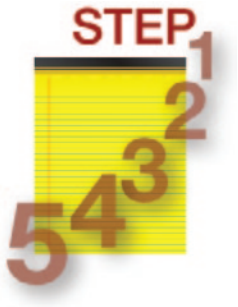


A "Clean" TEM

Graham J.C. Carpenter
Materials Technology Laboratories, NRCan, 568 Booth Street, Ottawa, Canada, K1A 0E3

Graham.Carpenter@NRCan-RNCan.gc.ca

"All microscopes cause contamination of the specimen under the electron beam," said our instructor at the EELS school I was attending. "Ours doesn't!" I responded. This only caused him to repeat his assertion.

Perhaps we were both right in our own ways. EELS has a fairly high sensitivity for detecting C, which is often deposited on the specimen surfaces during examination, particularly when a finely focused probe is used. At the time, I was thinking more of the dark blobs (often like "fir trees" in severe cases!) that are visible when contamination under the electron beam is fairly pronounced. In our laboratory, however, I have been fortunate to work for over 25 years on two microscopes—one of which had a field-emission Schottky source—which could be described as causing negligible contamination under all beam conditions, as long as the cold finger was in operation.

It has been stated many times that contamination is mostly caused by the specimen rather than the TEM. Electropolished thin foils, for example, may retain an invisible organic surface film that will decompose under the beam, causing carbon build-up. Biological samples are usually prepared by embedding procedures involving polymerized plastic that readily breaks down to form carbon.

Is it true that most problems are associated with the specimen rather than the machine? Any source of gaseous hydrocarbons in the region of the specimen can cause carbon deposition under the electron beam. Back-streamed oil from a diffusion pump or grease (including human fingerprints) on the holder near the specimen can cause carbon deposition. Carbon contamination was the bane of electron microscopy in microscopes until about the 1970s. The necessity of the liquid nitrogen cold "finger" near the specimen is testament to the difficulty of eliminating hydrocarbons from the sample chamber of all microscopes, apart from a dedicated STEM that employed ultra-high vacuum technology.

In fact it is easy to assess the state of a microscope using a specimen that is known to be free from the surface hydrocarbons that lead to contamination. Following experimentation in our laboratory using an ultramicrotome to prepare thin TEM samples from metals by Tom Malis, we found that one of the easiest ways to produce a metal test sample that is free from contaminants is to use a clean ultramicrotome. This technique works best with soft metals, so Al is a good candidate for making test specimens. It is simply necessary to ensure that everything connected with cutting and handling a thin specimen is free from grease, including any remaining from prior preparation of embedded samples or from handling.

The following precautions are suggested for ensuring the microtomed sections are as clean as possible:

- Clean the tweezers, specimen, Cu grids, diamond knife and boat with good purity acetone.
- Use gloves and tweezers to handle samples.
- Cut a few sections first onto distilled water and reject them.
- Collect subsequent sections, 30–50 nm in thickness on the clean grids.
- Place each in a small polyethylene embedding capsule, with a small hole, for storage in a vacuum desiccator.
- Never store specimens in gelatine capsules.

In order to check whether your microscope is clean enough for most practical purposes, it is then merely necessary to introduce a sample into the microscope and leave it under a focused beam for 5–10 minutes. Of course, unless the microscope operates in ultra-high vacuum, as in the case of a dedicated cold field-emission STEM, it is essential to thoroughly cool down the liquid-nitrogen cold trap before the test is carried out.

For someone in the market for a new TEM, if freedom from contamination under a finely focused beam is important, it is a good idea to check the contamination behavior of the microscopes under consideration. We did this and have been most happy with the resulting performance of our machines.

