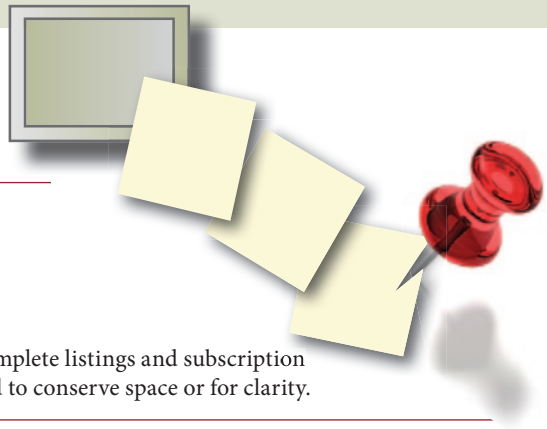

NetNotes

Edited by Thomas E. Phillips
University of Missouri

phillipst@missouri.edu

Selected postings from the Microscopy Listserver from January 31, 2010 to May 1, 2010. Complete listings and subscription information can be obtained at http://www.microscopy.com. Postings may have been edited to conserve space or for clarity.

Specimen Preparation:
BPA and mounting epoxies

Three years ago or so, when I last ordered two-part mounting epoxy for our lab and scrutinized the MSDS, I wouldn't have given a second thought to the fact that the main ingredient of the part is Bisphenol A, more commonly known as BPA. Since then, government and scientific reports have questioned its safety, a FDA report this year reinforced concerns about health effects, and companies are eliminating BPA from their products, especially those for babies, children, and pregnant women. Now it is time to order more mounting epoxy for our lab, and this time, upon reviewing the MSDS, BPA as a major ingredient raised a red flag. How have others dealt with this problem? Have you found epoxies free of BPA? Has anyone switched to, say, acrylic instead? Something else entirely? Should we even be concerned about BPA in mounting media? Are the alternatives just as potentially harmful? Any info about the safety of BPA in mounting media, alternatives to BPA-based epoxies, and other suggestions are welcome. ***Ellery Frahm frah0010@umn.edu Mon Apr 12***

We are coping every day with toxic/dangerous stuff. As professionals, we are supposed to handle these products accordingly and under these conditions I don't consider myself or my colleagues in danger. I have always been said that the components of epoxies are made inert after polymerization, so the trick is to work with gloves under a hood and to avoid adding the components in your coffee or licking the bottles. I am barely joking. I am pretty sure that avoiding any contact with your body should keep it safe. Even the polymerized form, you cannot say that it is over-manipulated. I am always shocked to see that we have to store ethanol and methanol with toxic substances and work with them under the hood, when 96% alcohol bottles are available in stores and that tobacco products are freely available all over the world. As I already pointed out before, eating red meat a lifelong, which is directly related to colorectal cancer, or smoking are probably far more dangerous than handling these kind of stuff properly a lifelong. In conclusion, I would say that yes you can search for alternatives if they exist. But don't let the MSDS overly concern you. Personally I use them more as a guide for correct handling rather than a warning for just handling dangerous stuff. Stephane Nizets nizets2@yahoo.com Tue Apr 13

I can understand your comments about MSDS sheets particularly when some companies seem to bombard you with several pages of information. However I do find that other sources tend to give very succinct and useful information. I would, however, say that I'm not happy about grouping methanol with ethanol. Ethanol may be recreational in appropriate quantities but methanol certainly isn't and toxic is quite accurate. Malcolm Haswell malcolm.haswell@sunderland.ac.uk Tue Apr 13

Specimen Preparation:
buffer for DAB

Does anyone know if DAB reaction product is compatible with Sorensen's buffer? The reason I ask is that we are going to do some TEM processing of DAB-stained tissue. The DAB staining will take place at another site. Then the stained tissue will be shipped to us for TEM processing. All of the references I have found use cacodylate buffer for the initial fixation and later TEM prep. However, we want to avoid shipping a hazardous material, and so would like to switch to Sorensen's buffer before shipping. Can anyone foresee a problem with doing this? ***Dorothy Sorenson dsoren@umich.edu Thu Apr 22***

The DAB reaction is sensitive to the buffer. There are several papers showing that the reaction is strong (strongest?) in imidazole buffer. Google Imidazole and DAB and these papers will pop up. Thomas E. Phillips phillipst@missouri.edu Thu Apr 22

I have found that some DAB protocols are sensitive to fading and so always do a quick rinse and then postfix with osmium. Then the tissues can go into a phosphate or other buffer for storage or dehydrated with ethanol stopping at 70% ethanol for storage. [My DAB-Nickel-GOD protocol uses 0.1M PO4 buffer.] Larry Ackerman Larry.Ackerman@ucsf.edu Thu Apr 22

