

# ATOMIC RESOLUTION STEM BELOW 10 KELVIN

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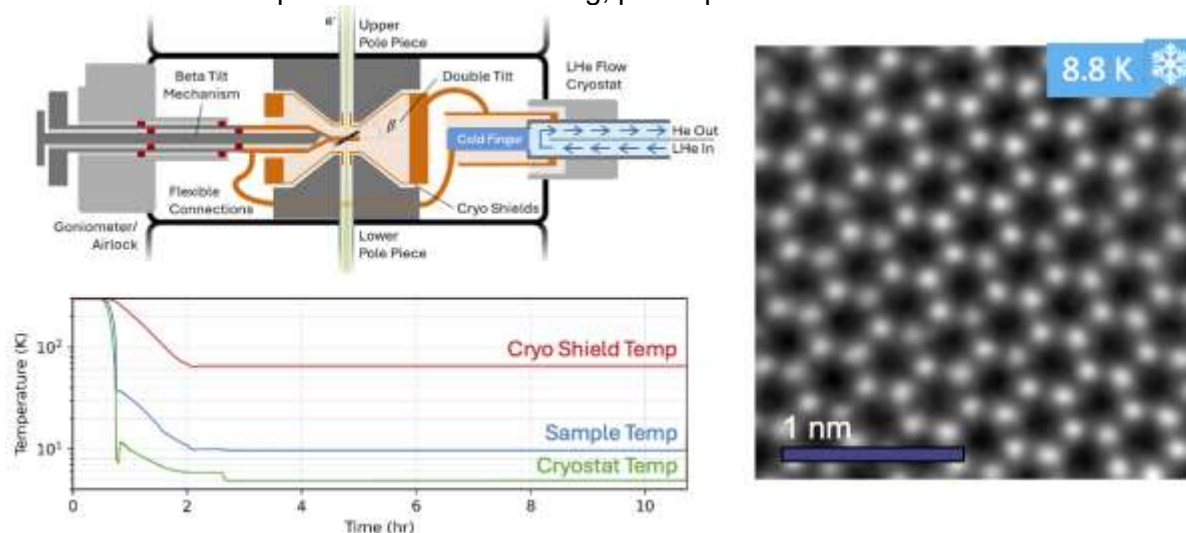
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Exploring the emergent properties of quantum materials requires cooling of the sample to temperatures much lower than liquid N<sub>2</sub>, driving interest in electron microscope sample stages and holders that can reach temperatures of 10 K and below. We have designed and built a system for this. [1] See Figure 1a. A liquid helium flow cryostat is rigidly mounted to the column. The sample is introduced via a separate port and coupled to the cryostat via a flexible braid, isolating vibrations from the cryostat and leaving the central bore of the sample rod clear for a motor-driven tilt mechanism and instrumentation wires for heaters, sensors, and biasing. The system includes thorough in-column cryo-shielding, protecting the sample from infrared radiation and improving the vacuum around the sample beyond the native UHV conditions. The helium consumption rate is 2-3 liquid liters per hour, so a 60-liter dewar has a holding time of 20-30 hours (or longer with a bigger dewar). The other capabilities of the Nion HERMES200 are maintained, including an ultra-bright cold-field emission source, less than 5 meV energy resolution EELS, open-source software, and flexible detector options.

Figure 1b combines this cryostage with a 4D-STEM detector to study monolayer MoS<sub>2</sub> at a series of temperatures from 8.84 K (shown here) to room temperature with high-resolution ptychographic phase reconstructions (ePIE) [2]. Individual atoms are clearly resolved. Built-in to Nion Swift, quick initial processing of the 12nm defocused 4DSTEM datasets with direct ptychography guides the prioritization of more-detailed (slower) iterative reconstructions. Latest results will be presented at the meeting, plus a peak at future directions.



*Fig. 1 (top left) schematic drawing of the Denali liquid helium cryostage with LHe flow cryostat, flexible cooling braid, removeable coupling to the sample holder with double tilt, thorough cryoshielding; (bottom left) cooling progression showing the initial cool-down and holding time >8 hours; (right) atomic resolution image of MoS<sub>2</sub> sample at 8.8 Kelvin reconstructed with iterative ptychography (ePIE) from a 4DSTEM data set. 1Å scan step, 12 nm defocus, dwell time=1 ms, I<sub>p</sub>=15 pA, detector=ELA (fixed after EELS)*

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# MERLIN T4, DATA DRIVEN DETECTOR FOR TEM

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Technological advances in transmission electron microscopy (TEM) detectors enable the characterisation of more challenging specimens, improved experimental performance, and new measurement approaches. Merlin T4, the newest detector developed by Quantum Detectors, is based on the Timepix4 ASIC from the Medipix collaboration at CERN [1], representing a generational step forward by combining and extending the strengths of earlier hybrid pixel counting detectors within a single platform.

