

A NEW METHOD FOR QUANTIFYING LIGHT DELIVERY IN A CLOSED-CELL GAS-PHASE *IN SITU* TEM SYSTEM.

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Photocatalytic materials are of growing interest for applications including CO₂ reduction, water purification, and solar fuel generation, yet photocatalysis remains underrepresented in *in situ* TEM studies [1,2]. A major challenge is integrating controlled light delivery while maintaining realistic gas environments, pressures, and accurate temperature control. Existing approaches often rely on specialized environmental TEM systems operating at relatively low pressures and can suffer from uncertainties in optical power delivery and photothermal effects [1].

Closed-cell gas-phase TEM platforms provide an alternative, enabling pressures up to 1 bar and temperatures approaching 1000 °C using MEMS-based chips that maintain realistic reaction environments. However, introducing light into these systems introduces challenges related to optical losses and illumination-induced temperature changes.

In this poster, we present a new methodology for introducing and quantitatively characterizing illumination in closed-cell gas-phase TEM (Fig. 1). The approach enables wavelengths from 350–2000 nm and combines a unique *ex situ* characterization with software-assisted measurements to determine optical losses and sample power density. Specialized MEMS chips maximize light throughput while maintaining calibrated temperature accuracy under illumination, establishing a practical framework for expanding *in situ* TEM studies of photocatalysts and other light-sensitive materials under realistic reaction conditions.

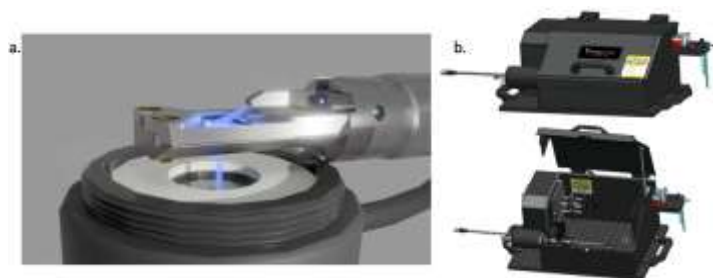


Fig. 1. a) Close up rendering of a new gas-phase holder (Protochips Sol for Atmosphere AX) with integrated optical fiber over a sensor for measuring power transmitting through the holder, which sits inside the *ex situ* characterization station and is performed before going into the TEM b) unique *ex situ* characterization station for measuring power density at the sample.

References

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High-resolution In-situ High-pressure and Temperature Gas Cell Transmission Electron Microscopy of Heterogeneous Catalysts at >20 bar and 450 °C

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A high-pressure transmission electron microscope (TEM) gas heating sample holder platform is presented which extends the operable pressure range for in-situ gas cell heating experiments to >20 bar. In-situ gas cell TEM has enabled investigation of morphological transformations in nanoscale catalyst systems exposed to experimental gases, revealing structure-property relationships and mechanisms affecting catalytic performance, but applicability in commercial systems has been limited in pressure to 2 bar. To reproduce real-world environmental conditions applied to heterogeneous catalysts, experimental pressures above 10 bar must be combined with temperatures of a few hundred degrees. Here we present a high-pressure gas environmental heating TEM sample holder that enables on-chip specimen heating, temperature sensing, and stable cells at pressures as high as 27.5 bar inside a microfabricated cell, see Fig 1 [1].

The high-pressure gas cell heating holder was demonstrated in a Titan 80-300 Environmental TEM (ETEM) at the Center for Functional Nanomaterials at Brookhaven National Laboratory to mitigate risk of microscope damage in the unlikely case of cell rupture. The holder integrity was demonstrated up to 20 bar of 1:3 CO₂/H₂ experimental gas combined with temperature up to 450 °C. To assess imaging quality and stability, Pt/CeO₂ nanocatalyst particles were imaged at 20 bar of 1:3 CO₂/H₂ experimental gas at 450 °C, see Fig 2. CeO₂ lattice fringes were resolved and distinguishable down to 3.1 Å, measured using the image fast Fourier transform (FFT). Excellent resolution is achieved with a zero-loss energy filter despite increased interference from the experimental gas at elevated pressures.

The combined high-pressure and high-temperature environment was demonstrated to be safe for usage inside the TEM yielding useful imaging and diffraction data. This capability to combine extreme environments will enable in-situ structural characterization and chemical analysis of heterogeneous catalyst transformations under typical industrial process conditions.

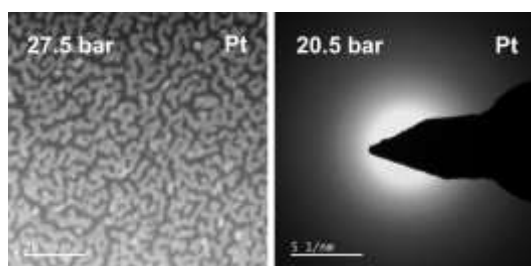


Fig. 1: PVD Pt in DF-STEM (left) at 27.5 bar of N₂ and SAED at 20.5 bar N₂ (right) [1].