Specimen Preparation:
LR white

I am having a problem with micro-bubbles in my LR White. Whether I polymerize the blocks in the oven or in the microwave I'm getting them. LR White's polymerization is exothermic, but would this cause the bubbles, which in turn are leaving holes in my thin sections? ***Tom Bargar tbargar@unmc.edu Wed Apr 21***

I apologize if you already know this but are you polymerizing in gelatin capsules with most/all of the air excluded, because LR white can be very fussy? If it's stored too long it can have problems but I don't recall bubbles. Malcolm Haswell malcolm.haswell@sunderland.ac.uk Wed Apr 21

Specimen Preparation:
liver

We have had consistent problems preparing liver for TEM. The tissue blocks are extremely brittle and nearly impossible to cut good thick sections for LM or ultrathins for TEM. There must be a trick specific for liver that we am not aware of. Would someone who processes liver for TEM please suggest a modification to our protocol? We fix in 1.5 % glutaraldehyde/1.5% phosphate buffered paraformaldehyde containing 0.05% tannic acid for several hours, rinse, postfix in 1% OsO_4 then dehydrate to 100% ethanol, rinse in propylene oxide, then infiltrate and embed in Spurr's epoxy and polymerize at 60-70°C overnight. ***Douglas R. Keene drk@shcc.org Fri Apr 30***

Could there be traces of water in the specimen or the resin? Spurr's resin becomes brittle (shatters like glass when you attempt to

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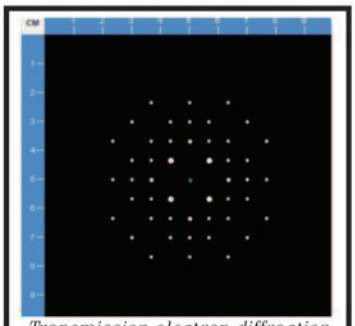
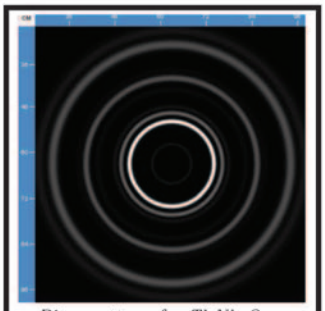
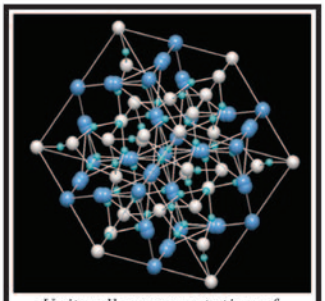


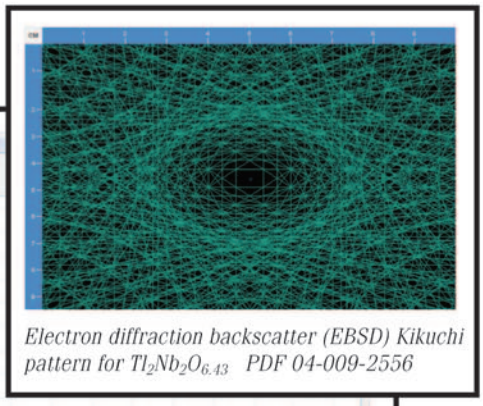
Transmission electron diffraction spot pattern for $Tl_2Nb_2O_{6.43}$ PDF 04-009-2556



Ring pattern for $Tl_2Nb_2O_{6.43}$ PDF 04-009-2556



Unit cell representation of $Tl_2Nb_2O_{6.43}$ PDF 04-009-2556



Electron diffraction backscatter (EBSD) Kikuchi pattern for $Tl_2Nb_2O_{6.43}$ PDF 04-009-2556

Electron diffraction powder pattern for $Tl_2Nb_2O_{6.43}$ PDF 04-009-2556

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trim it with a razor blade) when traces of water are present. Thick sections would be very difficult under such conditions. I haven't cut liver in many years, but I do recall that it is not one of the easier tissues to cut and tends to be crumbly or brittle, anyways. This would be exacerbated if residual water is present. Do you have any other embedments available, like Epon or relatives? John J. Bozzola bozzola@siu.edu Fri Apr 30 Fri Apr 30

Specimen Preparation:
samples preparation rate

I would like to know what is the suggested number of specimens for a technician to process to completion per year in an electron microscopy laboratory. I recall a publication from an MSA journal which suggested 250/technician. I would like to know if that is still accepted. ***Ronald E. Gordon ronald.gordon@mountsinai.org Tue Apr 6***

Our lab processed about 620 SEM and TEM biological samples last year, with the vast majority done by two technicians. Many (100's) miscellaneous other samples were prepared (negative stains, simple sample mounts with or without coating, particulates on coated grids, materials specimens, etc.), and many of these were done either by our four technicians or clients. My take is that 250 samples per year with full processing is a relatively light load, but keep in mind that we do primarily microwave processing which is way faster. Randy Tindall tindallr@missouri.edu Tue Apr 6