The Merlin T4 architecture supports two operating modes: data-driven and frame-based. In data-driven detection, each electron produces an event packet recording position (X, Y), time of arrival (ToA), and deposited energy (ToT), providing more than a ten-fold increase in counting capacity compared to Timepix3, and supporting beam currents up to 200 pA with 196 ps ToA precision. Frame-based operation reaches up to 40k fps with 8-bit dynamic range across the full 512 x 448 pixel array.

In this presentation, selected advantages for TEM will be demonstrated, focusing on high-speed event detection and four-dimensional scanning transmission electron microscopy data acquisition at native scan speeds, enabling multi-frame averaging and drift correction for improved signal quality (Fig. 1), while additional benefits for diffraction, spectroscopy, and correlative experiments will be also discussed.

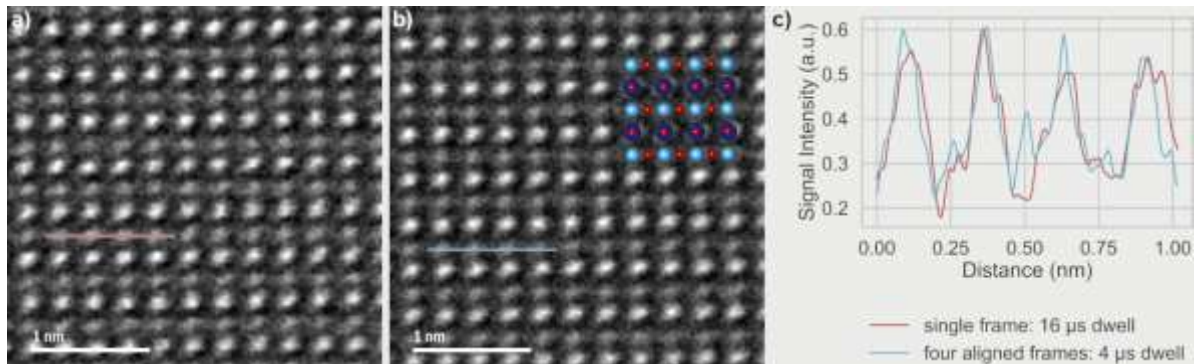


Fig. 1: ADF images of an 80 nm thick STO sample ([110]) acquired with the Merlin T4 and generated from a 4D-STEM dataset: a) single 16  $\mu$ s pixel dwell time scan; b) drift-corrected 4x4  $\mu$ s image showing improved contrast and reduced noise; c) line profile across Ti and O columns showing enhanced signal from weakly scattering oxygen. Data were acquired on a JEOL ARM 200cF at University of Glasgow, 200 kV, 38 pA probe current, 512x512 scan [2].

## References:

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[2] Acknowledgements: Fred Rendell-Bhatti, Kayla Fallon, Stephen McVitie, Donald MacLaren, MCMP group, University of Glasgow

# LIQUID HELIUM TEM SAMPLE HOLDER: SWIFT COOL-DOWN AND LONG HOLDING TIME

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Quantum materials host unique electronic and magnetic properties—including superconductivity, charge and spin ordering—predominantly observed at cryogenic temperatures [1,2]. While progress in cryogenic transmission electron microscopy (TEM) methodologies have led to the development of liquid nitrogen (LN<sub>2</sub>) cooled side-entry sample holders and cartridge-integrated microscopes tailored to suit the demands of life sciences, the exploration of such phase transitions within quantum materials typically necessitates adjustable temperatures with a base in the liquid helium (LHe) range [3].

LHe solutions for high-resolution imaging in electron microscopes have been developed with base temperatures as low as 1.5 K maintainable over a continuous five-hour timespan [4]. Unfortunately, such solutions, constructed in a cryo-stage setup, which combine superfluid helium alongside LN<sub>2</sub>-cooled shields, cannot be easily adapted to the tight spatial constraints of the much more technically versatile side-entry holders. Until recently, commercially available LHe side-entry holders have been limited by considerable mechanical and thermal instability, as well as short base-temperature holding times due to the limited cryogen storage capacity of the dewar attached to the holder.

