Contamination of TEM Holders Quantified and Mitigated With the Open-Hardware, High-Vacuum Bakeout System

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Abstract

Hydrocarbon contamination plagues high-resolution and analytical electron microscopy by depositing carbonaceous layers onto surfaces during electron irradiation, which can render carefully prepared specimens useless. Increased specimen thickness degrades resolution with beam broadening alongside loss of contrast. The large inelastic cross-section of carbon hampers atomic species detection. Oxygen and water molecules pose problems of lattice damage by chemically etching the specimen during imaging. These constraints on high-resolution and spectroscopic imaging demand clean, high-vacuum microscopes with dry pumps. Here, we present an open-hardware design of a high-vacuum manifold for transmission electron microscopy (TEM) holders to mitigate hydrocarbon and residual species exposure. We quantitatively show that TEM holders are inherently dirty and introduce a range of unwanted chemical species. Overnight storage in our manifold reduces contaminants by one to two orders of magnitude and promotes two to four times faster vacuum recovery. A built-in bakeout system further reduces contaminants partial pressure to below 10−10hPa (Torr) (approximately four orders of magnitude down from ambient storage) and alleviates monolayer adsorption during a typical TEM experiment. We determine that bakeout of TEM holder with specimen held therein is the optimal cleaning method. Our high-vacuum manifold design is published with open-source blueprints, parts, and cost list.

Key words: contamination, open hardware, TEM, transmission electron microscopy

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Introduction

Hydrocarbon contamination degrades resolution and hampers accurate spectroscopic analysis. The formation of insulating carbon films on specimens under electron irradiation has been reported early on and is attributed to the polymerization of organic vapors in a vacuum environment by electric discharge (Stewart, 1934; Love et al., 1981). Hydrocarbon deposition typically results in increased particle size or film thickness accompanied by a loss of contrast (Soong et al., 2012). Increased specimen thickness causes beam broadening which degrades resolution (Watson, 1947; de Jonge et al., 2019). In electron energy loss spectroscopy, large plasmonic excitations that scale with hydrocarbon thickness (Nerl et al., 2017) combined with scattering from a large carbon K-edge cross-section become so intense that they obscure core-loss signal from many elements of interest (Fraser, 1978; Griffiths & Walther, 2010; Egerton, 2011).

Besides notorious hydrocarbons, oxygen and water molecules also pose problems of beam damage and ice contamination in electron microscopy. Oxygen and water molecules absorbed onto specimen surfaces create highly reactive radicals when irradiated with electrons. These radicals can cause lattice damage by chemically etching the specimen—a process sometimes confused with knock-on damage (Leuthner et al., 2019). In cryo-TEM, water surrounding the sample is required to be in a vitreous state. Otherwise, water molecules can form crystalline ice that compromises the structural integrity of a specimen. The formation of crystalline ice also degrades image quality as they diffract electrons (Thompson et al., 2016). Hence, it is crucial that the presence of oxygen and water molecules be minimized during the sample preparation and storage phase.

These constraints on high-resolution and spectroscopic chemical imaging demand clean, high-vacuum microscopes with dry pumps. However, even the cleanest microscope columns suffer from impurities desorbed off specimen holders or the specimen itself (Bance et al., 1978)–especially problematic in experiments imparting high dose because hydrocarbon deposition scales with beam spot size and current density (Conru & Laberge, 1975). For aberration-corrected scanning transmission electron microscopy, the high-current density of electrons exacerbates organic polymerization onto specimens. The appearance of contamination can be insidiously delayed, as desorption of species is not immediate and hydrocarbon contamination is driven by surface diffusion of molecules across the holder and the specimen (Hettler et al., 2017).

Here, we present an open-hardware design of a high-vacuum manifold that stores multiple TEM holders to remedy
hydrocarbon and residual species exposure. To confirm the effectiveness of high-vacuum storage, we quantify the molecular species adsorbed onto TEM holder surfaces under various storage conditions using a residual gas analyzer (RGA) as part of our design. Partial pressure measurements by the RGA detect and infer chemical species from their mass-to-charge ratio (Stanford Research Systems, Inc., 2009). Users can directly assess the composition and cleanliness of holders or specimens. Initial RGA measurements across seven different TEM holders demonstrate that most are inherently dirty and ambient overnight storage will introduce a range of unwanted chemical species into the microscope.