At one time the 250 specimens per year was a valid number, assuming you are referring to embedding/sectioning/photographing each specimen. That was when both film and paper were developed by hand (no film/print processors). With the use of print/film processors, around 400 samples were easily performed per year per technician. The total number of samples also is dependent on the type of sample and what is wanted out of them. If only negative staining, several hundreds per 3 month period is not out of the question. I am in a clinical pathology lab, using a tissue processor at night, rapid polymerization during the next morning, sectioning and scoping in afternoon. On average I process 3–4 blocks per tissue, obtain thick sections on each, then thin section only one of the blocks. The scope is outfitted with a digital camera and digital images are sent to the pathologist for review. No printing is done. Last year with myself and a part-time technician, we processed just over 1000 samples. The use of specimens/technician/year is to be used only as a ballpark statistic. How many other duties does the technician perform? Such as: updating standard operating procedures, ordering, maintaining equipment, attending mandatory meetings, etc. Edward P. Calomeni edward.calomeni@osumc.edu Tue Apr 6

Specimen Preparation:
ZnO particles for SEM

Can someone suggest a sample prep method for mounting and/or coating 50 nm–150 nm sized zinc oxide particles suspended in water for SEM? ***Fred Hayes fahayes@ucdavis.edu Thu Apr 22***

I've never done ZnO particles, but I've done small diamond particles. The particles were a little bigger than yours. I took a nebulizer and sprayed the particles over a hot substrate. It was a multi-layer coating on glass that was sitting on a hot plate. The idea was to dilute the particles sufficiently and evaporate the water rapidly before the water drops could touch other droplets. It worked fairly well. I then ion milled the surface at a low angle and the diamond particles acted as a mask to allow me to look at the multi-layers in the SEM. The particles stuck to the surface very well. If your particles are sufficiently deagglomerated and the solution is dilute, I'll bet that this technique will work for you. Scott D. Walck swalck@southbaytech.com Fri Apr 23

Resuspending in Ethanol or methanol will increase the evaporation rate further. Alternatively, you can try and find a suitable filter. Stephane Nizets nizets2@yahoo.com Fri Apr 23

If you have access to a plasma cleaner, I would take a piece of polished doped silicon (scrap from microelectronics) and get it hydrophilic by a run in the plasma cleaner. Then you put a droplet of your solution more or less diluted with ethanol, methanol or acetone. The silicon will give you a nice flat conductive surface, and there should be no need of a metallization, using low kV for observation (less than 3 keV). It's probably useful to ultrasonic the solution a bit after dilution. If there is less enough water in it, the solution evaporates too fast and the particles shouldn't agglomerate too much. You will find big clusters and in between isolated particles. Jacques Faerber jacques.faerber@ipcms.u-strasbg.fr Fri Apr 23

I've done cells and particles before. I put a syringe filter (13 mm) on a 1–3 cc syringe, And gently filter a diluted solution of particles, cells, etc. through. Then I use the same syringe filter setup to put the processing chemicals through, if I use HMDS, I remove the syringe and process the filter unit in a scintillation vial, seems to work fine, though times are not excessive. Then dry, even in mild vacuum, and either disassemble or cut out the filter to mount and coat. At some points, the filter may crack , or I intentionally use a hypodermic needle to create one small hole up the exit port into the filter, to assist when gently pushing fluids through becomes hard. This still works fine. The final product is on the filter, in pretty good quantity even with scarce samples, though it will have the hills and valleys of the filter ridges. This could be good or bad depending, sometimes that even helps. For coating, I stand the stubs at different angles for multiple short coatings. Lou Ann Miller lamiller@illinois.com Fri Apr 23

As been mentioned, you need a conductive surface and the Si route should work fine. There are other quick methods that do work if you have a reasonable amount of particles to play with. I have used Formvar-coated TEM grids in the past with reasonable amount of success (Au, and Ag particles and clays from suspension) particles and a lot quicker of you have holey carbon grids (not lacy) in your EM unit. I also did some work on plain polished SEM stubs. One of my favorites is SPI sticky tabs. (from my experience, the other manufacturers are not as sticky). We left some of them for two weeks in the field to collect dust particles. Just glue down on a stub, drop the solution on, let it evaporate and if you are fortunate, happy viewing. We are currently also investigating different filers and we hope to get to a good suggestion soon. Stephan Coetzee s.h.coetzee@gmail.com Sat Apr 24

The simple method that I would use is to coat a cover slip with gold and then to deposit "blobs" of the media having given it a chance to mix through placing in an ultrasonic bath for a few minutes. As the glass should be perfectly clean all that you will see are your particles. With very fine structures and low kV there is often no need to coat with this method. Steve Chapman protrain@emcourses.com Sat Apr 24

Specimen Preparation:
sputter coater consumption rate

We are in the process of purchasing our first SEM here at my company and I was asked approximately how often we'll need to replace the target for the sputter coater. I work in R&D for a pharmaceutical company, so pretty much all of our samples will need sputtering. Does anyone have any idea how long (# hours of use) an Au target would last for? ***Heather Eberhardt heather.eberhardt@gmail.com***

Sputter-coater targets are like brakes on a car: depends on how hard (thickness of coating) and how often you use them. Also, it depends on the thickness of the target itself. Ask the manufacturer.

