

NetNotes

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Selected postings from the Microscopy Listserver from March 1, 2016 to April 30, 2016. 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: cleaning SEM mounts

In response to a question asking for a good method for cleaning aluminum specimen mounts so they can be re-used:

Depending on what the mounting medium was, you may be able to remove contamination with a solvent, although it will probably still require some elbow grease. Sonicating the mounts may help speed up the process. Conductive tape is removable if you take it off after the SEM examination is complete, but the longer it stays on the mount, the more difficult it becomes to remove. If you have access to metallography prep equipment, the easiest thing to do is just grind the top of the mount down on an abrasive disc, exposing fresh aluminum. Then, depending on the surface you want, you can leave it with a rough finish, or use finer grits to get a more polished surface. **Jeff Hall** jhall@2spi.com Thu Mar 10

Most of the adhesives and paints are soluble in acetone or methanol, including tape gums, carbon paint, silver paint, and cyanoacrylate glues. Isopropanol and ethanol can also work, but require more scrubbing, as it just softens the tape adhesive. I have had success in cleaning batches of SEM stubs by soaking them in acetone, wiping any tape off, then sonicating them in acetone or alcohol. Any harder residues can then be removed with a quick brush over fine sandpaper. It is a bit time consuming, but it has worked pretty well for me. **Jake Jokisaari** joki@umich.edu Thu Mar 10

For cleaning large quantities of mounts, the use of a shaker will minimize, if not eliminate, scrubbing. Either a “real” lab shaker, if you have one, or the pneumatic type used for paint mixing. Put used mounts into container (steel, SST, Al, PTFE, etc...), fill with acetone, put in the shaker, leave running in a fume hood either overnight, or even over the weekend. Soaking for a day prior to putting the mounts in the shaker may work better. Or, run during the day and switch off overnight for safety. Can change acetone and shake a little more if lots of carbon tape was used. Rinse with alcohol, dry, and re-use with virtually no scrubbing. **Valery Ray** vray@partbeamsystech.com Thu Mar 10

This is an interesting discussion and a topic we have discussed a few times here at Ted Pella, Inc. given that we are manufacturers and suppliers of aluminum SEM stubs and Carbon Tabs. So far, our back of the envelope calculations say it is not economical to go through this cleaning given the cost of labor, chemicals and materials to get them ready for use again. Aluminum stubs range in price from around \$0.23 each for the small 12.7 mm size — to \$1.60 for the 25 mm size — to as much as \$3-7.00 for more complex mounts with 45° angles or adjustable surfaces. So spending more than a few minutes and minimal chemicals/wipes, and it becomes a losing battle cost wise. However, that doesn't factor in your cost to place an order or shipping. If you don't have a simple purchasing ability for supplies and must deal with an onerous system, that cost in time and labor

could perhaps justify cleaning the carbon adhesive off. One also has to factor in the environmental cost of chemicals and disposal. Likewise, what is the cost recovery of recycling the aluminum which is the more valuable 5000 and 6000 series materials? We at Ted Pella have considered offering a system of specialized solvent/sonicator, grinder or bake-off but the costs don't seem to justify cleaning and buying replacements is. But of course we like selling more stubs so take all that with a grain of salt. So, question to you with grad students — This would seem like an interesting paper for the EM community to determine the economics and best practice. Ted Pella, Inc. would be willing to consider funding this research and publication of results. Please contact me privately to discuss. **Mike Toalson** mike_toalson@tedpella.com Thu Mar 10

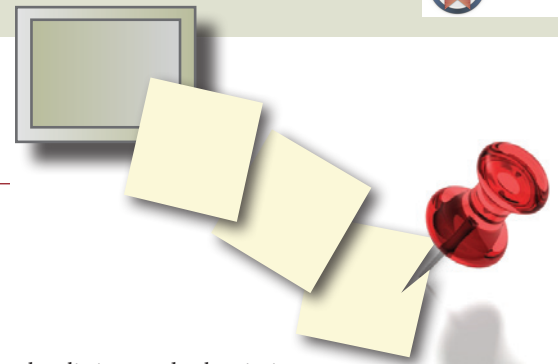
A year or two ago, we managed to make a set of insulating stubs by cleaning them. Our cleaning procedure was much as described in the last few days (razor blade scrape, followed by solvent shaking, followed by grinding). I cannot really fathom how the stubs lost conductivity (and batches we cleaned previously were fine) but lose it they did. Set us back months troubleshooting that one. Perhaps a cautionary tale in addition to the economic argument. **Tobias Baskin** baskin@bio.umass.edu Thu Mar 10

