# University of Pittsburgh

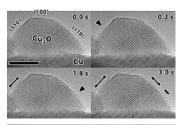
# **Environmental Transmission Electron Microscopy (ETEM) Catalysis Consortium**

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.

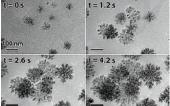
### Dynamic In situ Environmental Characterization

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<sup>-2</sup> Pa.
   Currently attached gas sources include H<sub>2</sub>, O<sub>2</sub>, CO, CH<sub>3</sub>OH, and CH<sub>4</sub>. 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.



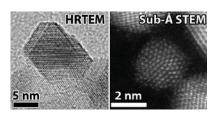
Layer-by-layer facet-preferenced nucleation and growth of  $Cu_2O$  island on Cu (100).



Growth of dendritic carbon nanoparticles in water/methanol solution

### **Structure and Morphology Determination**

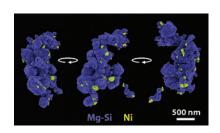
A powerful range of imaging and diffraction modes for determining the crystal structure, orientation, size, and morphology of materials from  $\mu$ m- to sub-Å-scale.



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

### **3D Structure and Composition (Electron Tomography)**

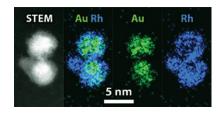
True, three-dimensional nm-resolution reconstructions of internal and external specimen structure and composition. Includes TEM, STEM, and EDXS tomography.



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

### **Elemental Composition, Mapping, and Chemical State**

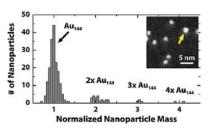
Energy-Dispersive X-ray Spectroscopy (EDXS) enables the quantification of elemental composition and even mapping of each element's spatial distribution, uniformity, or ordering. Electron Energy-Loss Spectroscopy (EELS) allows the direct investigation of nano-scale chemistry and electronic structure of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



Elemental distribution in coreshell 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<sub>144</sub>\* (See: S.D. House, et al., Ultramicroscopy, 2017, 182, pp. 145-155.)

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## **Core Instrumentation**

	HRIF	MA	(A) Diffre	Ction Quant	STEM	JEM EDS P	Area Area	Palin	EELS.	EV Tono	Gas Gas	Heat	Liquid	SEMB	st al
Hitachi H-9500 ETEM	1.0		х			50	x				х	x	х		
FEI Themis G2 AC-S/TEM	1.0	0.7	х	х	х	<1		х		х					
JEOL JEM-2100F S/TEM	1.0	2	х	х		< 5		х	1.0						
Zeiss Sigma 500 FE-SEM		х				х		х						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				

<sup>\*</sup>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
<i>In situ</i> ETEM Experiment	Gas/heating, structure/morphology characterization and evolution. (Staff-assisted)	8	\$760 / 1360 / 1960

### Want to Learn More?

**ETEM Catalysis Consortium** 

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