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They usually have a good idea. On average, we replace our target every 2–3 years. My recommendation is that if you coat a lot of specimens, figure a replacement every year and a half. There is something to be said about having one or two in reserve and ready to go when needed. John J. Bozzola bozzola@siu.edu Wed Apr 14

Do not buy Au (pretty looking specimens, grainy coating). Go instead with Au-Pd target. Longevity depends very much on type of coater, thickness of target and frequency of use. Ballpark estimate: 0.5–3 years. Vladimir M. Dusevich dusevichv@umkc.edu Wed Apr 14

Specimen Preparation:

polishing epoxy mounted cross sections

I am a new user to epoxy and will be mounting cross sectioned samples which will require polishing after the cure is complete. I will be using Pelco epoxy resin with fast curing epoxy hardener. The samples are electroplated plastic parts. I hope I have chosen the right product for my application. The cross sectioned pieces are being prepared for SEM evaluation. An older model Buehler grinder/polisher with water will be used for the sample prep. In reference to respiratory health, what precautions does someone need to take during the epoxy polishing process? Is wearing some type of mask recommended? If so, is a disposable particulate respirator mask sufficient? There is an attachment which could polish the samples overnight when no employees are in the lab. Would it be safe for me to enter the lab without respiratory protection to turn the grinder off and remove the samples? ***Cathy Shelton cmshelton@srggi.com Tue Apr 13***

After the epoxy cures, the biggest worry would be the dust generated by the grinding. Keeping the sample and grinding surface wet will eliminate the dust. I have always felt that as long as I did my epoxy mixing and cure in a hood and kept the water running while I sectioned, I didn't have any respiratory worries about grinding epoxy. Our safety people are in agreement with me. I've been polishing epoxy-mounted cross-sections for 25+years and the worst thing that's happened to me is grinding my nails down at odd angles. On another note: I've never been a fan of fast-cure epoxies; they tend to have a lot of residual stress in them and tend to pull away from the samples. You'll just have to see how it works with your samples. Becky Holdford r-holdford@ti.com Tue Apr 13

The water used in the grinding process provides several benefits. The removed media gets entrained into the water and goes down the drain instead of potentially into the air. Thus, there is no need for respiratory protection. The water also cools the sample, protecting the sample from heat damage. The entrained particles do like to settle out in the slow-flowing drains, which may eventually plug. But, that will be a different question to post! I would assume your final polish is a diamond paste (or similar) on a felt cloth; no protection is needed here as well. The felt cloth essentially holds the debris. As the felt wears out, this debris does get spun off, but the oil/grease in the paste entrains the particles. Occasionally a sample gets multiple facets from a poor grinding step; a water cooled belt sander is then nice to have to flatten the sample out and start over. Rick Ross richard.ross@allison-transmission.com Wed Apr 14

Specimen Preparation:

SEM of glass filters

I have some glass filters that I want to do SEM on. I know they will charge like mad unless I use extremely low KV. However, my 'free' SEM is limited to one that is not able to be used at that voltage. Over the years, I have looked at these types of filters but I was wonder if there are there any new techniques out there? I don't need high magnification. ***Vicky Bryg victoria.m.bryg@nasa.gov Sun Jan 31***

Can you coat them with Au/Pd or some other sputter coated metal? It is thin and can be wiped off after use/analysis. I would not recommend pure Au. Pd is probably better. It would seem that the main issue is to remove the coating without affecting the optical quality of the glass. Perhaps you could try a sacrificial specimen and see if this works. I have done this on cover slips without any problems. But of course, these are different specimens. Gary Gaugler gary@gaugler.com Sun Jan 31

By the time I connect to grid you probably will receive multiple responses with similar suggestions, but anyway: from my somewhat limited experience of imaging photolithography masks (Quartz substrate charges like mad and retains charge under the surface) E-SEM in wet mode has inherent charge neutralization mechanism which works beautifully for dielectric materials. I am sure you could find an E-SEM in one of the universities nearby. Valery Ray vray@partbeamsystech.com Sun Jan 31

LM:

uranyl glass as fluorescence standard

I received a request for information regarding uranyl glass related to fluorescence intensity. Quote: "The average NADH fluorescence intensity of each surface area is related to the fluorescence intensity of a fluorescence calibration glass(uranyl) and is expressed in arbitrary units." Can anyone shed any light on what is uranyl glass? ***Alex Besenyo bioanalytics@ibilabs.com Fri Mar 26***