We routinely clean off old stubs. Partly because they're easily cleaned and reused, partly because we have a nice source of free undergraduate labor. Waiting for your next dehydration step? Clean stubs! The chemical costs aren't great, and the cost-recovery of recycling the aluminum isn't great either, so cleaning seems best. Plus, there's no shipping and handling costs for cleaned stubs vs. new ones. But so far we haven't managed to make insulating stubs. **Phil Oshel** oshel1pe@cmich.edu Thu Mar 10

Just a thought, but for a multiuser facility, it might not make economic sense for a microscopist to clean them, but it might be a good facility-level fix to put all the used stubs in a box and make it available to users with a standard operating protocol. That way, anybody needing and lacking one can grab one from the box and clean it themselves. It takes care of the labor aspect at least, and is essentially immediate recycling. **Jake Jokisaari** joki@umich.edu Thu Mar 10

I do not even bother to clean them. They are consumables. Mind you, if you don't clean the old ones properly you make your SEM/vacuum dirty. If you buy them via the internet for a 100 pieces they cost 23 euro's. The time spent cleaning them cost more. **Gert ten Brink** g.h.ten.brink@rug.nl Sat Mar 12

The only thing I can think of for making insulating stubs is this: glue solubilized in solution plus insufficient washing coating the stubs. As for the economics of cleaning: Hard to believing that throwing 100 stubs in acetone and waiting for the shaker to do its job is worth more than \$2. Just don't throw each stub at a time in 100 solutions on 100 shakers and clean them sequentially. **Stephane Nizets** nizets2@yahoo.com Tue Mar 15



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Specimen Preparation: drying molecular sieves

For drying (“removal of bound water”) of 3 A molecular sieve, the “web” recommends at least 300° Celsius (572°F). Would 250°C (482°F) for several hours be sufficient? Has anybody tried to dry molecular sieves in high vacuum or in a microwave oven? Has anybody a suggestion for a simple test / indicator if there are traces of water in ethanol or acetone? **Peter Heimann** peter.heimann@uni-bielefeld.de
Fri Mar 18

I’ve been using 200–250°C for “several hours” to dry molecular sieve for years. Works well. I do make sure to have some molecular sieve that has an indicator dye mixed in with the regular. It will revert to the “blue = dry” state in the oven, but it does wear out over time and needs to be refreshed. No handy test for traces of water, sorry. But, I do keep molecular sieve in my absolute ethanol. Either loose and handle with great care to not stir up dust, or in dialysis tubing. **Phil Oshel** oshel1pe@cmich.edu
Fri Mar 18

Are you thinking of “Drierite” (calcium sulfate) or molecular sieve? I haven’t seen colored molecular sieves. **Henk Colijn** colijn.1@osu.edu
Fri Mar 18

Molecular sieve. Indicating sieve can be bought, but it’s pricey, so I mix it 1:4 or 1:5 with non-indicating. I’m using mSorb. **Phil Oshel** oshel1pe@cmich.edu
Fri Mar 18

I keep the alcohols dry with dialysis tubing filled with sieve, and separate tubes of cupric sulfate as an indicator. It will turn blue when water is present. Roughly annually I have to recharge the sieve in the oven at 275°C overnight and recharge the cupric sulfate by heating in a crucible. I usually use a plumbers torch with spread flame or whenever I run the self-cleaning on the oven I pop last years in. When it turns white it is ready to go. Been using the same material 15 years now. **Scott Whittaker** whittaks@si.edu
Fri Mar 18

Specimen Preparation: sputter coater for FESEM

In response to a question about what type or quality of sputter coating is needed for samples that will be examined in a Field Emission SEM:

Au/Pd is probably too coarse. We had used gold and the grains were very evident at 50kx. We bought an iridium sputter coater and the grains are small enough that I have never seen them. We often use less than 5 nm of coating thickness. **Warren Straszheim** wesaia@iastate.edu
Tue Apr 12

I would guess that you are going to be working at a resolution that will exceed the grain size of Au/Pd. While there are certainly exceptions, I typically suggest the following as a very rough guideline: Gold up to ~10K magnification; Gold/Palladium up to ~50K magnification; Platinum up to ~100-150K magnification; above 150K magnification, either Chromium, Iridium, or Osmium. Each has its own advantages and drawbacks: Chromium is probably the cheapest option, but oxidizes very quickly into a non-conductive film. Iridium targets can be very expensive, but are available for most coaters. Osmium coating is done in a dedicated coater that will not sputter other materials, but the film is inert and amorphous. The source material is relatively cheap, but the coaters are an investment. A really good source of information is “Handbook of Sample Preparation for Scanning Electron Microscopy and X-Ray Microanalysis” by Patrick Echlin, who used to teach at the Lehigh Microscopy School. **Jeff Hall** jhall@spi.com
Tue Apr 12

It depends mainly on your samples and resolution you want to get. However, for FEG you should use a high resolution sputter coater.

I have good experiences with platinum coating (~ 3 nm) for biological samples. For x-ray microanalysis we use 10 nm of silver coating (biological samples). In some cases we do not coat the samples at all. But all depends on detectors you have on your FEG and the mode of FEG SEM you use. **Oldřich Benada** benada@biomed.cas.cz
Wed Apr 13

My experience with chromium is that it seems to oxidize during deposition, such that freshly deposited films that are covered with a cap that blocks exposure to air are up to half oxygen when analyzed. Nevertheless, under HRTEM, they appear to have grain size so much smaller than any TEM specimen thickness so the grains are not visible. Another way to get extremely fine grain size is to use ion-beam deposition rather than sputtering or evaporation. Osmium, whatever its merits from a grain size consideration, is hazardous, so you would need to implement a safety program around it, which could make it more trouble than it is worth. **John Mardinly** john.mardinly@asu.edu
Wed Apr 13

Do you really need a coater? As we bought our first FE-SEM, we asked ourselves the same question, and finally bought... nothing. Our FE-SEM is now more than 15 years old, and in 95% of the cases, playing with accelerating voltage, beam current, detectors and scan speed, it’s not necessary to coat. Our new FE-SEM is now not far from the door, but without any coater again. In the mean time I build a multi-purpose evaporator fitted among others with a magnetron source. I then bought an Ir target, to be able to try and in case it’s necessary. I use it from time to time for fabric samples, because I’m too lazy to follow the drifting of the low energy conditions for these samples. But they need only low mag and could be observed on a good W type SEM with the classic 20 nm grain size gold coating from our 30-year-old Balzers. The only coating which remains necessary on FE-SEM is carbon for EDX. Of course, as we work in material science (but often on oxides) and not in biology, our samples are probably different from yours. **Jacques Faerber** jacques.ferber@ipcms.u-strasbg.fr
Thu Apr 14

The systems we used at Intel were the VCR IBS, and later, the Gatan PECS (still available). These systems use an argon ion beam striking a target of your choice, and the material sputtered from the target lands on the specimen. The resulting film has an extremely fine grain size. I believe there was a description in one publication of Ron Anderson at IBM using a Gatan duo-mill to do ion beam deposition by putting the target where the TEM specimen should be, and putting the actual specimen ~1 cm away with a custom made fixture. **John Mardinly** john.mardinly@asu.edu
Thu Apr 14

I would agree that a coater capable of laying down a fine layer of Iridium is the least complicated solution, albeit not the cheapest due to target cost. We install a lot of coaters with Iridium targets for this very purpose (Cressington 208HR). Anyone that would like a copy of an internal study on coating resolutions, please email me directly. You can also have a look at this comparative study: A comparative study of thin coatings of Au/Pd, Pt and Cr produced by magnetron sputtering for FE-SEM. *Journal of Microscopy*, Vol 189, Pt 1, January 1998, pp 79-89. **Mike Toalson** mike_toalson@tedpella.com
Mon Apr 18

