

## Microscopy101

# Increased User Safety from Glass-Free Liquid Nitrogen Dewars

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In a multi-user facility where students cycle through every few months as part of short-term projects and internships, maintaining safe operation of the equipment as well as ensuring the safety of users is of paramount concern. Thanks to films such as *The Terminator*, most users are aware of the freezing potential of liquid nitrogen (LN). However, users may not appreciate the explosive power associated with the rapid conversion of liquid nitrogen to gaseous nitrogen. When liquid nitrogen comes into contact with a surface warmer than its boiling point ( $-196\text{ }^{\circ}\text{C}$ ), a 700-fold expansion of liquid to gas occurs. This conversion can easily turn a broken glass vacuum dewar insert into lacerating projectile bits.

In response to two explosive incidents involving liquid nitrogen dewars in our biology department, I looked for an alternative that might minimize future dangerous events. We were fortunate that in both previous cases, which occurred in two unrelated laboratories, no one was injured. In the first, a break in the wall of a glass-lined dewar created a directed blast into a nearby paper towel dispenser, shattering the plastic cover. In the second case, an icicle of water, created by long-term use of the anti-contaminator on our transmission electron microscope (TEM), formed between the glass-lined dewar and the arm of the anti-contaminator assembly (Figure 1a). When a user gave the dewar a slight twist to break the icicle, the twist instead broke the LN-filled glass liner. When the LN hit the metal walls of the dewar, it vaporized. The rapid expansion of LN within the narrow bore of the dewar, coupled with the fragmentation of the glass into minute pieces, led to an upward explosion of glass fragments that the user described as a “stream of confetti shooting to the ceiling.” For weeks afterward, we found glass “confetti” in and around crevices in the TEM room. Discussions with our service engineers revealed that similar breaks in glass-lined dewars have occurred in labs around the world.

As we looked for a replacement of our 1-liter glass-lined dewar (Figure 1a), we were also interested in finding a larger dewar that would allow for automated image capture over longer time periods than the 3–4 hours provided by our 1-liter dewar. Another local laboratory tried a 2-liter stainless steel wide-mouthed coffee dewar obtained from a local big-box store. The wide mouth was useful for insertion of the copper braid into the LN (Figure 1b). Since then we have used a similar dewar with great success. Because the commercial coffee dewar was not as tall as the 1-liter scientific dewar normally used, we initially used a surplus Styrofoam™ shipping container for gallon bottles of solvents (Figure 2a). The inner diameter of this container measured almost exactly the outer diameter of the stainless steel dewar and raised the dewar up the inch or so necessary for proper location on the TEM manufacturer's support stand. With usage over time, however, the Styrofoam container shed little

Styrofoam particles around the room. We subsequently replaced the Styrofoam container with a surplus plastic pipette washing cylinder cut down in height (Figure 2b) and filled it with a thin foam spacer to hold the dewar in the cylinder.

Although the major benefit to using the stainless steel coffee carafe dewar is the reduced chance of user injury, there are financial benefits as well. The 2-liter coffee carafe dewar costs approximately \$50, compared to \$160 for a 1-liter glass-lined dewar or \$450 for a 1-liter scientific stainless steel dewar. Using the larger 2-liter dewar, our normal TEM operating vacuum improved with LN in the anti-contaminator dewar, and the time between dewar refills was significantly increased.

The interface between the coffee carafe and the Styrofoam lid on the instrument does frost up more than the standard 1-liter dewar; some care must be taken to catch the frost into the plastic or Styrofoam enclosure rather than have it fall onto the equipment during dewar refill or removal. As a result of catching this frost, water from the melting frost slowly accumulates in the space between the outer container wall and the outer wall of the carafe. This water should be poured off prior to refilling the dewar with liquid nitrogen.



Figure 1a: Standard 1-liter glass-lined dewar on a TEM.

