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History of High Vacuum and Critical Point Equipment used in EM Specimen Preparation

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Part 2

An interesting sidelight of building our own 3" diffusion pumps is that as we built ever larger vacuum equipment, larger pumps were required. Motivated by our success with the 3" pump, we gradually built a series of larger pumps going up one size at a time to 10" diameter.

Critical point drying was invented by Tom Anderson and described in his fine paper in 1952. He was a bit ahead of his time as the widespread use of the technique was not until the arrival of the SEM in about 1970.

I became involved in critical point drying when Dr. Gennaro of New York University asked if I could make him a unit.. He sent me a copy of Tom's original paper and we visited Tom's lab for a demonstration.

With Tom's dryer, the chamber opening and closing involved a high pressure threaded seal. Fortunately, he had working with him a man big enough to be a NHL defensive end who manhandled a three foot wrench to seal and unseal the chamber.

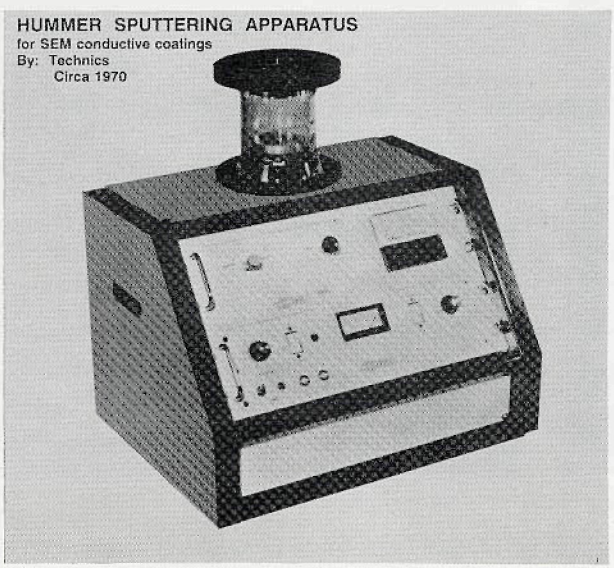
They say necessity is the mother of invention. As we obviously could not sell a "NHL defensive end" with each unit, we designed and built the first commercial critical point drying system with a much easier sealing system and a rupture disk to prevent overpressure - and we still make them.

Arthur Cohen of Washington State University developed another method of critical point drying using two freons instead of carbon dioxide and amyl acetate. Cohen added a viewport and electrical heating. A commercial unit patterned after Cohen's was made by Bomar in Seattle, WA.

With the SEM, dielectric specimens required electrically conductive coatings to bleed off electrons. This was first done in evaporators and many tilting and rotating mechanisms were employed to expose the entire surface to the evaporant. While I believe that we sold the first commercial one, I suspect that someone in Japan might have invented the procedure.

Almost all conductive coating is now accomplished by sputter coating using gas scattering to distribute the sputtered material. Original sputter coaters were straight DC and required relatively high voltage. This produced quite a lot of heat as well as electron bombardment of the specimen. At first, microscopists used any sputter apparatus they could buy, but as higher magnification became available, smaller specimens were employed and straight DC sputtering created damage.

An early DC sputtering unit was made by Ray Mathis, Inc. While not designed for electron microscopy work, it was probably the first commercial laboratory sputter system. Pat MacMahon, after working for M.R.C., did make up a few DC sputter coaters for EM work. The first widely used DC sputtering system for electron microscopy, however, was made up by Technics and was called the "Hummer" (see following Figure).



We at Denton Vacuum then introduced the magnetron sputterer for EM work. It employs lower voltages and produces much higher yield per unit of power which is much better for biological specimens. All EM sputterers now sold are magnetron. We added one further wrinkle, a grid anode which further reduces electron bombardment of the specimen. Other techniques such as cooling the specimen have also been used. Some of these mechanical pumped units are convertible for carbon evaporation with argon bleed.

Another answer without a question for a good many years was freeze drying. In this method, the specimen is quick frozen on the bench using liquid nitrogen in one way or another and placed on a cool surface in a vacuum chamber. The cooling in the chamber is done by multi-stage Peltier cooling.

The specimen must be kept solid to preserve the basic structure while the water sublimes. I believe the first system for this procedure was invented by Pearse in England and built by Edwards. It has a port at the top where a potting resin can be placed and dropped onto the specimen after dehydration. Then the current on the Peltier stage may be reversed to provide heat and set the resin.

For TEM work, the specimen must then be sectioned. As with freeze drying, this procedure did not receive wide use until the scanning electron microscope came along.

Russell Steere was a pioneer in the process known as freeze fracture or freeze etching. He did this work while a student of Robley Williams who had moved to the University of California, Berkeley. Steere spent three years at Berkeley working on this process.

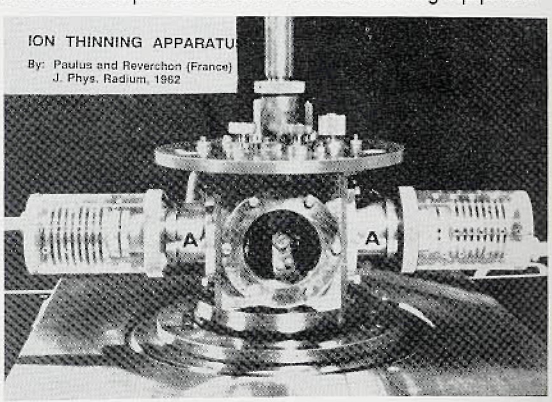
Balzers entered the field and later made the first production units specifically for freeze fracturing. They introduced a cleverly designed microtome for fracturing. Balzers makes most of the freeze etching equipment today.

As the field progressed, the necessity for minimizing specimen contamination was apparent. Steere's system, built by Denton Vacuum, utilized a liquid nitrogen cooled shroud around the specimen which is fractured, shadow cast and carbon is replicated inside this shroud.

The specimen is shadowed and replicated through two small apertures. This, I believe, was the first really clean freeze fracturing. Another approach is to pump the entire chamber to a very low pressure using cryopumps and liquid nitrogen traps. A Frenchman, Jacques Escaig, made some very sophisticated, complicated and expensive systems using ion and cryo pumps.

All drying and freezing techniques are for specimens containing water. One preparation method used on electrically conducting specimens was called cathodic etching. This was first used in 1948 by Don McCutcheon of Ford. The specimen was made the cathode in a DC discharge. Today I don't think this technique is used since there are now so many variations available of ion bombardment, plasma etching, etc. that it boggles the mind.

Many hard specimens such as metals, cermets, ceramics, semiconductors are prepared for TEM examination by ion beam thinning. Ion beam thinning is a standard method for TEM specimens in which the specimen is ground very thin, then placed in a vacuum system and bombarded with dual ion guns until thin enough for examination. This technique was developed in France by Paulus and Reverchon in 1961 (see following Figure). The first production systems were made in France, I'm told, by Alba, but no one seems to have any photos or literature on them. Several companies now offer ion beam thinning equipment.



It is now circa 60 years since the first electron microscope and we are now even writing with molecules in scanning tunnelling microscopes. I cannot imagine what the next 60 years will bring. ■

To argue the number
is a difficult win.
As there is but one angel
dancing on the head of a pin

. . Compliments of Jean-Paul Revel