G2 AC-S/TEM**	**Zeiss Sigma 500 FE-SEM**
Internal User	45	55	35
External, Non-profit	90	150	70
External, Commercial	135	225	105
ECC Staff (add-on)* Int/NP/Com	50 / 80 / 110	50 / 80 / 110	50 / 80 / 110

**Currently, all ETEM and AC-S/TEM usage must be ECC staff-assisted*

Example Typical Costs

Experiment Type	Services Performed	Hours	Total Cost (Int/NP/Com)
Structural/ Compositional Characterization	2 specimens typical, 3 for a quicker screening, or 1 for more in-depth characterization (HRTEM + ADF STEM + EDXS mapping). (Staff-assisted)	4	$380 / 680 / 980
***In situ* ETEM Experiment**	Gas/heating, structure/morphology characterization and evolution. (Staff-assisted)	8	$760 / 1360 / 1960

Want to Learn More?

ETEM Catalysis Consortium
3700 O'Hara Street
SB 60-63 Benedum Hall
Pittsburgh, PA 15261

E-mail us at: **AskECC@pitt.edu**

@PittECC

engineering.pitt.edu/ECC