You may be interested in checking out the prices on this web site. I remember Pt far outpacing Au back some years. I suppose Ir was even pricier. Now, Ir comes in at \$525/troy ounce, Pt comes in at \$981, and Au comes in at \$1242 – the most expensive metal in the table. I suppose that is a supply and demand thing and that many individuals have turned to investing in gold in recent years. Ir and Pt apparently don’t rate. <https://apps.catalysts.basf.com/apps/eibprices/mp/> **Warren Straszheim** wesaia@iastate.edu
Mon Apr 18

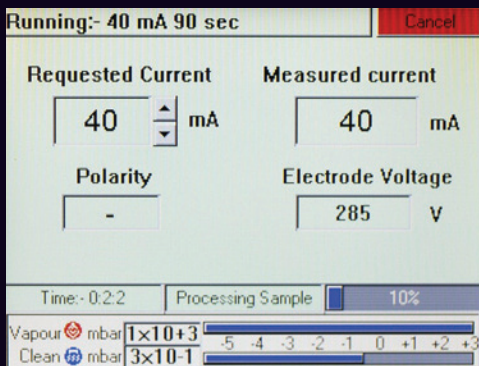
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Clean Chamber



Vapor Chamber



Instrumentation: lubrication

Does anyone have advice about lubricating lab instruments? We often have controls, gears, sliding blocks, shafts that seem to need a little help. Nothing like a big overhaul or major service, just a little tightness. I know about the need for special lubricants in specific places, but do you think it would be OK to try to loosen up some things by applying lubricants as needed? Have you found anything that seems to work well and is compatible with scientific applications? **Jon Krupp jkrupp@deltacollege.edu Wed Apr 6**

We used molybdenum disulfide paste for such jobs in our surface science lab. No vapors and no spreading, like oils do. Also works for lubricating nuts and bolts that might seize after equipment baking. Comes in little tubes. **Larry Scipioni les@zsgenetics.com Wed Apr 6**

I've found a small amount of white lithium grease works for sliding surfaces of focusing racks, moving blocks in microscope heads for picking camera/eye, things like that. Even used a bit of Braycote, a true high-vacuum grease, to stop the EDS on our STEM from screaming as it inserted/retracted. A *tiny* bit—stuff is hideously expensive. **Phil Oshel oshel1pe@cmich.edu Wed Apr 6**

EM:

historical cost of electron microscopes

I have a question posed by a colleague, which I think is better answered by the experts who read this. Here is the question: "What do you know about the pricing of electron microscopes in the early days? NMR started to get really expensive in the late 1970's with the development of bigger and bigger superconducting magnets. I am guessing that EM reached the same price points earlier. Could you venture a guess about when it was that a commercial EM was first on the market for more than a million dollars?" As a bonus question (from me), does anyone know when the first high-voltage microscopes came on to the market, and what their cost was? I'm writing from the MSA submission form because I am unable to get through using the regular connection. I am subscribed so will see any messages posted to the list. **Paul Webster paulwebsterphd@gmail.com Wed Mar 30**

I have been in the commercial end of EM since the late 70's. I have worked for 4 different column companies and three EDS companies. The price of a scanning electron microscope in the late seventies ranged from \$15k for an ISI Mini SEM to \$125,000 for a full featured SEM. TEMs were about \$75,000 for a biological unit and \$175,000 for a Philips 420 type System. In the 80's the entry level SEMs were about \$35 to \$50,000. High end SEMs were still about \$125,000. The onset of technology changes (Field Emission SEMs and intermediate voltage TEMs) in about 1985 correlated migration of semiconductor companies to routine EM use. This saw the price of FE SEMs and IM TEMs to rise steeply. I was in sales for Philips in 1989 and a CM20 was about \$400K while a CM30 was \$500K. With the addition of STEM, EDS, and EELS, they could approach \$900,000. On the SEM front, Hitachi took advantage of their early lead in field emission SEM by offering them at about \$250,000. Prices rose slowly through the 90s. I believe the first \$1 million EMs were full 200 mm wafer inspection and CD SEMs. TEMs broke the barrier in the mid-nineties with the introduction of Field Emission TEMs. By the end of the nineties dual beam FIB systems were over \$1,000,000. FE SEMs were up to \$400K and TEMs started to hit the \$1.5 million mark with the introduction of the Tecnai F30 and JEOL 2010F. Now we see prices jump again in the early 2000s with the introduction of aberration corrected TEMs at 2 to \$4 million. FE SEMs were up to \$450K and a new generation of mini SEMs took hold at about \$50,000. Prices have risen for newer technologies in the 2010s, while the cost of a full featured floor standing SEM has remained at about \$150,000. Interestingly, the price of EDS has