It is a glass that has a uranyl salt incorporated into it. The slides are generally yellowish if I remember correctly. I bought some years ago but not sure where. They give off a strong fluorescent signal that if resistant to fading so they can be used as a calibration aid. The trick is measuring the "real" signal of the unknown without it fading. Tom Phillips phillipst@missouri.edu Fri Mar 26

As well as providing a fluorescence 'standard', I believe that uranyl glass slides could also be used to background correct uneven Hg lamp illumination, in a similar manner as done with transmitted light illumination, as their UV-excitation fluorescence was so uniform. I always wanted a uranium glass slide, having worked so long on the health effects of uranium and it's unpleasant cousins in the actinide series [Pu-238 & Pu-239] and as they just seemed cool [they literally look brilliant in the right light, e.g. UV from the sun or microscope]—and they are useful as that fluorescence standard under the microscope. You can accurately measure the concentration of uranium in solution using its fluorescence [although I used 'delayed neutron analysis' for uranium in tissue]. However now that the health effects of uranium are better understood, you seldom see things made from this material on the scientific supermarket shelves any more—no doubt improved health and safety makes it a pain to handle for non-military applications and buyers are now wary of uranium's radioactive and toxic properties. Likewise their use as a bright yellow colorants in pottery glazes and glass has largely been abandoned, and predictably it's made them quite collectable as secondhand items as owning the final product is generally safer and far easier than producing it [not that I fancy eating off 1930s radioactive 'FiestaWare red' dining plates, although I'm sure it's all 'safely contained' in the glaze]. Uranium glass may just contain the uranium oxide, producing a clear yellow/yellow-green glass, or other minerals may be added to produce an opalescent white [Vaseline] glass—and these household items can be easily bought on eBay. I have never managed to find a supplier of uranium glass 3 × 1 inch slides in the UK, although there was a supplier of the sheet glass in the US: Newport Industrial Glass, Inc., 1631 Monrovia Ave., Costa Mesa, CA 92627, but this was from a listerver posting by George McNamara way back in 1995 [see below], the company still exists though. I never got around to contacting them. I have used suspensions of ultrafine 0.044 μm fluorescent fluorescein microspheres in solutions and gels

as microscopy standards [Duke Scientific now Thermo Scientific, also see Polysciences Inc] and I had some colored plastic 3 × 1" slides back at UCL that autofluoresced quite well [no idea where they came from]. Used as a microscopy standard:
http://jcm.asm.org/cgi/reprint/30/5/1294.pdf
http://jcm.asm.org/cgi/reprint/27/3/442.pdf
http://www.springerlink.com/content/v122261751832k04/fulltext.pdf
Uranium Glass in science:
http://www.sis.org.uk/bulletin/92/Brenni.pdf
Keith J. Morris kjmorris@well.ox.ac.uk Tue Mar 30

Have you tried our FluorRef slides? They are an excellent substitute for many of the things recommended below (background correction, demonstrating light paths, settting consistent power levels for lasers, etc.). They come in four different colors, to match the fluorescence of conventional fluorochromes. You can learn more about them at MicroscopyEducation.com. Barbara Foster bfoster@mme1.com Tue Mar 30

Are these 'Fluoref slides' plastic and in four pretty colors to the eye? If so I presume they are my "I had some [4] colored plastic 3 × 1" slides back at UCL that autofluoresced quite well [no idea where they came from]." They must have come supplied with a microscope system at some point, and were all in one slide box suggesting a set. Shame your website has no pictures of them, unless these are 'on the library' virtual shelves somewhere. I used submicron 0.044 µm microspheres at a few different concentrations in gel to create standards to investigate the 'linearity' of the confocal system rather than background correct or use as a 'reference' fluorescence standard, so these Fluoref standards would be useful [although they do lack the single color fluorescence retro charm of uranyl glass slides]. Keith J. Morris kjmorris@well.ox.ac.uk Tue Mar 30

EM:
shielding column from electromagnetic interference (EMI)

We recently were forced to move our JSM6500F SEM. We now have problems both with AC and DC EMI. Has anyone tried to shield their column from EMI using a sheet of mu metal around the column, instead of using a field cancelation system? Our system is almost unusable at 1 kV. ***Pat McCurdy patrick.mccurdy@colostate.edu Thu Apr 22***

I have not used mu metal around the column, but was successful in reducing interference levels, in a past situation, by (mostly) enclosing the chamber working distance with a mu metal cylinder. Of course, had to leave some room for signal electron exit. Along this line, if the interference is significant, shielding only the column may not be enough. The chamber (or in my case the W.D. electron path) may need to also be shielded. FYI: For the most effective mu metal shield, it must not be work hardened. That is, if bent, it should be re-annealed. Woody White woody.white@areva.com Fri Apr 23