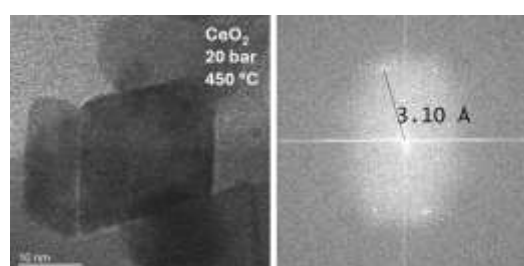


Fig. 2: BF-STEM (left) and FFT (right) showing resolution of 3.1 Å at 20 bar and 450 °C.

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Open Gas-Cell Transmission Electron Microscopy at 0.5 Å Information Limit

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Transmission electron microscopy (TEM) has advanced rapidly for visualizing matter at the atomic-scale. Ultimately a spatial resolution of ~ 0.5 Å resolution can now be obtained routinely, enabling visualizations at single-atom sensitivity and determination of the three-dimensional atomic structure of nanostructured materials [1,2]. Yet many nanostructures restructure under ambient or reactive environments, as their surfaces adapt dynamically to changes in their environment[3]. Hence, combining TEM at ~ 0.5 Å resolution with gas-cell technology would open up for visualizations of atomic processes relevant to catalysis, crystal growth, and corrosion. Herein, we report on such a new transmission electron microscope, VISION PRIME [4]. It builds on an ultra-stable Thermo Fisher Scientific SPECTRA ULTRA ETEM platform, featuring include a 5th-order objective-lens aberration corrector (CETCOR PRIME) for achieving a 0.5 Å information limit, a four-stage differential pumping system for confining ~ 10 mbar gas pressures near sample, a Wien-filter monochromator for extending the information limit and low-dose-rate Nelsonian illumination, and a Falcon 4i camera for sensitive electron detection. The microscope performance will be outlined and includes an 0.5 Å resolution and hourly optical stability, even in presence of reactive gas environments, enabling focal-series exit wave reconstruction. Examples of its benefit for studies of the dynamic and functional behavior of nanoparticles catalysts at the atomic-level will be given [2,4].

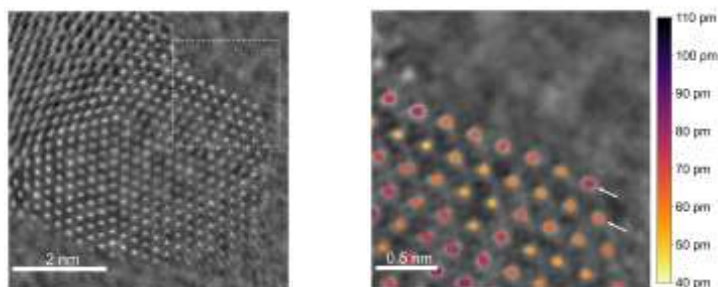


Fig. 1: Exit wave phase image of nanocrystalline Au on an amorphous carbon film (left), with the atomic column diameters superimposed as colored discs (right). Adapted from [4].

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GIS free lamella preparation for in situ heating TEM

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The general FIB procedure to protect and attach the sample to a suitable transmission electron microscopy (TEM) support, it commonly involves the use of a gas injection system (GIS). Unfortunately, the GIS deposited metal become mobile when the sample is heated for in-situ temperature dependent observations. We found the presence of platinum in the region of interest (ROI) at temperature as low as 250°C. The motion of platinum over the ROI can hinder the observation or quantification of the actual sample.

For this reason, we developed a work flow that avoid the use of GIS deposited metals. In brief, we start by protecting the sample with a silicon mask. Successively, the lamella attachments are done by redepositing locally sputtered material generated by the focus ion beam (FIB). In more details, we redeposit sample material to attach the sample to the nanomanipulator needle as shown in Fig. 1. Afterward, we attach the lamella to the in situ chip by making a small hole on the side pads of the lamella [2]. An SEM image of the lamella attached to a Protochip heating chip is shown in Fig. 2.

In addition to avoid the platinum for protection and welding, the initial site of the lamella is prepared with a dedicated trapezoidal geometry [2]. The chosen shape allows top and bottom milling after attachment to the chip, as well as providing enough material for the GIS free attachment.