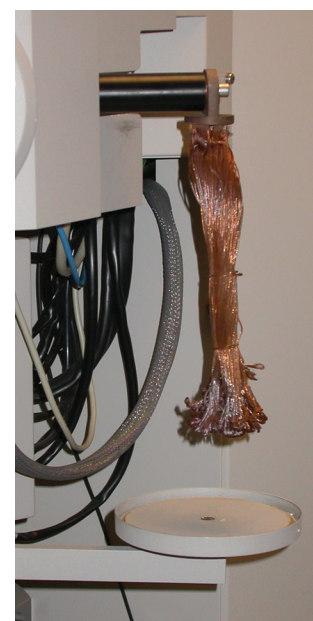
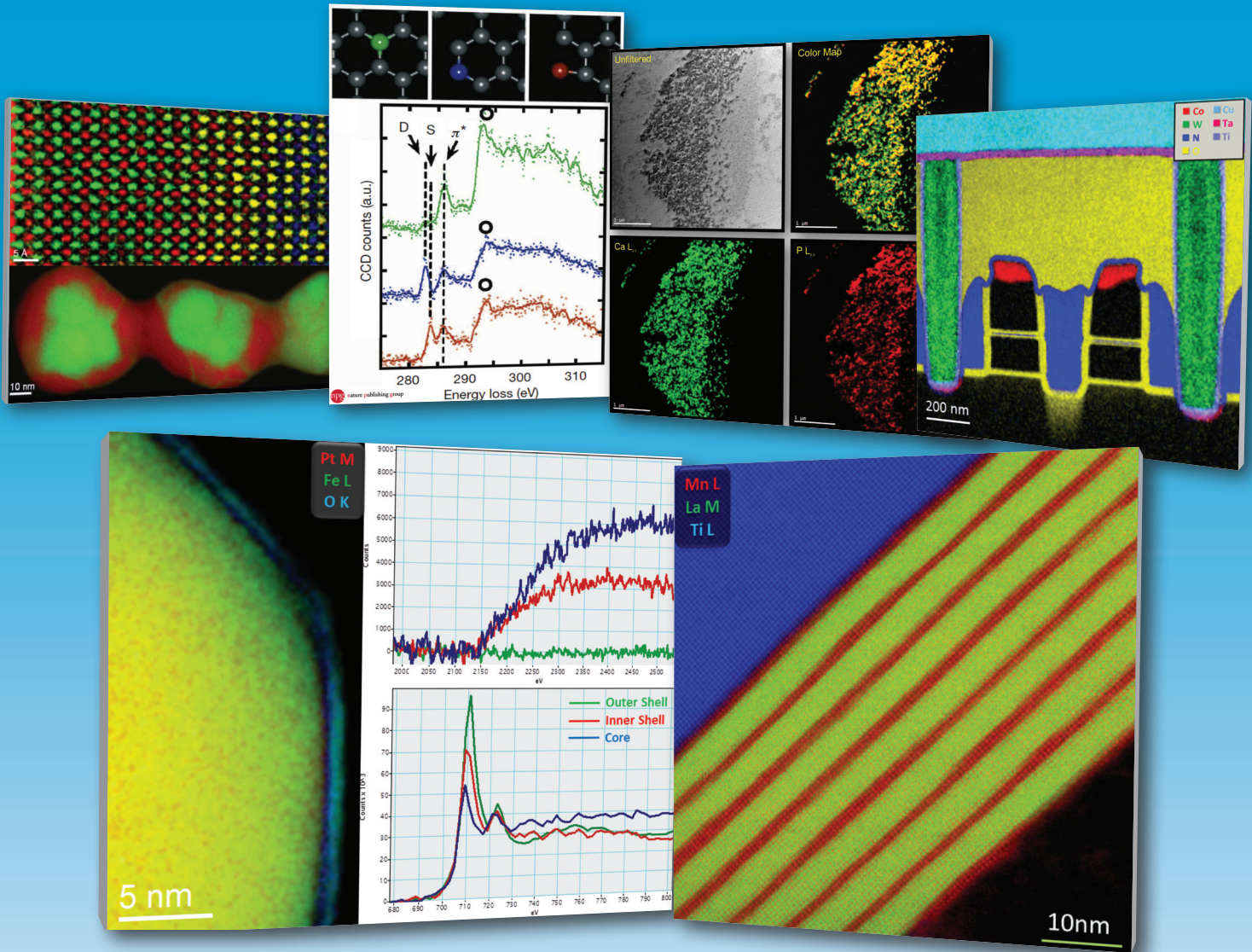


Figure 1b: Copper braid that transmits cooling from the dewar to the specimen chamber inside the microscope column.

