Atomic-Resolution Cryo-STEM Across Continuously Variable Temperatures

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Abstract

Atomic-resolution cryogenic scanning transmission electron microscopy (cryo-STEM) has provided a path to probing the microscopic nature of select low-temperature phases in quantum materials. Expanding cryo-STEM techniques to broadly tunable temperatures will give access to the rich temperature-dependent phase diagrams of these materials. With existing cryo-holders, however, variations in sample temperature significantly disrupt the thermal equilibrium of the system, resulting in large-scale sample drift. The ability to tune the temperature without negative impact on the overall instrument stability is crucial, particularly for high-resolution experiments. Here, we test a new side-entry continuously variable temperature dual-tilt cryo-holder which integrates liquid nitrogen cooling with a 6-pin micro-electromechanical system (MEMS) sample heater to overcome some of these experimental challenges. We measure consistently low drift rates of 0.3–0.4 Å/s and demonstrate atomic-resolution cryo-STEM imaging across a continuously variable temperature range from ∼100 K to well above room temperature. We conduct additional drift stability measurements across several commercial sample stages and discuss implications for further developments of ultra-stable, flexible cryo-stages.

Key words: atomic-resolution, cryo-stages, cryo-STEM, side-entry TEM holder, variable temperature STEM

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Introduction

Following the development of the first electron microscope (Knoll & Ruska, 1932) and the availability of practical commercial instruments in the late 1930s, it was less than two decades before the first experiments to cryogenically cool samples inside a transmission electron microscope (TEM) were reported (Leisegang, 1954; Honjo et al., 1956; Fernández-Morán, 1960). These early demonstrations already recognized the potential of low-temperature electron microscopy to reduce contamination (Leisegang, 1954), to image frozen hydrated biological specimens (Fernández-Morán, 1960), and to study low-temperature crystalline phases and phase transitions (Honjo et al., 1956, Fernández-Morán, 1960). The temperature range was expanded shortly after with the introduction of liquid helium-cooled stages (Venables, 1963; Valdè & Goringe, 1965; Matricardi et al., 1967; Watanabe & Ishikawa, 1967; Colliex & Jouffrey, 1968; Hörl, 1968), which enabled the study of superconductors, solidified gases, and magnetic domains in the 1960s (Blackman et al., 1963; Goringe & Valdrè, 1964; Boersch et al., 1966; Matricardi et al., 1967; Watanabe & Ishikawa, 1967; Colliex & Jouffrey, 1968). Within the materials community, however, the push towards higher resolution over the following decades shifted experimental focus predominantly to more stable (i.e., ambient) working conditions.

As atomic-resolution imaging and spectroscopy of crystalline materials at room temperature has become almost routine, the interest in cryogenic electron microscopy for the physical sciences has been renewed (El Baggari & Kourkoutis, 2019; Minor et al., 2019; Zachman et al., 2020). Recent results have demonstrated the potential for cryogenic scanning transmission electron microscopy (STEM) and electron energy loss spectroscopy to explore quantum materials phenomena including low-temperature spin states (Klie et al., 2007), emergent charge density phases (El Baggari et al., 2018), and interface charge transfer (Zhao et al., 2018). Atomic-resolution low-temperature experiments reported to date, however, have been limited to single temperatures set by the choice of cryogen, i.e., liquid nitrogen or helium. Richer exploration of many novel quantum materials will require stable imaging conditions at continuously variable temperatures to tune into phases with narrow stable temperature windows or to track phase transitions as they occur.

For this purpose, Zandbergen et al. (HennyZ Co.) have developed a continuously variable temperature (CVT) liquid nitrogen specimen holder. The side-entry CVT cryo-holder achieves fine temperature control across the ∼100–1,000 K range by integrating local heating of the sample via 6-pin MEMS control with liquid nitrogen cooling. Previous measurements with a single-tilt version showed single atom imaging at liquid nitrogen temperature (Hotz et al., 2018), but dual-tilt capabilities are critical for crystalline...
materials. Here, we present the implementation and performance characterization of a dual-tilt CVT cryo-holder in an aberration-corrected FEI Titan Themis 60-300. We demonstrate sub-Å-resolution STEM imaging and low instantaneous drift rates of 0.3–0.4 Å/s across the temperature range. In addition to the local MEMS heating, this performance is enabled by active control of the CVT cryo-holder rod temperature which reduces thermal sample drift. We conclude with similar performance analyses of several commercially available room temperature and cryogenic sample stages for comparison and discuss broader trends in this area of instrumentation development.