stayed at about the same for 30 years: \$45K. **Joe Robinson joetherob@gmail.com Wed Mar 30**

EDS systems seem to me to be going up as well. I won't name companies, but I paid just over \$65k in 2011 for the EDS system on my SEM, but was quoted \$93K (after discount) for the EDS on my new S/TEM, and was recently quoted \$147k+ for an EDS system on a FE-SEM. Some of this increase must be due to the change to SDD detectors from Si(Li) detectors, but not all of it. **Phil Oshel oshel1pe@cmich.edu Thu Mar 31**

Besides the hardware upgrades over the decades, let's remember the software to run it all. Software is a big part of the package—who does not expect a good user interface? **Roseann Csencsits roseann.csencsits@schafercorp.com Thu Mar 31**

According to my old advisor, Wil Bigelow, the JEOL 100CX TEM/STEM that I used for my thesis cost \$300K back in 1977. Seemed like a lot of money back then. **John Mardinly john.mardinly@asu.edu Thu Mar 31**

In the early 1960s I purchased a JEOL JEM6A TEM that operated at 80 and 100 KV and was fully equipped (rotating-tilting stage, heating stage, tensile deformation stage, and even a Cine camera) for \$35K. It had a guaranteed resolution of 20 Å, with a stated potential of reaching 10 Å under ideal external conditions. This microscope served the teaching and research needs of the graduate students and faculty of our department for thirty years. This was one of the first TEMs to compete with the Siemens TEMs. Hitachi made a similar instrument that sold for a similar price. At about that time RCA sold a Model EML TEM that operated at 50 kV, and was specifically designed for biological applications, at a much lower price. **Wil Bigelow bigelow@umich.edu Thu Mar 31**

When I started at Wool Research Organization, New Zealand in 2003 (now AgResearch), we had a gorgeous Philips EM300, which had been bought new in 1968. It kept working (with help in the form of some organ transplants from friends at The University of Otago) until 2006 when we decommissioned. I have the brochure replete with photographs of an attractive young (female) scientist peering devotedly into the instrument and changing the Wehnelt assembly etc. It was a top range 80-100kV TEM of its day. The handwritten notes on the brochure margins indicate \$41,000, presumably New Zealand Dollars (newly introduced the year before in a change from Pounds). When introduced a NZ\$ was apparently valued at ½ an NZ pound (which had been pegged to the British pound). Educated guess is therefore about GBP20,000 for the instrument back in the day. Hopefully that stacks up. **Duane Harland duane.harland@agresearch.co.nz Thu Mar 31**

The JEM-6A was pretty old TEM. I used JEM-6C until 1985 for observation on biology samples. It used many vacuum tubes and big resistors and we had to put those things in separate room. In 1986, we bought a JEM-1200EX with ASID10 (SEM/STEM attachment). I paid \$450K. **Vincent Wang vincent.wang@schafercorp.com Fri Apr 1**

The ANL AAEM (VG HB603Z) the first 300 kV CFEG DSTEM/CTEM/AEM was purchased in 1984, it's price tag was ~ \$1.5M. The instrument included: STEM, TEM (yes TEM in a VG!), SCEM, SEM, XEDS, EELS, AUGER, SIMS, LEED, Prep Chamber, TF Evaporators, High Pressure Gas Reaction Cell, CCD imaging/diffraction camera, Hot/Cold/RT/Be and double tilt holders. After 20+ years, the instrument's "last light" was on Nov 19, 2008 and at decommissioning it was still operating at 300kV. Its parts were redistributed and still populate several research instruments, some of which are not electron microscopes. **Nestor Zaluzec anl.nestor.zaluzec@gmail.com Fri Apr 1**