Using overnight storage and bakeout inside our high-vacuum manifold, contaminant partial pressures are reduced by approximately four orders of magnitude to $10^{-10}$–$10^{-12}$ hPa (Torr) (below the RGA detection limit). Overnight high-vacuum storage reduces residual gas levels across the whole spectrum by one to two orders of magnitude. Bakeout further reduces organic and light species by another two to three orders of magnitude. Heavier species above 35 amu including pump oil are mostly below the detectable limit ($<10^{-10}$ hPa (Torr)).

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Results

Contaminants Present on TEM Holders

The RGA spectrum for a typical TEM holder (Fig. 1a) highlights the range of species adsorbed onto holder surfaces, spanning organics of various carbon compositions, viscous pump oil, water, and oxygen, which totals a manifold pressure of $10^{-4}$–$10^{-5}$ hPa (Torr). Initial RGA measurements taken across six other TEM holders regularly used and stored under ambient conditions with partial pressure lowered after overnight storage in the manifold.

Vacuum systems using oil-based rotary and diffusion pumps are often considered “dirty” systems because they produce
backstreaming of oil (Postek, 1996). The oil vapor can polymerize under the electron beam, resulting in the deposition of amorphous carbon on the area of investigation (Ennos, 1953). Oil-free systems, such as scroll, turbomolecular, and ion pumps, should replace oil-based pumps for evacuating S/TEM columns (Mitchell, 2015). In addition to improvements in pumps, contamination within the column can be mitigated with a cold trap (Ennos, 1953), also known as an anticontamination device (ACD). The ACD is a liquid nitrogen-cooled trap that condenses vapors near the specimen to minimize their redeposition (Yoshimura et al., 1983). It should be mentioned that any other cold surface in the system will act the same way (e.g., x-ray detectors) (Reimer & Wächter, 1978).

Side-entry TEM holders also use long-term, high-vacuum greases based on perfluorinated polyether (PFPE) oils and polytetrafluoroethylene thickeners that outgas and contaminate specimens. X-ray photoelectron spectroscopy (XPS) analysis of a TEM specimen grid that has gone through 10 pumping cycles in a modern TEM equipped with ACD shows emerging fluorine peaks compared with a clean grid that was never loaded into a TEM (Supplementary Fig. S1). However, XPS does not show a significant increase in carbon signals. Vacuum grease should be used in minimal quantities. Vacuum grease may also accumulate in the loadlock or goniometer of electron microscopes and should be cleaned semi-regularly. For TEM o-rings, micronized PFPE grease (e.g., Y VAC, Braycote Micronic 1613, Krytox) is preferred. Avoid UHV hydrocarbon-based grease (e.g., Rheolube) that releases hydrocarbon byproducts, pure Fomblin with viscosity issues, or clear silicon-based high-vacuum grease (e.g., Dow Corning) with relatively high vapor pressure ($10^{-4}$ hPa (Torr)). As aforementioned, diffusion pump oil is a source of contamination and must not be used on o-rings.

**Clean Holders With High-Vacuum Storage and Bakeout**

Our high-vacuum manifold effectively reduces contaminant partial pressures by approximately four orders of magnitude to even below $10^{-10}$ hPa (Torr) when used for overnight storage and bakeout (Fig. 1a). With overnight high-vacuum storage alone, residual gas levels across the whole spectrum reduce by one to two orders of magnitude ($\sim 10^{-7}$ hPa (Torr)). A built-in bakeout system is substantially effective at removing problematic pump oils and reducing atmospheric species (i.e., CO and H$_2$O) by an additional two to three orders of magnitude down from high-vacuum storage and approximately four orders lower compared with storage in ambient air. The adsorption coverage described by the Langmuir isotherm is a worst-case scenario (sticking coefficient = 1) that will form one monolayer per second at pressures of $10^{-6}$ hPa (Torr) (Jousten, 1999); reducing partial pressures of contaminants below $10^{-10}$ hPa (Torr) (the RGA detection limit) slows the monolayer adsorption time to ~5 h. We consider partial pressures below $10^{-15}$ hPa (Torr) to be negligible.