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Top Images, left to right:

- (Top) Colorized elemental map showing Sr  $L_{2,3}$ -edges (green), Ti  $L_{2,3}$ -edges (red), La  $M_{4,5}$ -edges (yellow), and Mn  $L_{2,3}$ -edges (blue). Data captured using a Gatan Enfium™. Sample courtesy of Prof. David Smith, Arizona State University.
- (Bottom) RGB composite EELS SI image of Au/Pd nanoparticle; Au  $M_{4,5}$ -edges at 2206 eV in green and Pd  $L_{2,3}$ -edges at 3173 eV in red. Data captured using a Gatan GIF Quantum™ER. Sample courtesy Dr. Jianfang Wang of The Chinese University of Hong Kong.
- ELNES of individual atoms in graphene. Different states of atomic coordination are illustrated at top. ELNES of carbon K (1s) spectra shown on bottom. Green, blue and red spectra correspond to the normal sp<sup>2</sup> carbon atom, a double-coordinated atom and a single coordinated atom, respectively. Data captured using a Gatan Quantum™ER Low-Voltage Special. Data courtesy of K. Suenaga and M. Koshino (AIST, Tsukuba, Japan). Figure 1 from: K. Suenaga et al. "Atom-by-Atom spectroscopy analysis at graphene edge"; Nature 468, 1088 (2010). Permission to use Figure 1 granted by K. Suenaga and Nature Publishing Group, Copyright © 2010, rights managed by Nature Publishing Group.
- Unfiltered, conventional TEM image and elemental maps of a capillary blood vessel captured using a Gatan GIF Quantum™ER. The Ca and P elemental maps were extracted from an EFTEM-SI dataset acquired using Gatan's DigitalMicrograph™ software. EFTEM-SI is capable of revealing relative concentrations below 1% as shown in the P elemental map. Sample courtesy of Dr. Wenlang Lin, Mayo Clinic.
- Color composite EELS composition map from data acquired using a GIF Quantum™ERS system on a 300 kV probe corrected STEM with 1.0 nA beam current. The EELS data was acquired at 4.1 ms/pixel with a 20 mR convergence angle and 42 mR collection angle. The 200 x 200 pixel map took under 3 minutes to acquire (164 s). The black areas are Si, which has been omitted for clarity. TEM facilities courtesy FELMI and TU Graz, Austria.

Bottom Images, left to right:

- (Left) Colorized elemental map based on Pt  $M_{4,5}$ -edge (red), Fe  $L_{2,3}$ -edge (green), and O  $K$ -edge (blue) intensities. Data captured using a Gatan GIF Quantum™ERS and 300 kV probe corrected STEM with 180 pA beam and 5 ms exposure.
- (Right) Extracted Pt  $M_{4,5}$  edges (upper) and Fe  $L_{2,3}$  edges (lower) from the thin outer shell (green), low density inner shell (red) and core (blue). Despite the sub-nm proximity of the outer shell to the core, no Pt is detected. The Fe-L2/L3 ratio and peak position vary significantly with the Fe chemistry of the layer. Sample and TEM facilities courtesy McMaster University, Canada.
- Colorized 1k x 1k elemental map of LaMnO<sub>3</sub> / SrMnO<sub>3</sub> superlattices grown on SrTiO<sub>3</sub> (Mn - Red, La-green, Ti L-blue). Data was acquired on an Enfium™ (UHV special) coupled to a 100 kV NION UltraSTEM. Image courtesy Mundy, Adamo, Schlom & Muller, Cornell University. (Results published in Monkman & Adamo, et al, Nature Materials, vol 11, 2012).



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Figure 2a: Coffee carafe in a surplus Styrofoam™ container.



Figure 2b: Coffee carafe in a surplus plastic pipette washing cylinder.

In summary, a simple stainless steel coffee carafe can substitute for a scientific-grade glass-lined liquid nitrogen dewar. This low-cost coffee carafe provides increased lab safety and longer times between refills.

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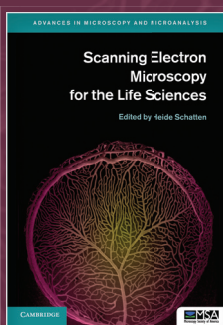
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