You say it's almost unusable at 1kV. At what kind of magnification? Perhaps you should try and go after the source of the EMI and see if you can reduce it. Mu metal around the column (and chamber) can be helpful, but if the problem is really severe, it may not do what you want. If the source is localized, it may be easier to shield than the column, although the shielding JEOL provides works pretty well. Ken Converse kenconverse@qualityimages.biz Fri Apr 23

EM:
handedness

This is not really a technical question, more just something that I've been pondering for a little while. I've noticed through my own experience as well as through several Google image searches that most SEMs seem to be "Right handed," as in the controls are to the right of the SEM column. I've noticed this across manufacturers and models. I was wondering if anyone had an explanation as to why this is, or if it is just random? It seems to me that it would be convenient for some to have the controls on the left hand side. ***Justin A. Kraft kraftpiano@gmail.com Fri Apr 16***

The only SEMs that I'm aware of that put the electronics on the left are ETECs. I believe their thinking was that most people are right-handed and would have the best control of the specimen stage using their right hand. Ken Converse kenconverse@qualityimages.biz Fri Apr 16

Philips in the 1970's were the only major SEM manufacturer who had the column on the right of the instrument. Once the column unit becomes self contained, with its power supplies within the one unit, the position of the desk is a customer option. For example, the Zeiss SEM the column has been self contained for many years and only needs a desk for the computer on the left or right. Steve Chapman protrain@emcourses.com Fri Apr 16

This interesting observation brought several thoughts to mind. If the console is on the right-hand side, then my left hand is closest to the column for such tasks as aligning apertures and opening chamber doors. As I sit at the chair at the center of our ESEM console (Electroscan 2020), the joystick for stage movements is mounted on the extreme left of the console, again favoring left-hand manipulation. And finally, has anyone else found that our engineering colleagues are disproportionately left-handed? That is, a larger proportion than the average population. Roger A. Ristau raristau@ims.uconn.edu Fri Apr 16

EM:
noise from gun chamber

Today we found in our SEM Jeol JSM 35CF a "rattling" noise in the upper part of the anode chamber where is the white ceramic part. Please see the image, http://www.kaker.com/test/5.jpg. Microscope condition: 25 kV, HV on, Filament off, vacuum is OK. I am especially interested about the reason for this nosie. ***Henrik Kaker henrik.kaker@guest.arnes.si Thu Feb 18***

I am very confused by the rattling noise, could it be a tinkling noise due to high voltage discharge? A check would be to lower the kV and see if the noise became less, if it does it's discharge! What causes high voltage discharge? 1. Poor vacuum in the gun, this does not often show up on the penning gauge as it may be positioned a long way from the gun area. 2. A dirty gun chamber (if it has an oily ozone smell that's discharge) which should be cleaned until all the smell has gone away. Clean the walls and the ceramic as I believe it is glazed therefore it is to be treated like a metal surface. Remember that an ammonia solution will dissolve tungsten. Steve Chapman protrain@emcourses.com Thu Feb 18

TEM:
maintenance

We're using a JEOL JEM 1011 TEM in our Histology Dept. since Jan. 2005. The microscope and its ancillary devices (rotary pump, chiller, UPS, etc.) were on for 24 hours almost all this time. My question is: how frequently we should change the rotary pump oil and also which maintenances do you recommend we should performed periodically? ***Necat Yilmaz nyilmaz@mersin.edu.tr Mon Mar 29***

To enhance their efficiency and their life we would suggest you change the rotary pump fluid at least once a year. The fluid is performing a number of tasks (1) Lubrication (2) Anti corrosion (3) assisting with the vacuum seal. If you have a compressed air driven system with its own compressor its fluid should also be changed, but

most of all the tank should be drained of the moisture which will have built up inside (compressing air produces moisture). Other areas requiring attention are the "O" ring on the specimen exchange rod, the specimen seat(s) and possibly the objective and condenser movable apertures. If astigmatism levels are high or you are able to see dirt on the apertures when they are visualised during operation they need cleaning (high vacuum heating) or replacing. If you are regularly using 100 kV every few years the gun chamber needs a good clean as the build up of contamination will cause more micro discharge. Over a period of up to five years the viewing screen(s) will degrade through contamination (depends on the instrument manufacturer) and should be replaced. In all of the above check out the instruction manual prior to making any attempt to carry out any action. Steve Chapman protrain@emcourses.com Tue Mar 30