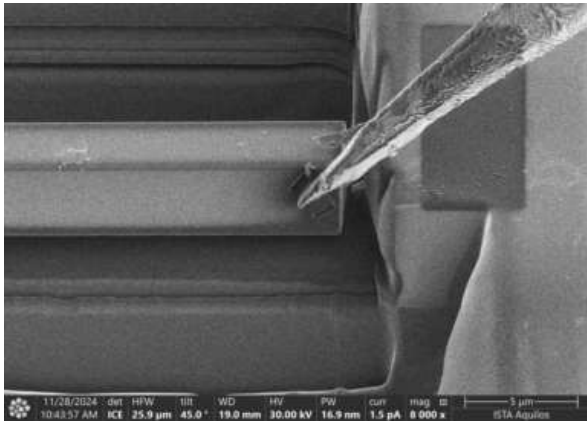


Fig. 1: Sample attachment to needle

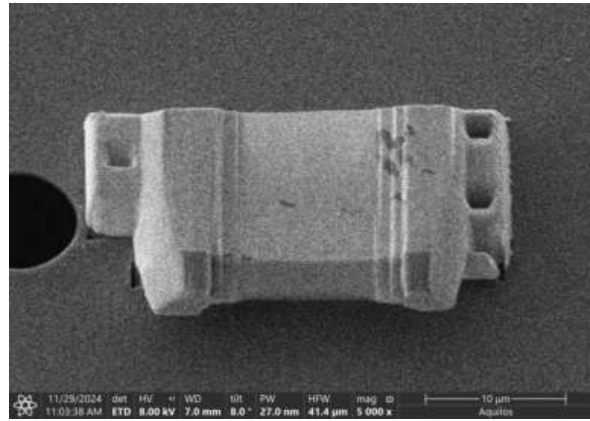


Fig. 2: Lamella attached to *in situ* chip

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INTEGRATED IN-SITU EM PLATFORM FOR CROSS-SCALE CHARACTERIZATION UNDER COUPLED MULTI-FIELDS

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Understanding material behavior across different scales (from microns to atoms) under realistic multi-field environments is crucial for advancing both fundamental and applied materials science. We have developed an integrated in-situ EM platform that enables dynamic, cross-scale observation during simultaneous mechanical, thermal, and electrical loading. The system is designed to function within SEM, FIB, and TEM columns, employing modular MEMS-based stages and specialized holders that ensure compatibility and precision across these imaging modalities.

This versatile platform allows researchers to track microstructural evolution from the microscale down to the atomic level, under complex thermo-electro-mechanical stimuli. It has been applied to investigate a range of phenomena including deformation mechanisms in metals, phase transformations, and interfacial dynamics in energy materials.

To support studies on air-sensitive specimens such as solid-state battery components, the platform is integrated with a vacuum/controlled-atmosphere FIB-EM transfer system, ensuring seamless and contamination-free sample transfer across glovebox, FIB, and various electron microscopes.

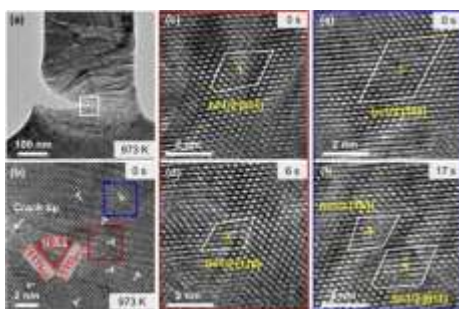


Fig. 1: Tungsten fractures at 700°C in a ductile manner via a strain-induced multi-step BCC to FCC transformation and dislocation activities within the strain-induced FCC phase [1]

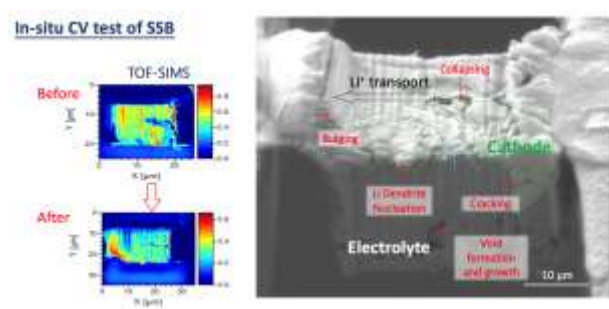


Fig. 2: In-situ cyclic voltammetry testing of sulfide electrolyte in a FIB. Lithium-ion transport pathways, dendrite growth and cathode cracking phenomena were observed in situ under SEM.

References:

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Investigation of Twisted Free-standing Membranes Using Advanced Microscopy

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In recent years, ferroelectric polarization-induced topological structures — such as polar skyrmions, vortices, flux-closure domains, and related configurations — have attracted significant interest due to their emergent physical phenomena and potential applications in next-generation electronic devices. To date, however, these complex topological patterns have been predominantly realized in substrate-supported superlattice systems, where limited structural tunability and fabrication complexity constrain their broader implementation and practical integration. In contrast, twisted free-standing thin films have recently emerged as a promising alternative platform, offering enhanced structural flexibility and tunable polar properties. In these systems, the formation of polar textures is strongly governed by strain gradients and lattice rotations induced by controlled twist angles. Despite their potential, direct investigation of the interfacial structures and individual layers remains highly challenging, as elucidating the underlying mechanisms requires characterization with true atomic-scale resolution [1–3].