Background

Side-entry cryo-holders consist of a long hollow metal rod at one end of which the sample is mounted and held in the microscope column. The sample is thermally coupled through the length of the rod to a cryogenic cold sink outside the microscope, usually either a dewar or lines of a cryogen. Compared to the early dedicated cryo-stages, the introduction of commercial side-entry cryo-holders has made cryo-electron microscopy more widely available (Henderson et al., 1991). Atomic-resolution cryo-STEM with such a setup is, however, complicated by (1) thermal contraction of the macroscopic metal rod which can result in dramatic directional drift of the sample and (2) mechanical coupling of the sample to the external environment via the cryogen reserve resulting in vibrations from boiling or bubbling within the cryogen.

Most successful cryo-experiments to date have been performed at cryogen-set temperatures—usually near ~90 K (liquid nitrogen) or ~10 K (liquid or cold gas helium)—in order to preserve a fixed thermal equilibrium. In principle, variable temperature control is possible with some commercial cryo-holders through resistive heating of the sample rod (Tao et al., 2011; Zhao et al., 2016; Berruto et al., 2018) or by regulation of the cryogen flow through the holder or stage (Valdre & Goringe, 1965; Harada et al., 1992; Qiao et al., 2017). In practice, the spatial resolution obtained at intermediate temperatures has so far been limited.

Generally, the most stable imaging conditions are reached after allowing several hours for the temperature throughout the holder, from cryogen to sample, to fully equilibrate. A new equilibrium must be reached whenever the holder is heated to a new temperature, thus requiring additional time to achieve stable conditions after each temperature change. Directional sample drift is also caused by evaporation of the cryogen in the dewar over the course of the experiment. This slow reduction in the cryogen level results in a continuous change to the thermal equilibrium of the system. Attempts to reach intermediate temperatures by heating all or part of the holder rod not only increase the rate at which the cryogen is depleted, but can also lead to bubbling inside the dewar for some target temperatures, further reducing the imaging stability.

Boiling, bubbling, and other disturbances to the cryogen couple directly to the sample through the holder rod (Henderson et al., 1991), giving rise to a number of imaging artifacts such as "tearing" of atomic sites in high-resolution serial recording techniques like high-angle annular dark-field (HAADF) STEM (Klie et al., 2011). By ensuring proper isolation of the cryogen from the environment and eliminating nucleation sites for bubble formation inside the dewar, random disturbances can be mostly avoided at the base temperature of the holder. Limitations due to the remaining thermal drift have been recently overcome by developments in acquisition and processing of fast frame rate STEM data, enabling sub-Å-resolution imaging near liquid nitrogen temperature (El Baggari et al., 2018; Savitzky et al., 2018). The successful expansion of these high-resolution techniques to variable temperature in situ experiments will be a major step for cryo-STEM applications in physics and materials science.

Materials and Methods

CVT Cryo-TEM Holder

The complete HennyZ dual-tilt side-entry CVT cryo-holder system is shown in Figure 1. Continuously variable temperature control is achieved through the integration of 6-pin MEMS heating in a side-entry liquid nitrogen cryo-holder. The MEMS chip (Fig. 1a) enables local heating directly at the sample without disrupting the thermal equilibrium of the holder. After cooling and equilibrating the entire system, activating the MEMS device heats the sample to the desired temperature above the cryogenic baseline, covering a temperature range from ~100 K to the maximum temperature of the MEMS chip.

