Plasma Cleaning EM Stages, Specimens, and Columns:

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This note was edited from two posting to the MSA mail server.

In my 20 plus years of experience, I have found that nearly every specimen contaminates to some degree (some slower, some faster) in an electron microscope regardless of the manufacturer. Most of this contamination comes from the specimen, and it's magnitude is a function of the sample (metallic, semiconductor, organic, etc.), the method of preparation (electrochemical, chemical, microtoming, ion milling, etc.), the microscope conditions, the probe and probe current - plus a number of less well controlled factors. Reactive gas plasmas can fortunately be used to mitigate the contamination process and frequently reduce the problem to negligible levels. Basically, one places the specimen/stage in a "low energy plasma" where the gas acts like a catalyst for a localized surface chemical reaction. The energy of the plasma breaks weak bonds of the hydrocarbon compounds on the surface which then make these species somewhat volatile so that they can further react with the gas in the plasma. The subsequent action of the gas provides an additional chemical process which converts the compound into a gaseous phase which can be easily removed by the mild vacuum conditions. For example, here are some reactions which could be used if we were dealing only with pure carbon:

$$C + 2H_2 \rightarrow CH_4 \; ; \; C + CO_2 \rightarrow \; 2CO \; ; \; C + O_2 \rightarrow \; 2CO \; ; \text{ etc.}$$

Lots of other possibilities exist depending on the materials and "gas".

The effectiveness of this processing/cleaning depends upon the gas, it's pressure, and power of the plasma, but is dramatic in most situations. We have been able to minimize or remove specimen-borne contamination to a level where one can operate for eight or more hours without significant contamination. Of course, when it reappears (usually due now to the microscope) the specimen and stage is just "reprocessed" and one can continue working.

When critical small probe work is being done, I would recommend that both specimen and stage be plasma-processed before microanalysis, especially in LaB6 and FEG systems. If, after an experiment starts, the specimen begins to contaminate, the sample and stage can be removed from the microscope and processed off-line, then the analysis resumed.

An advantage of plasma cleaning is that it is nearly a room temperature process. I've measured temperature rises in a thermocouple TEM stage of about 5^0 C under cleaning conditions, which is less than one gets using a 150W flood lamp! Hence there is generally little temperature effect on most samples.

I also routinely apply this process to stages which have Be cups. If you operate under the correct plasma conditions, you will not sputter or etch material from or onto the stage. Sputtering only occurs when the power level of the plasma is too high (> 30W) and you enter the etching or "ashing" regime.

Once you clean the stage, the next area to consider is the immediate surroundings, which are also sources of contamination. The column of the microscope can also be plasma cleaned, by introducing a plasma into the column (with the e-beam off, of course).

EDS detector windows might also be cleaned by the plasma, but care must be taken. If the EDS window compound is a strongly linked polymer, then high energy plasma is needed to disassociate the OH bonds to make an appropriate reaction proceed. This will only occur if you are running plasmas on the order of 50eV, or with very chemically reactive gas compounds (i.e., something more than just Ar, 0_2, etc.). In this regime you can easily "etch" polymers and thus also a hydrocarbon based EDS window. If, on the other hand, you run with only very low energy plasmas (<10eV) this should not be a problem. I would recommend you test the EDS window material first. As in most materials, some compounds are most resistant than others.

My best guess is that there will be a set of conditions which will not cause problems for most hydrocarbon based thin window detectors, however, I have yet to test any here at ANL. On the other hand, if we are talking about cleaning only the EDS window on a detector installed on a column, I would not recommend this be done with plasmas, as the cleaning time will be far too long. It would be better to remove the detector and mount it on a special system, after it was cleaned by traditional methods.

Generally for plasma processing, I would recommend a power level of about 5-10W, pressure of about 200mT, processing time of about 5 minutes, and a two stage cleaning: first, pure Ar, then a second 5 minutes in O_2. The Ar disassociates the most volatile compounds first which then get swept away quickly. The O_2 finishes the job on the stuff that is more strongly bound. These figures are for cleaning specimens & stages; columns and EDS windows will need some testing for each case and likely will need a longer processing time.

Unfortunately, once surface hydrocarbons are removed, you may find out that your sample exhibits electron sputtering in the microscope. This is an effect which we calculated and showed would happen if the conditions are right (Zaluzec and Mansfield, 1987, Proc. AEM-87). Adding back a very thin layer of spectroscopically pure carbon sometimes cures this problem, with minimal contamination effects. ■

Note: the use of reactive gas plasma for cleaning the interior of an electron-optical column, samples, and specimen stages is covered by a patent owned by ANL, my employer.