In this work, we fabricated a series of twisted free-standing oxide ferroelectric membranes and systematically investigated their structural and polar configurations. Using aberration-corrected scanning transmission electron microscopy (STEM), we directly visualized a variety of emergent polar topological structures and clarified the mechanisms driving their formation. Our results provide new insights into the evolution of complex polar textures in twisted free-standing membranes and establish a foundation for future studies and potential device applications.

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Superconducting devices for operation in TEM

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Low energy dissipation in superconductors is advantageous for their operation under reduced heat transfer conditions on a thin membrane in the high vacuum environment of a transmission electron microscope (TEM). We have developed a technology for the preparation of 20-nm-thick single-crystalline SrTiO₃ (STO) membranes, which can be used directly for the epitaxial growth and study of electron-beam-transparent YBa₂Cu₃O_{7-x} (YBCO) and SrRuO₃ films [1]. This approach avoids the damage to the films that is introduced by traditional TEM sample preparation methods, such as mechanical grinding and focused ion beam milling.

Nanobridge Josephson junctions (nJJs) and nanoSQUIDS can be used for the local excitation and detection of magnetic fields up to THz frequencies. We have developed Nb-based nanoSQUIDS with proximity effect nJJs on 100-nm-thick low-strain SiN membranes for possible applications in the TEM [2]. Electron beam lithography, an HSQ resist mask and high selectivity reactive ion etching with pure SF₆ gas were used to pattern nJJs with widths down to 10 nm, which is comparable to coherence length in thin films of Nb. The superconducting films were deposited and nanostructured under standard conditions on bulk Si substrates, followed by wet chemical etching of the substrate in 20 % KOH to the back side of the SiN buffer layer.

We have also developed step-corner Josephson junctions for high- T_c superconducting electronics, including SQUIDS, nanoSQUIDS and quantum circuits [3]. Superconducting quantum nanodevices exhibit high sensitivity, low intrinsic noise and low dispersion at high frequencies, making their use as emitters and detectors in close proximity to samples under investigation on TEM grids attractive. In contrast to the conventional step-edge high- T_c Josephson junctions, this approach makes use of a single grain boundary in a superconducting film grown over the bottom corner of a step edge, thereby allowing the reproducible creation of ultimate high- T_c Josephson junctions, which can be integrated at any position in a sophisticated high- T_c superconducting circuit.

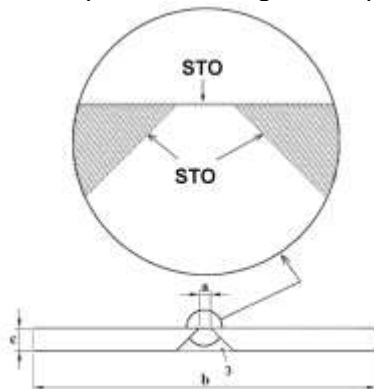


Fig. 1: A TEM grid consisting of a single-crystal STO membrane bonded to an STO substrate [1].

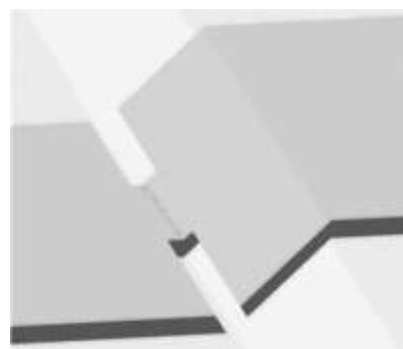


Fig. 2: Josephson junction on a single grain boundary located at a selected corner of the substrate step [3].

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In-Situ Microwave Excitation and Spatially Resolved Magnetic Resonance in Transmission Electron Microscopy using Broadband Radio Frequency Sample Holders

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The integration of high frequency excitation in transmission electron microscopy (TEM) enables the investigation of dynamic material response on the nanometer length scale. Here, we report a TEM specimen holder platform that introduces broadband microwave excitation directly to a TEM sample, while maintaining imaging and diffraction capabilities.

We have used this approach to visualize the effect of microwave fields on electron beam deflection in low-angle diffraction experiments. Ongoing magnetic resonance experiments in the TEM include the detection of electron spin resonance markers and ferromagnetic resonance from nanoscale magnetic structures through their influence on the microwave field and electron-beam response, allowing resonance phenomena to be studied directly in the microscope rather than only through external spectroscopy.

This specimen holder platform supports broadband excitation up to ~67 GHz and exchangeable chip-based samples with reproducible electrical contacts. Ongoing developments include the integration of additional experimental stimuli, such as electrical biasing and cryogenic operation, in order to enable TEM studies that combine microwave excitation with electronic and temperature control.