The MEMS chip is calibrated by assuming a linear relationship between the resistance, R, across the chip in the holder and the temperature, T,

\[ R(T) = bT + c \]  

where b and c are constants. Further improvements in temperature calibration will take into account nonlinearities over the full temperature range. The value b for a given chip is determined before use by linear fit across a controlled temperature sweep, so that c can be calibrated for each experiment in the microscope by measuring R at known T. In all experiments reported here, we perform the final calibration after loading and allowing the holder to settle prior to cooling. Sample temperatures in Figures 2, 3, and 5 are reported as measured by the calibrated MEMS chip. In cases where the slope b used for a given MEMS chip is incorrect, the temperature values read off the chip can be post-corrected by linear interpolation between two known endpoints: for instance, the ambient temperature before cooling the holder and the baseline
Fig. 2. Baseline single-frame HAADF-STEM images of polycrystalline Au nanoparticles on C support in the CVT cryo-holder show sub-Å resolution without image registration, drift correction, or other post-processing. (a) Ambient conditions with no cryogen or MEMS heating. (b) No cryogen with MEMS heating to 373 K. (c) Liquid nitrogen cooled to ~100 K with no MEMS heating. Inset FFT amplitudes show sub-Å information transfer under all conditions. Scans are 2048 × 2048 pixels with dwell times of 2 μs/pixel for total frame times of ~8.5 s, oriented with fast scan direction horizontal.

Fig. 3. Single-frame HAADF-STEM images of polycrystalline Au nanoparticles on C support in the CVT cryo-holder at intermediate temperatures of (a) 123 K, (b) 173 K, and (c) 223 K as measured by the calibrated MEMS chip show sub-Å resolution without image registration, drift correction, or other post-processing. Minimal scan tearing from disturbances in the liquid nitrogen can be observed. Scans are 2048 × 2048 pixels with dwell times of 2 μs/pixel for total frame times of ~8.5 s, oriented with fast scan direction horizontal.

Fig. 4. Registered HAADF-STEM images of crystalline Bi0.35Sr0.18Ca0.47MnO3 in the CVT cryo-holder at calibrated intermediate temperatures of (a) 158 K, (b) 198 K, and (c) 238 K show sub-Å resolution. The dual tilt capabilities of the CVT cryo-holder are necessary to orient the sample along a primary crystalline axis: tilts used here are α = −7.3° and β = −9.7°. Images are registered stacks of 50 frames at 1024 × 1024 pixels with dwell times of 0.25 μs/pixel for total frame times of ~0.34 s.
liquid nitrogen-cooled temperature \( \sim 100 \) K. The temperatures reported in Figure 4 are calculated with this method.

The CVT cryo-holder also has a ceramic Ti meander tube near the end of the sample rod designed to rigidly connect but thermally isolate the cooled holder tip from the outer cylinder of the rod arm. The cylinder can be heated by a separate temperature controller to match the temperature of the microscope goniometer. This helps maintain thermal equilibrium across the system, thereby further reducing macroscopic drift from expansion or contraction of the rod arm.

The full set-up includes a dual-axis sample tilt cable connection, external control (not pictured) of both the rod and MEMS heaters, and a large 14 L liquid nitrogen dewar mounted to the side of the microscope column into which the holder’s copper cooling braids are suspended (Fig. 1b). The large reservoir extends the total imaging time before the cryogen has to be replenished, though the baseline temperature at the sample will slowly increase as the liquid nitrogen level reduces. This change only effectively has an impact on experiments at the lowest possible temperature, as the MEMS sample heating offsets such variations at intermediate points.

One drawback of a large dewar is increased sensitivity to external disturbances (Henderson et al., 1991). Unlike standard side-entry holders, however, the specimen rod is not physically connected to the dewar but only makes contact to the cryogen through the Cu cooling braids. Mounting the dewar directly on the microscope column ensures isolation from other vibrations in the room. The use of fine gauge wire for the cooling braids reduces the strength of mechanical coupling to the liquid nitrogen.

### Sample Drift Measurements

Drift measurements reported in the Results section (see Figs. 5–7) are intended to represent typical day-to-day working conditions for each of the holders discussed. Drift rate values were acquired by measuring shifts via cross-correlation between 1 s image frames taken successively over 250 s after allowing several hours to mechanically stabilize in the microscope. Multiple experiments were conducted for each holder and the mean drift speed calculated for each. The data presented in this report are the individual experiments whose mean drift rates are closest to the average of all such measurements for each holder. Sample position plots show the relative motion of the sample with respect to an arbitrary \((x, y)\) coordinate from the middle image frame of the series. Color scales of drift plots indicate frame order from darkest (first) to lightest (last); arrows near the beginning of each series indicate the general drift direction. Instantaneous velocities at each frame were calculated using the leapfrog method (Savitzky et al., 2018); histograms of their magnitudes accompany each drift plot with solid and dotted vertical lines marking the mean speeds \((\mu)\) and standard deviations \((\sigma)\), respectively.