By facilitating organic desorption through bakeout, the presence of light species is reduced by several orders of magnitude, while pump oil and most of the heavier species above 35 amu were below the RGA’s detectable limit ($<10^{-10}$ hPa (Torr)) (Fig. 1a). Gas molecules on a surface can be described as a distribution of binding energies. Molecules across all binding energy states are more likely to desorb with an increasing bakeout temperature. Even with vacuum bakeout limits (150–250°C) well below the average binding energies (0.73–1.08 eV/molecule), molecules in low binding energy states can slowly desorb over long pumping time (Matthewson & Gröbner, 1999) and minimize migration driven by the surface diffusion of organic molecules that do not immediately desorb in vacuum (Dayton, 1961; Hettler et al., 2017). Following Boltzmann statistics, baking at 130°C increases the desorption rate by 35% over room temperature. After baking, the manifold achieves its lowest total pressure of $10^{-7}$ hPa (∼7.5 × $10^{-8}$ Torr) even when a holder is stored.

We determine that thermal bakeout in vacuum exhibits higher performance over chemical and plasma cleaning. Chemical methods usually leave organic residues, while plasma cleaning may damage carbon-containing specimens and only removes thin layers of surface hydrocarbons. Cleaning the holder with solvents (i.e., acetone and/or methanol) can remove the majority of organic contamination, but commonly leaves organic residue (McGilvery et al., 2012). Fortunately, overnight manifold storage will remove organic residue introduced from a chemical clean. Supplementary Figure S2 shows that chemical cleaning with alcohols introduces organic peaks in residual gas levels resembling that of acetone. These alcohols will desorb after 6 h storage in high vacuum. Plasma cleaning can immobilize and remove thin layers of surface hydrocarbons, which is sufficient for superficial contamination. The holder in Supplementary Figure S2 was subsequently plasma cleaned for 10 min on a 40 W-rated plasma cleaner operating at the standard RF plasma frequency (13.56 MHz) with a mixture of oxygen (25%) and argon (75%). The holder is immediately transferred to the manifold stationed on the same lab bench. RGA data show that the carbon level remains relatively unchanged and that only peaks ~18 amu increase after plasma cleaning. This could be moisture adsorbed onto the surface during the transfer from the plasma cleaner to manifold.

The oxygen plasma chemically reacts with hydrocarbons and converts it into CO, CO$_2$, and H$_2$O, which are subsequently evacuated by the vacuum system (Isabell et al., 1999). One major limitation with plasma cleaning is its potential to damage any carbon support films or carbon-containing specimens that may reside on the holder. Our design can address this issue by removing contaminants while preserving any samples capable of withstanding moderate heating.

Bakeout of TEM holders and the specimens held therein are carried out inside the manifold at 130°C for 48 h as organic molecules desorb at this temperature (Jousten, 1998; Grinham & Chew, 2017) without degrading o-rings (Viton ~225°C, Buna ~120°C) and internal wiring components of a TEM holder. Calibration of bakeout temperature is based on thermal readings with a Gatan #652 heating holder made of beryllium copper and tantalum. Bakeout is also tested on JEOL single- and double-tilt holders. In all these cases, vacuum heating is equally capped at 130°C and reached $10^{-7}$ hPa (Torr) total pressure. While (austenitic) stainless steel and aluminum alloys make up many vacuum components, certain grades (i.e., aluminum alloy 7000 series, stainless steel free-machining grades) and some metals, such as brass, cadmium, zinc, tellurium, and Pb-based solder flux, are not vacuum compatible in the first place (Coyne, 2013). In situ gas or liquid cell holders may contain polymer tubing (e.g., PEEK) that cannot be baked above 100°C. We opted for Viton o-rings due to its high thermal stability and low outgassing and permeability. Small outgassing and permeation rates are essential to reach low base pressures. Lubrication of o-rings in minimal quantities (one drip for the entire o-ring) is necessary to protect it from abrasion and degradation by atmosphere. We use Klüberalfa Y VAC O-ring grease due to its high thermal stability.
and low vapor pressure. We recommend annual replacement of o-rings and vacuum grease or when o-rings have experienced frequent heat cycling.