Yes, they were a lot cheaper when half the price of a microscope was not add-ons from Gatan. Of course, they did a lot less. **John Mardinly john.mardinly@asu.edu Fri Apr 1**



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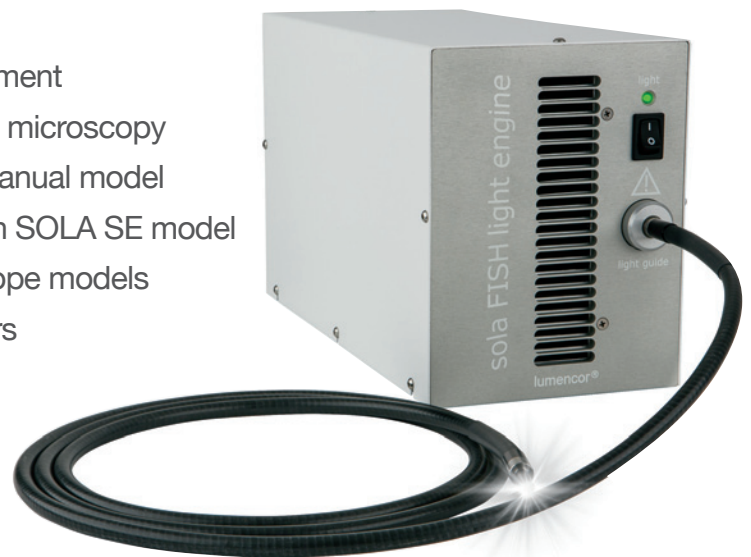
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TEM:**LaB₆ filaments**

*The huge issue I have been having for the past couple of years is the quality, reliability, and life span of my LaB₆ sources. Now this is not an issue with just one scope or just one vendor. Tungsten filaments work fantastic and hit all the numbers perfectly in both of our 120kV Tecnais. When we put a LaB₆ in, really weird things happen. Before you get suspicious and think we just started up with LaB₆, this is all that we have ever used on our tools, I just had to test the scopes with W to make sure I wasn't imagining things. We need LaB₆ because of the brightness and hours of use; each scope is used 40-50hr/week. So much for the introduction, now on to the issues: 1. I have not seen a Maltese cross on either a Kimball or Denka in 5 years or so, or about 5- 8 filaments, on either of my Tecnais. The engineers and I just undersaturate and go for max brightness. 2. Filament life used to be 2500-3000 hours; the last five tips have flamed out around 500 hours. BTW the vacuum has been rock steady in both scopes. 3. I have had two tips fail due to micro cracks in the carbon crucible. 4. The last five tips drift for the first 100-200 hours, and we are talking X-Y and Z. This is to the point that the deflectors can't compensate and I have to break vacuum and re-center manually. 5. My 24-Volt power supply for the filament is fine, swapped them around my scopes with the same result. If the darn things didn't cost nearly \$900.00 I might tolerate this. I have to give high marks to EMS, they have been listening to my complaints and have made good on the tips that have failed out of the box. But the quality of the product I really have questions about. Has Kimball changed their carbon supplier, is it softer? Why are there cracks? And the last question is: have any of you guys had the same or similar issues? And the absolutely final question is am I missing something? After 30+ years of ripping scopes apart I might just have lost the magic touch or there is a vortex of LaB₆ instability issues in Ithaca. **John L. Grazul jlg98@cornell.edu Wed Apr 20***

We've had similar issues with the LaB₆ filament image (odd shape) and used the gun tilt to max out the screen current as an alignment technique. We also have the filament image moving across the screen when saturation is being approached. But the latter is what I've been told is due to some dirt on the final anode or some nearby electrode perhaps in the accelerator. This wandering is happening with W filaments too and is less critical at lower magnifications. I went to put a new LaB₆ filament in the other day and it just broke off, much to my embarrassment and I'm putting off buying a new one for a while. Our microscope is a JEOL 2000FX and vacuum isn't a problem so long as we replace the ion pump every five years. The new one that broke while I was putting it in was a KP (the Denka are welded and robust). The thing that has my blood boiling is the so-called long-life FEI gallium sources that don't last more than three months. And as you may know they come in at over \$2000 each, as do the expendable extractors similarly priced. The one we got with the new install lasted very nicely, but since then not so much. **Rob Keyse rok210@lehigh.edu Wed Apr 20**