### Results

The stability of the dual-tilt CVT cryo-holder is assessed in STEM mode on an aberration-corrected FEI Titan Themis. Operating at 300 kV, the microscope was tuned to sub-Å resolution at room temperature using a convergence semi-angle of 21 mrad and HAADF collection angles between 68 and 340 mrad. The CVT cryo-holder was loaded into the microscope and allowed to settle for \( \sim 1.5 \) h at room temperature to minimize the influence of initial drift as the holder settles in the goniometer. Prior to cooling, we performed benchmark tests under ambient conditions (Fig. 2a) and with the MEMS chip turned on to heat the sample (Fig. 2b). We observed no change in performance with activation of the MEMS heater during experiments up to temperatures of 823 K. In line with our measurements, the temperature stability, precision, control, and uniformity in similar MEMS chips are well documented up to the maximum temperatures, usually \( \sim 1073 \) K (800°C) (Allard et al., 2009; Mele et al., 2016; Perez-Garza et al., 2016; van Omme et al., 2018; Gaulandris

![Fig. 5.](image-url) Drift performance of the liquid nitrogen-cooled CVT cryo-holder at intermediate temperatures measured by sample positions plotted over 250 s time periods at (a) 123 K, (b) 173 K, and (c) 223 K. (d–f) Corresponding instantaneous drift speed histograms with mean speed \((\mu)\) and standard deviations \((\sigma)\) marked by solid and dotted vertical lines, respectively.
et al., 2020). Cooled to the cryogenic baseline of $\sim$100 K without MEMS heating (Fig. 2c), imaging performance with the CVT cryo-holder remains nearly unchanged. Small amounts of scan tearing from minor cryogen bubbling is visible in certain regions of the images and as vertical streaking in the fast Fourier transform (FFT) amplitudes. Corresponding inset FFTs show sub-Å information transfer consistently for STEM images at all temperatures. Each scan is $2048 \times 2048$ pixels with a 2 $\mu$s/pixel dwell time for a total frame time of $\sim$8.5 s, with all images oriented such that the fast scan direction is horizontal. Imaging resolution can be further improved through fast frame acquisitions coupled with image registration techniques (Savitzky et al., 2018).

Activating the MEMS chip to access continuously variable intermediate sample temperatures results in no observable degradation of imaging stability. Figure 3 shows single-frame HAADF-STEM scans of polycrystalline gold nanoparticles deposited on a carbon-coated MEMS chip at three intermediate temperatures ($\sim$123, 173, and 223 K). Stability and performance of the holder is unaffected by dual-axis tilts which are necessary to orient crystalline samples for projection down a primary axis. Figure 4 shows HAADF-STEM images of $\text{Bi}_{0.35}\text{Sr}_{0.18}\text{Ca}_{0.47}\text{MnO}_{3}$ at intermediate temperatures, preserving sub-Å information transfer and clear distinction of both the A-sites (Bi, Sr, and Ca) and B-sites (Mn) suitable for quantitative analysis. These results demonstrate that sub-Å-resolution STEM imaging which is today almost standard under ambient conditions can now be achieved at arbitrary cryogenic temperatures down to the base temperature of the CVT cryo-holder.

Local MEMS heating leaves the thermal equilibrium of the holder system effectively unchanged and introduces little to no additional drift when varying the sample temperature. Rapid changes across tens of K require only a few minutes of settle time to reach stable conditions. Figure 5 shows sample drift measurements for the same intermediate temperatures shown in Figure 3. Each measurement was taken within 5 min after a temperature adjustment of 50 K. The average drift rates, which range between 0.3 and 0.4 Å/s, are only slightly above the cryogenic baseline drift for this holder of 0.2 Å/s without MEMS sample heating (see Figs. 6b, 6d).