SEM:
high voltage tank oil

I know I might be opening up a Pandora's box of warnings here, but I am in a situation where I have two extra HV tanks that are sitting around not being used in microscopes, and I was thinking that it would be more practical if I could remove the oil somehow and store it appropriately so that I can access the components. One of the tanks already has only half the oil it started with (It was like that when I got it) so I can't use it as-is anyway. My question is first of all, what is an appropriate method of handling the oil—are gloves and an apron sufficient, or should I go full haz-mat? Once I drain the tanks, what is a safe/effective method of storing the oil? Will a standard PTFE 5-gallon paint-bucket type container be OK, or do I need to get a neoprene bottle of some kind? Finally, what is a good, safe method to clean up the remaining oil from the tank and the components so that they can be handled safely? ***Justin A. Kraft kraftpiano@gmail.com Fri Apr 2***

When were they manufactured? PCBs were banned in the US around 1977. I don't believe the newer dielectric oils are a problem, besides being messy. http://www.ehso.com/pcbs.htm http://www.campuserc.org/resources/EHSguide/TSCA/Pages/PCBs.aspx http://www.greenfacts.org/en/pcbs/l-2/1-polychlorinated-biphenyls.htm

The above links should give you some help in terms of determining if PCBs are an issue. Ken Converse kenconverse@qualityimages.biz Tue Apr 6

SEM:
sputter coater failure

We have a problem with a Balzers SEM-Coater SCD 040. There is no current. All fuses are o.k. Does anybody know how to solve this problem? ***Boris Reznik reznik@ict.uni-karlsruhe.de Fri Mar 19***

Did you check out the internal fuses? There are some on the internal power supply boards. Al Coritz sampleprep@earthlink.net Fri Mar 19

In our SCD040 this is a typical failure. Normally one of the rectifier diodes on the high voltage power supply fails (1M400X). Most common cause is trying to do ion etching. We did not attempt to modify the bridge as it is easy to repair and a kind of fuse. Francisco José Kiss kiss@demet.ufrgs.br Sat Mar 20

We finally found the solution- the connecting HV cable has been buckled and therefore there was no current. Boris Reznik reznik@ict.uni-karlsruhe.de Fri Mar 26

SEM:
calibration of *X* axis

Our JSM-5600LV SEM produces images with 12.5% compression of the X axis. We bought this scope second hand from the USA and we think this compression may be on purpose; it converts the image from 4:3 to 3:2, because iif you expand 12.5% the X axis to correct the compression you get: 4+12.5%:3 = 4.5:3 = 3:2 Whatever the explanation is, we would like to correct this X compression. Is it possible to do it ourselves and if so, can anybody give us instructions how to do it? ***Yorgos Nikas eikonika@otenet.gr Thu Apr 22***

I wondered whether the distortion may also be linked to slow scan controls and TV technology, as I've learnt that TV standards, e.g., PAL and NTSC, don't have square pixels like a 'normal' PC. We have similar but distinct distortion issues to correct with our Japanese (NTSC?) and European (PAL) SEMs. FEI gave us a program for their SEM, but for our older JEOLs and their add-on digital capture we use Photoshop to correct the images: Image Menu–Image size Select "Resample Image" option, but deselect the "Constrain proportions" option and then you will be able to add 12.5% to the 'width' pixels dimension box (not document), independently of the height dimension. The 'actions' palette gives you a way of making an automated macro to adjust them in bulk by recording the steps you've established to do this. Remember to reset 'constrain proportions' when you've finished to avoid distorting any other images you may be working with! Peter Davies P.Davies@swansea.ac.uk Thu Apr 22

EDS:
chemical dating

I was talking to a friend of mine the other day, and we were thinking that it might be possible to get a very rough age estimate of some mineral formations using EDS and some methods such as Rb/Sr dating. We think it might be possible to measure the relative ratios of these two elements in a sample (At least on a polished surface of a mineral) and get a rough estimate of a date easily before doing more detailed analysis. I was just wondering if anyone has done something similar, or if anyone knows if it even can be done. We recognize that there would be a number of assumptions made about the sample, such as uniform consistency, etc..., but we think it might be possible. Any thoughts from the group? ***Justin Kraft kraftpiano@gmail.com Thu Apr 1***

The only reliable chemical dating method uses the U/Th/Pb systematics. Usually this is done with the mineral monazite. You have to very accurately measure the concentrations of these elements. The assumption is that all the Pb in the mineral comes from the decay of Th and U. There will not be a lot of Pb depending upon the initial concentration of Th and U and the age of the mineral. The method work well on very old minerals (many hundreds of million years) and using wavelength-dispersive spectroscopy. I don't think you would get a very accurate age using EDS. Go to Mike Jercinovic's home page to see what's being done: http://www.geo.umass.edu/faculty/jercinovic/ Ken Livi klivi@jhu.edu Thu Apr 1 11