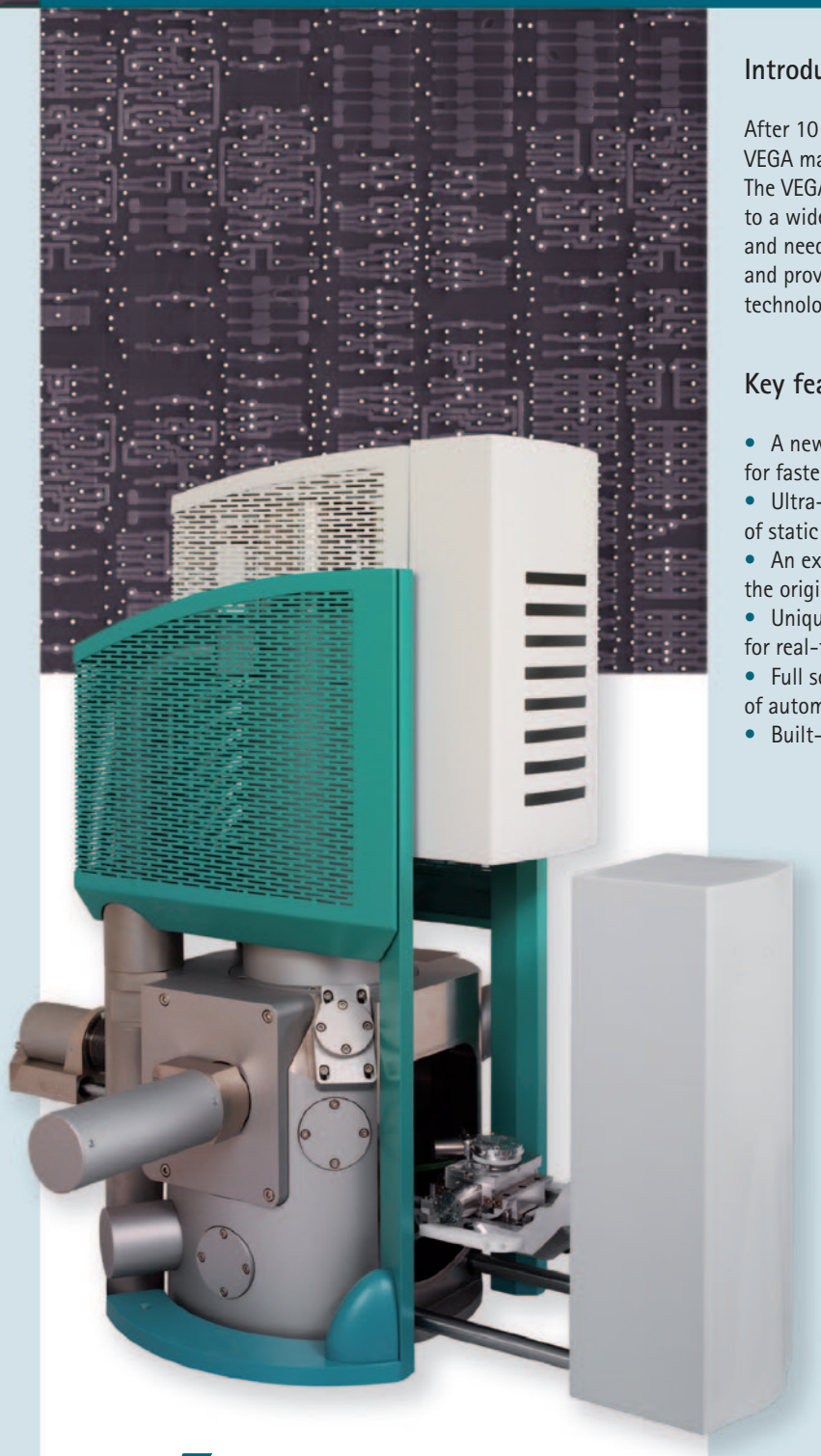
A question that can also arise is whether the quality of the microscope vacuum may degrade during use over a period of years, leading to contamination problems. My experience has been that with careful maintenance on a day-to-day basis, a clean vacuum may be retained for a considerable time. Certainly, with side-entry specimen holders it is nice to have a plasma cleaner to keep the high-vacuum end of the rod shiny and clean. However, for many years these were not available and we found that careful handling (particularly avoiding touching the tip of the holder with the fingers) and storage in a desiccator were effective in keeping the holders clean. Even when an oxide film had built up on the surfaces of some copper holders over a period of years, this had no serious effect on the contamination rate.

Equally important, with a holder that has a sliding O-ring seal, we have adopted the habit of checking the O-ring when a change of specimen occurs. For anyone who doubts this may be worthwhile, I suggest checking the O-ring under a low power binocular microscope. In most cases the result is quite a shock, revealing fine fibers that arise from clothes, paper, etc. (Figure 1), as well as occasional small pieces of metal that come from friction during insertion of the holder into the

 doi: 10.1017/S1551929510000477

Scanning Electron Microscopes

VEGA 3



Introducing New Generation of SEMs

After 10 years of continuous development, VEGA matured into its 3rd generation. The VEGA 3 series were designed with respect to a wide range of SEM applications and needs in today's research and industry and provides users an advantage of the latest technology.