The low drift rates observed across all measured temperatures are enabled in part by the active rod heating and the Ti meander described in Figure 1. Active compensation through the rod heater helps maintain a constant temperature equilibrium of the holder inside the microscope goniometer, reducing the characteristic directional drift by more than half. Drift measurements in Figure 6 demonstrate the effect of active heating of the rod to 17.6°C, near the measured temperature of the goniometer. The rod heating reduces the average sample drift speed by $\sim$60%, from 0.48 Å/s without rod heating to 0.20 Å/s with heating. As a point of comparison, the average sample drift rate of a standard side-entry dual-tilt cryo-holder (Gatan Model 636) in our Titan Themis was measured to be 0.75 Å/s at its base temperature near liquid nitrogen (see Fig. 7a).

The slow depletion of cryogen over the course of a long experiment results in a “moving target” equilibrium point as the submersion of the cooling braids slowly decreases. Although a large dewar slows these effects, another way to further compensate this variation is to maintain a constant cryogen level in the dewar so that the cooling braids remain submerged by the same amount over the course of an entire experiment. By mounting an optical monitor inside the nitrogen dewar, it is possible to monitor the level of the cryogen as it boils off and compensate for this depletion by slowly lowering an insulating volume into it, displacing the remaining liquid nitrogen to a constant level. One such system has been tested on a similar CVT cryo-holder in a Philips CM200 with promising results, but no such technique has yet been implemented on the Titan Themis in the experiments described here.
Fig. 7. Drift performance comparison of commercial room temperature and cryogenic side-entry holders and dedicated stages. Sample drift plots and corresponding instantaneous drift speed histograms for several commercial sample stages: (a) side-entry dual-tilt Gatan 636 liquid nitrogen cryo-holder in a Titan Themis; (b) side-entry dual-tilt FEI room temperature holder in a Titan Themis; (c) dedicated liquid nitrogen-cooled cryo-stage in a Thermo Fisher Talos Arctica; (d) room temperature Nion UltraSTEM in-column cartridge. 10× magnifications are included for the (c) Arctica and (d) UltraSTEM.
Discussion

Advances in hardware for cryogenic electron microscopy over the past decades have been primarily driven by needs in the life sciences. This has led to the development of modern dedicated liquid nitrogen-cooled stages which support rapid sample exchange, more automated data acquisition, and increased sample throughput (Williams et al., 2019). These dedicated cryo-stages, however, lack the dual-axis tilt control which is often indispensable for the physical sciences and currently only available in side-entry cryo-holders. Side-entry holders also offer additional flexibility to more easily incorporate sample manipulation such as electrical biasing or MEMS heating, as in the CTV cryo-holder discussed here. Careful design ensures high stability in both drift and vibrations to enable sub-Å cryo-STEM imaging not only near liquid nitrogen but also continuously across a wide temperature range (Figs. 3, 5).

While neither continuous temperature control nor dual-axis tilting is currently possible in dedicated cryo-stages, their superior drift performances compared to side-entry designs are quite striking. Figures 7a and 7c show drift measurements from a standard side-entry dual-tilt Gatan 636 cryo-holder in the Titan Themis and a Thermo Talos Arctica with a dedicated cryo-stage, both near liquid nitrogen temperature. The dedicated cryo-stage offers a nearly 10-fold decrease in sample motion compared to the 0.75 Å/s drift of the side-entry cryo-holder. For ambient temperature experiments, the difference in stability between in-column stages and side-entry holders is less severe but still clear. The dual-tilt cartridge of a Nion UltraSTEM in-column stage exhibits very low drift rates of <0.1 Å/s, less than half the 0.2 Å/s drift of a standard FEI dual-tilt side-entry holder in a Titan Themis (Figs. 7d, 7b).

Further improvements to the stability of dual-tilt cryo-holders or the development of stable cryo-stages with either dual-tilt or tilt-rotate capabilities will be particularly important for slow-scan techniques such as 4D-STEM and spectroscopic mapping. Some experimental flexibility including sample heating has already been demonstrated with room temperature in-column stages (Hudak et al., 2017; Sang et al., 2018), but has as yet not been realized in dedicated cryo-stages. Future developments for high-performance specimen holders and stages will ideally seek to combine the benefits of both designs, marrying the ultimate stability of in-column stage designs with the flexibility and variable controls of new in situ side-entry holders.