It's not a bad idea, but one of the major challenges to doing Rb/Sr dating with EDS is the poor X-ray resolution of EDS. Consider that one would be attempting to analyze trace amounts of Rb and Sr in silicate minerals, meaning there will be a huge Si K-alpha peak at 1.740 keV. The L-alpha line for Rb is at 1.694 keV (a 46-eV difference), and the L-alpha line for Sr is at 1.807 keV (a 67-eV difference). Even the best EDS systems have resolutions no better than 120 or 130 eV. In other words, EDS couldn't resolve Rb and Sr L peaks from the Si K peak. WDS on an electron microprobe, though, can resolve X-rays on the order of 5 eV (http://probelab.geo.umn.edu/electron_microprobe.html). An alternative, of course, is to use the K-alpha lines for Rb and Sr, which have energies of 13.395 and 14.165 keV, respectively, but one would need an accelerating voltage of 25 kV or 30 kV to effectively excite those X-rays. If the silicates in question could handle high accelerating voltages without beam damage, that might be an option. Otherwise, one would have to instead use an electron microprobe,

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and even then, tiny Rb and Sr peaks in the presence of a huge Si peak might still be a challenge and require some work. The other problem is that one would not know the ratio of Sr-87 to Sr-86 without adding another technique or making an assumption about the mineral. As Ken noted, there is also monazite dating, using U/Th/Pb, that has been done in a few electron microprobe labs, including, most notably, Mike Jercinovic's lab at UMass. It has also been done by researchers at our lab at the University of Minnesota (http://probelab.geo.umn.edu). Ellery Frahm frah0010@umn.edu Thu Apr 1

EDX:
penetration depth program

I would like to ask if anyone knows of a shareware program that is able to calculate the EDX penetration depth? From what I was told, it is the shareware program which goes by the name of casino, but an online search returned with 'other' results as one would imagine. ***Jhun Yew jhun.yew@gmail.com Sun Mar 14***

If you add electron penetration to the search you'll find http://www.gel.usherbrooke.ca/casino/What.html I think this is what you're looking for. Ken Converse kenconverse@qualityimages.biz Sun Mar 14

You can download Casino from my company's technical page along with a number of other freeware programs useful for microscopy: http://www.probesoftware.com/Technical.htm The Casino download link is at the bottom of the page. John Donovan donovan@uoregon.edu Sun Mar 14

Please see the link, http://www2.arnes.si/~sgszmera1/html/monte_carlo.html. Henrik Kaker henrik.kaker@guest.arnes.si Sun Mar 14

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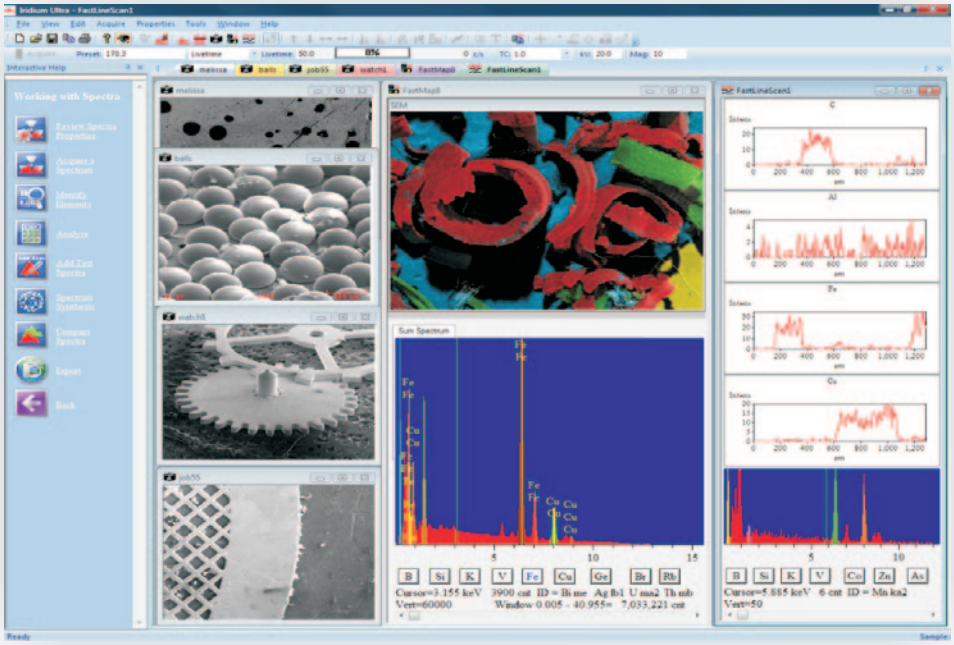

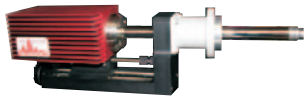

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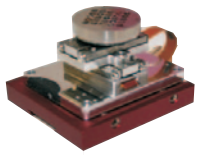

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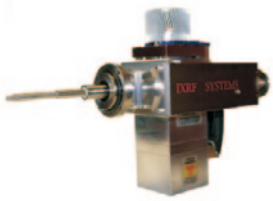

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