Key features of the VEGA 3 Generation:

- A new high-performance electronics for faster image acquisition and signal processing
- Ultra-fast scanning system with compensation of static and dynamic image aberrations
- An extended range of scanning modes using the original Wide Field Optics™
- Unique Tescan In-Flight Beam Tracing™ for real-time beam current and spot size optimization
- Full software control and high level of automation without any mechanical adjustments
- Built-in scripting for user defined applications

TESCAN

PERFORMANCE IN NANOSPACE

Tescan USA
508 Thomson Park Drive
Cranberry Twp., PA 16066
Phone: 724-772-7433
Email: info@tescan-usa.com

www.tescan.com

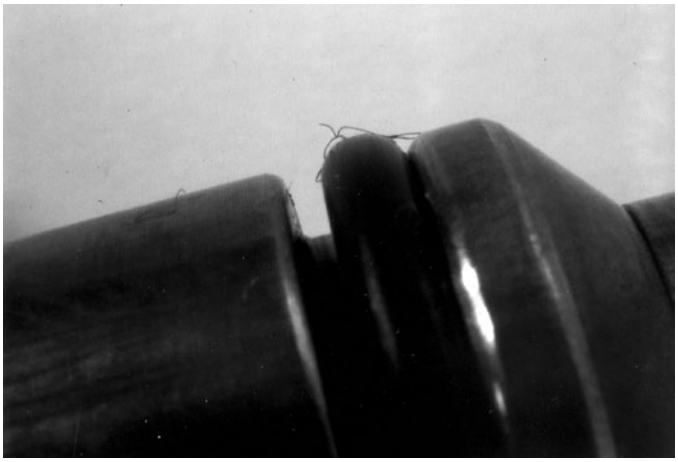


Figure 1: Fibers visible on the O-ring and shaft of a TEM side-entry specimen holder prior to cleaning.

microscope. Over time, many of these contaminants will end up trapped in the microscope at the entrance to the airlock where they will remain and can degrade the vacuum until the goniometer is stripped for cleaning (something no service engineer is very keen to do, and it is not particularly desirable as long as the goniometer works well!).

There is a particularly simple method for cleaning off these fibers and particles from the O-ring using paper masking tape from a hardware store. A small piece of tape is simply folded over with the sticky side outwards, held in a tweezers, and used to remove contaminants while observing the O-ring under the binocular microscope. I have found that this is particularly effective in removing debris without removing the vacuum grease that is essential for maintaining a good vacuum seal. After using this technique for many years, I have never seen any evidence of the elastomer glue on the masking tape adhering to any part of the specimen holder, let alone to the slippery O-ring.

For holders where there is a ceramic tip that rests on a bearing, it is essential to keep the sliding surfaces clean so as to minimize friction and avoid drift problems. This is another example where the ubiquitous masking tape can be used. Simply take a clean piece of masking tape and, under the binocular microscope, clean the bearing at the end of the holder. I have done this for many years with beneficial effects. Using the binocular microscope, it is easy to check that the rubber-based viscous glue does not adhere to the bearing, something I have never observed to happen.

It might be argued that attempting to sustain a high level of the microscope vacuum by a simple cleaning procedure will be too much trouble. In response, I can only point out that if a specimen were free from hydrocarbons, the only way a focused beam would produce visible contamination spots in either of our microscopes would be by warming up the cold finger. Even creating carbon deposition in a reasonable time that could be detected by EELS (for example, for calibration purposes) has often been impossible with the cold finger in operation.

MT

https://doi.org/10.1017/S1551929510000477 Published online by Cambridge University Press

Expand your Knowledge of Microscopy with MSA Membership!

Whether your primary focus is in light, electron, or scanning probe microscopy, or the biological or the physical sciences, MSA takes your knowledge to the next level!

Members Receive:

- A personal subscription to MSA's official journal, *Microscopy and Microanalysis*, and MSA's popular bi-monthly magazine, *Microscopy Today*.
- Peer Networking through the Society's Focused Interest Groups and Local Affiliated Societies.
- Discounts on books, journals and other educational materials.
- MSA Awards Programs, Scholarships, Speaker Opportunities, and more!

MSA
Microscopy Society of America

Join MSA Today!
Visit: www.microscopy.org or call 1-800-538-3672