Conclusions

To date, even successful high-resolution cryo-STEM experiments still make important sacrifices when compared to standard room temperature conditions. Side-entry cryo-holders like those used in (Klie et al., 2007; El Baghari et al., 2018; Zhao et al., 2018) suffer from instabilities caused by thermal contraction and cryogen boiling, precluding the effective use of longer acquisition techniques such as 4D-STEM or spectroscopic mapping. On the other hand, dedicated cryo-stages boast improved stability but at the expense of the dual-axis sample tilt critical for crystalline materials.

The HennyZ dual-tilt cryo-holder system described here demonstrates both drift and internal vibration stability which enables sub-Å STEM imaging across continuously variable temperatures from ~100 to 1000 K. The combination of MEMS sample heating for intermediate temperature access, rod heating to help mitigate thermal drift, and dual-tilt capabilities offers increased experimental flexibility. These proof-of-concept experiments mark significant progress for the accessibility of variable temperature cryo-electron microscopy, opening the doors to a new range of high-resolution in situ cryo-experiments including the real-time observation of phase transitions, temperature cycling, and access to phases which are stable only in narrow temperature windows.

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References

Allard LF, Bigelow WC, Jose-Yacaman M, Nackashi DP, Damiano J & Mick SE (2009). A new MEMS-based system for ultra-high-resolution imaging at elevated temperatures. Microsc Res Technol 72, 208–215.

Berruto G, Madan I, Murooka Y, Vanacore G, Pomarico E, Rajszewski J, Lamb R, Huang P, Kruchkov A, Togawa Y, LaGrange T, McGrouther D, Ronnow H & Carbone F (2018). Laser-induced skyrmion writing and erasing in an ultrafast cryo-lorentz transmission electron microscope. Phys Rev Lett 120, 117201.

Blackman M, Curzon AE & Pawlowicz AT (1963). Use of an electron beam for detecting superconducting domains of lead in its intermediate state. Nature 4902, 157.

Boersch H, Bostanjoglo O, Lischke B, Niedrig H & Schmidt L (1966) Electron microscopy with liquid helium cooled stages. In 6th International Congress for Electron Microscopy, Kyoto, Japan, Aug. 28 – Sept. 4, 1966, Maruzen, Tokyo, pp. 167–168.

Colliex C & Jouvrey B (1968). Un nouveau porte-objet inclinable refroidi à l'hélium liquide. J Microsc 7, 601–608.

El Baghari I & Kourkoutis LF (2019). Developments in electron microscopy of exotic states at oxide interfaces: Cryogenic imaging and advanced detectors. Appl Surf Sci 482, 27–30.

El Baghari I, Savitzky BH, Admasu AS, Kim J, Cheong SW, Hovden R & Kourkoutis LF (2018). Nature and evolution of incommensurate charge order in manganites visualized with cryogenic scanning transmission electron microscopy. Proc Natl Acad Sci USA 115, 1445–1450.

Fernández-Morán H (1960). Low-temperature preparation techniques for electron microscopy of biological specimens based on rapid freezing with liquid helium II. Ann N Y Acad Sci 85, 689–713.

Gaulandris F, Simonsen SB, Wagner JB, Molhave K, Muto S & Kuhn LT (2020). Methods for calibration of specimen temperature during in situ transmission electron microscopy experiments. Microsc Microanal 26, 3–17.

Goringe MJ & Valdrè U (1964). Observation of solid neon by transmission electron microscopy. Philos Mag 9, 897–900.

Harada K, Matsuda T, Bonevich J, Igarashi M, Kondo S, Pozzi G, Kawabe U & Tonomura A (1992). Real-time observation of vortex lattices in a superconductor by electron microscopy. Nature 360, 51–53.

Henderson R, Raeburn C & Vigors G (1991). A side-entry cold holder for cryo-electron microscopy. Ultramicroscopy 35, 45–53.

Honjo G, Kitamura N, Shimaoka K & Mihama K (1956). Low temperature specimen method for electron diffraction and electron microscopy. J Phys Soc Japan 11, 527–536.

Höfl EM (1968). Liquid helium cooled stage with a transverse magnetic field. Rev Sci Instrum 39, 1027–1028.