High-Vacuum Storage for Faster Pump down

A TEM holder exposed to ambient air for 10 min (roughly the time to load a specimen) after high-vacuum storage achieves partial pressure recovery two to four times faster than that of a holder stored in ambient air. Figure 2 compares the manifold total pressure of TEM holders exposed to ambient air for over 1 day—as found in a typical TEM facility—and those stored in high vacuum with only 10 min of ambient exposure. These pressures are measured at the top of the manifold vacuum column. We define full recovery to be the minimum total pressure a manifold with a stored TEM holder can achieve that remains constant thereafter for at least 1 day. Partial recovery is defined to be the 4.0 × 10⁻⁷ hPa (Torr) mark in Figure 2 and lower pressures. In our comparison, full recovery takes 6 h versus 3.5 h, while partial recovery takes 2 h versus 30 min. Thus, partial recovery is four times faster and full recovery reaches nearly two times lower pressure in half the time.

Given that mounting a sample takes around 10 min, these results suggest that users can achieve faster pressure recovery within the TEM column and have more efficient microscopy sessions with high-vacuum storage. A typical TEM column requires evacuation down to 10⁻⁷ hPa (Torr) by turbomolecular (dry) or diffusion (oil) pumps operating in the high-vacuum range. When a TEM holder is clean, the manifold can achieve a total pressure as low as 10⁻⁷ hPa (~7.5 × 10⁻⁹ Torr), which is the usual limit of o-ring-sealed systems. The improved vacuum and pumping speeds reflect the overall reduction of adsorbed species for TEM specimen holders stored under high vacuum.

Materials and Methods

Open-Hardware Design of a TEM Holder Manifold

Our design consists of a 2-tier structure that can store up to ten TEM holders. The number of tiers and ports can be easily customized to suit each facility’s needs. In our case, we opted for a 2-tier structure to store both our FEI and JEOL holders. The whole manifold is assembled on top of a Pfeiffer Vacuum HiCube 80 Eco turbopump station supported by an 80/20 extruded aluminum frame (Fig. 3a). The Pfeiffer pumping station includes a control unit for the turbopump and a diaphragm backing pump. Pump down and ventilation are controlled using the display unit, from which turbo speed, frequency, and total pressure of the system can be inspected. Pressure and mass measurements are collected with a Pfeiffer Vacuum Pirani/cold cathode (PKR) pressure gauge and an SRS RGA (Fig. 3c).

The RGA sorts and detects the ion current of residual gases based on their mass-to-charge ratio. We have opted for the 100 amu model, which allows for the identification of the common contaminants found in electron microscopy applications (gas species ranging from 1 to 100 amu). The RGA is installed on the manifold backside for structural stability and to provide sufficient space for the quadrupole mass probe. The RGA also includes an electronic control unit, and cable wires for computer connection while avoiding interruption during holder insertion and removal. Mass spectra using the RGA are acquired from an adjacent computer.

Each tier contains an array of butterfly valves that opens each holder to vacuum. The pressure measurements in Figure 2 were taken with several ports solely sealed by butterfly valves, indicating that these valves are sufficient at isolating the vacuum against atmosphere at or below that of the TEM o-ring seal limit (~10⁻⁷ hPa (Torr)). Scientists may also substitute more expensive gate valves that can handle higher pressure differentials, or all metal angle valves that can tolerate higher temperature bakeouts. However, total pressures lower than 10⁻⁷ hPa (Torr) are not easily achieved in an o-ring-sealed vacuum system. Due to the unique diameters, the pipes were custom made by MDC Vacuum Products. The designs for custom pipes to fit a JEOL TEM holder are shown in Supplementary Figure S3. The bakeout system consists of a quartz lamp to an electrical feedthrough installed in the mini-side port of storage flanges (Fig. 5). This allows for radiative heating of the TEM holder tip and specimen (if mounted) up to ~150°C while in vacuum and prevents the polymer o-rings from overheating as shown in Figure 5a. Bakeout temperature is varied with the applied electric potential. To achieve higher vacuum, we opted for ConFlat (CF) flanges over ISO-KF/ISO-LF.