We present recent innovations of a lightweight, ultra-low-temperature LHe TEM sample holder, with eight electrical contacts for biasing capabilities. From room temperature, a base temperature of 5.2 K (as measured adjacent to the sample) can be attained within one minute and sustained with a stability of +/- 2.5 mK for days. Here, we demonstrate our recent achievements in the latest LHe cryo-TEM setup.

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# Shining a Light on Catalysis: A New *In Situ* Transmission Electron Microscopy Solution for Probing Photocatalytic Processes

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*In situ* electron microscopy (EM) is a gateway to observe nanomaterials in their native environment. This technique has revealed the fundamental mechanistic pathways that control nanomaterial synthesis, stability and performance optimization. More specifically, gas phase EM (GP-EM) enables dynamic, real-time imaging of catalyst processes under realistic pressures, gas environments and sample temperature [1-2]. *In situ* studies of catalyst materials enable researchers to probe and align a material's structural evolution with real-time changes in its environment and observe fundamental phenomena such as (photo)catalyst deactivation [3], nanoparticle synthesis [5-6] and identify targets within these materials to improve their performance [7-8].

In this talk, we will introduce a unique, new *in situ* capability that combines relevant environmental conditions with photoillumination of the material. Using a precisely aligned optical fiber, light is delivered directly to the sample at the nanoscale. By connecting an *in situ* gas cell system to an external compatible light source, samples can be excited from 350 to 2000 nm in combination with high-pressure environmental conditions inside an electron microscope. The AXON Synchronicity software embeds the calibrated power density and photo illumination parameters into the image metadata during an *in situ* experiment to facilitate the measurements. This design offers users unprecedented flexibility to perform a wide range of light-induced experiments in relevant gaseous environments at the nanoscale opening the door to robust operando studies of photocatalysis processes.

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# METEOR 2.0: ADVANCING INTEGRATED CRYO-FLUORESCENCE IMAGING FOR TARGETED CRYO-FIB MILLING AND HIGH-THROUGHPUT CRYO-ET

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Correlative cryo-focused ion beam (cryo-FIB) milling enables fluorescence-guided lamella preparation, expanding the range of biological material accessible to cryo-electron tomography (cryo-ET). A key bottleneck in this workflow, however, remains the efficient and precise correlation between fluorescently-labeled regions of interest (ROIs) and the milling position, particularly for high-throughput applications [1-3]. We present Delmic's latest generation of integrated cryo-fluorescence microscope (cryo-FM), METEOR 2.0, designed to sustain high throughput lamella milling while enabling precise targeting of the ROI.

METEOR 2.0 has a >8x larger field of view, which accelerates fluorescence mapping and facilitates robust 3D cryo-FM - cryo-FIB correlation through increased fiducial capture. The redesigned optical system achieves a >3x gain in signal intensity, while a high-speed 6-slot filter wheel enables efficient multi-channel sampling. These improvements provide the precision and sensitivity required for high-confidence ROI targeting in both on-grid lamella milling and serial lift-out workflows.

## References:

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# A Platform for Multimodal Ultrafast Electron Microscopy

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We present the Quantum Scanning Electron Microscope (QSEM), a newly developed multimodal ultrafast electron microscope designed to probe material dynamics and quantum optical phenomena with high spatial and temporal resolution. Ultrafast electron microscopy enables femtosecond-scale access to out-of-equilibrium structural and electronic processes at the nanoscale. QSEM employs femtosecond-pulsed linear photoemission from a Schottky field emitter and offers simultaneous transmission, reflection, and secondary-electron detection across 100 eV–30 keV. By bringing femtosecond temporal resolution and fully multimodal detection to the SEM architecture, QSEM establishes a new approach for ultrafast measurements and additionally will enable advanced modes such as energy-filtered imaging, electron spectroscopy, cathodoluminescence and correlated electron–light detection schemes. As an initial demonstration, raster-scanned nanobeam diffraction resolves nanoscale structural heterogeneity in the layered quantum material 1T-TaSe<sub>2</sub>. Together, these capabilities position QSEM as a versatile platform for ultrafast investigations of structural, electronic, and quantum-optical phenomena in advanced materials, as well as quantum-coherent electron–light interactions.