Re: The Shroud of Turin

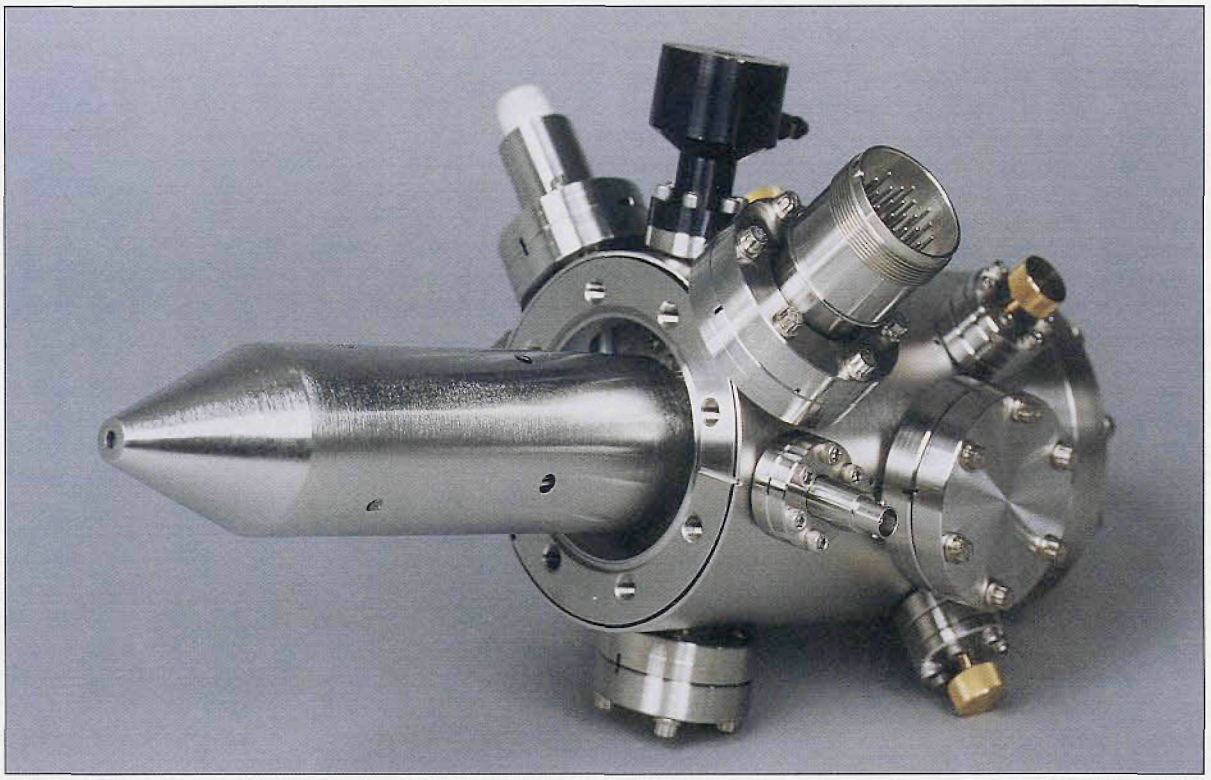
In our February/March issue, we published an excerpt from the new book "Judgement Day for the Turin Shroud" by Dr. Walter McCrone. This article covered one part of the technical analysis conducted by Dr. McCrone. The complete analysis is covered in the book and should be of interest to many microscopists.

In addition, Dr. McCrone reviews in some detail the continuing debate between the scientific and a small part of the "religious" communities. Included, for example, are copies of much of the correspondence over the some 20 years in which the analysis was conducted.

I found this to be an extremely interesting book and strongly recommend it to our readers.

. . . Don Grimes, Editor

The price is $36 plus $4.50 S&H. You may order your copy of "Judgement Day for the Turin Shroud" by Fax (312-842-1078), phone (312)842-7100), or eMail (wmccrone@mcri.org).



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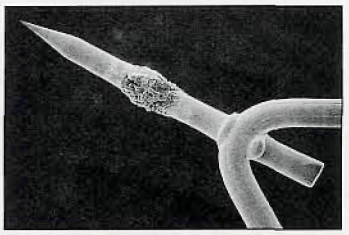

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