Operation of the manifold is straightforward and requires only a few minutes of training. Holder exchange requires shutting off the turbopump and complete ventilation of the chamber to avoid damaging the turbo blades. The turbo will audibly wind down within 5 min as the pumping station self-vents. The display panel can be used to inspect turbopump speeds to ensure the blades are not rotating before exchanging the holder. The butterfly valve of the holder flange in exchange needs to be shut off to keep the rest of chamber in low vacuum (10⁻⁴~10⁻³ hPa (Torr)). Some resistance may be felt when pulling the holder out due to higher pressure outside the manifold. A more cavalier approach of closing the butterfly valve and removing the holder against vacuum can be done without winding down the turbo. Before turning the turbopump back on, the butterfly valves for all unused ports must be closed. Dummy holders may be optionally inserted into unused ports for additional safety. Dummy holders are easily machined from the included plans (Supplementary Fig. S4).

Parts and Assembly

Assembly is completed in multiple stages starting with the first tier. It is recommended to work with nitrile gloves, maintain...
clean working surfaces, and wrap components with aluminum foil to minimize atmospheric exposure. Each tier consists of a six-way cross connected to four tees (Fig. 4a). Extra or unused flanges should be blanked. The butterfly valve and custom pipe are tightened to the tees by 2″ hex bolts. For uniform orientation, the hand wheels on the valves should all be pointing right (Fig. 4b). After assembling the tier, it will be attached onto the pumping station cross. A conical reducer is placed between the 4.5″ turbopump flange and the 2.75″ six-way cross flange (Fig. 3d). The PKR gauge and RGA are installed on the top and back of the six-way cross, respectively (Fig. 3a). The ionizer cage side of RGA should go inside six-way cross, while the probes point behind and align with the holes on the electronic control unit until complete contact is achieved. When connecting vacuum components, a fresh copper gasket is first placed against the knife edge seal of a CF flange. At the mating flange, bolts are hand tightened with small increments in a crisscross star sequence to prevent overtightening on one side and a bad seal.

The Pfeiffer HiCube 80 model has a maximum rotation speed of 90,000 rpm and a frequency of 1,500 Hz. When these parameters cannot reach the maximum values, a leak may be occurring at the vacuum connections and the system should be partially disassembled and pumped down to search for leaks. If a CF flange is reopened, any used copper gasket must be replaced. Once the chamber is fully assembled and tested for leaks, the system is left to pump down overnight to reach an initial base pressure. To achieve lower pressure and remove any remaining adsorbed molecules, the manifold is baked overnight at 130°C with a BriskHeat silicone rubber heating tape. Note that the heating tape can damage the butterfly valves if wound too closely around them. An initial pump down is critical when installing the RGA as it cannot operate at pressures above $10^{-4}$ hPa (Torr). The RGA electronic control unit must be removed prior to this process, but bakeout of the RGA mass probe is still recommended.

Setting up the bakeout chambers requires the following steps. A bi-pin-type quartz lamp and power supply wiring are soldered.
to the Type-C 9 pins subminiature electrical feedthrough. Lead and halogen-free solder are used to avoid outgassing in vacuum. The soldered quartz lamp is cleaned with a flux remover and organic solvents (e.g., acetone and methanol) in an ultrasonicator. The lamp is fitted to two pins on the vacuum side of electrical feedthrough and assembled on the mini port with the lamp body inside manifold (Fig. 5c). To begin bakeout, power supply wiring is connected to the electrical feedthrough (Fig. 5a). Our calibration using a Gatan heating holder determined that bakeout temperatures of $\sim 130^\circ$C can be achieved with 39–40 V at a constant current of 0.22A from a 120 V, 50 W-rated quartz lamp.

**Conclusion**

Hydrocarbon contamination has been an ongoing challenge for microscopists as it plagues image resolution, contrast, and chemical sensitivity. Here, we quantitatively show that TEM specimen holders are a notable source for hydrocarbon contamination and water vapor during an electron microscope experiment. To significantly mitigate this problem, we constructed a high-vacuum TEM holder manifold with bakeout that provides roughly a four-order magnitude reduction in water vapor and common organic species. Our TEM holder manifold design is published as an open-hardware project with a parts list (Table 1) and design plans (Supplementary material). In the future, we hope to see the development of higher vacuum inside the microscope column and routine implementation of dry systems such as turbomolecular or ion pumps.

**License**

The design here is released under the CERN-OHL-W version 2 license (https://kt.cern/ohlv2) to promote collaboration among hardware designers and to provide a legal tool which supports the freedom to use, study, modify, share, and distribute hardware designs and products based on those designs.

**Supplementary material.** To view supplementary material for this article, please visit https://doi.org/10.1017/S1431927620001762
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