I've had some issues over the past several years with the Kimball Physics LaB₆ cathodes. I'm not sure which brand you are using. After meticulously setting the depth and centering the cathode, I run out of gun tilt when warming it up. After pulling it back out of the scope, I find that the cathode is still perfectly centered in the Wehnelt. I often go through 3 or 4 iterations before I can get a usable setting of the cathode, each time finding that it looks perfectly centered outside of the scope. My suspicion is that the legs have slightly different resistances and that one leg warms up more than the other pushing the cathode to the side. When the cathode cools back down, the tip re-centers itself. This theory is pretty hard to verify since I can't directly observe the tip while it is warm. If I can get the tip centered enough I do see the "Maltese cross" figure though sometimes it's a bit off center.

One thing that may affect your lifetime is the filament limit for saturation. I have noticed that if I initially set the cathode to be just saturated, then check it 20-30 minutes later I find I can back the filament drive off by 1 or 2 clicks and it is still saturated. I assume that as the components warm up, the saturation point changes slightly. I do have to tell the students that this is the correct way to operate so that they don't reset the limit themselves. So, I now set my filament limit to be at least one click undersaturated. This way, as it warms up, it will bring itself to the proper saturation point. Note that the Kimball Physics documents indicate that the evaporation rate will triple in going from 1800K to 1850K. This will bring the lifetime from ~2000 hours down to a 600-700 hour range. If the tip isn't oxidized by one of the students, I can still get close to 2000 hours from a cathode. I did have one student destroy one in 20 minutes by letting the cold trap warm up while it was hot. Kimball Physics has some useful info in the technical documents on their web site. **Henk Colijn colijn.1@osu.edu Wed Apr 20**

Sorry, my experience is only with Tungsten but what about if you position the cathode a little bit shifted to the opposite direction you suspect it shifts after warming. So you may correct the problem and verify your theory at the same time. **Yorgos Nikas eikonika@otenet.gr Wed Apr 20**

We too have been having the same problem with Denka LaB₆ filaments on a JEM-3010. We have taken the last two out after aligning them because they appear to be tilted only to find that they are perfectly aligned! Over time the last one got better and we got closer to the Maltese cross. We have not seen a sharp decline in lifetime however. **Alan Nicholls nicholls@uic.edu Thu Apr 21**

We too have had problems with Denka LaB₆ on our FEI T20. Short life and bad drift. We have to take the Wehnelt out several times to re-center the LaB₆ as it runs out of gun tilt. We are experimenting with whether to just turn down the filament current for overnight or if it is best to turn it off completely. After a long spell of just turning it lower we are now turning it on in the morning and off at the end of the day. Its early days but I think we have better lifetime. Next time we change filament we have plans to try a different cathode assembly. **Chris Gilpin gilpin@purdue.edu Thu Apr 21**

LaB₆ users, I am not losing my touch and there is no LaB₆ death vortex hanging over Ithaca! I have a whole slew of responses that concur with the issues I have been having. If any of you who are having the same issue please get in touch with me because if we don't tell the vendors and the suppliers that their product has changed for the worse they will still keep selling us garbage because either they don't know or just don't care. By the way, not one retort from Kimball or Denka... yet. I think all we want is a LaB₆ that shows us a Maltese Cross, doesn't drift X-Y & Z to the point that the deflectors can't compensate (this is a common issue, you take the gun out and the tip still looks perfectly centered, you apply a current and it tilts toward the Emerald City), and a long life; just the way they used to be ten years ago. I can't make it to M&M this year or next, that's when the ankle bracelet comes off, but this may be a good topic for a round table because we the users are not imagining things. Send me some more horror stories, I will compile them and post them or you guys just throw them up on the Microscopy list, do not respond to Phil, I may owe him a keg if you reply to him. **John Grazul jlg98@cornell.edu Thu Apr 21**

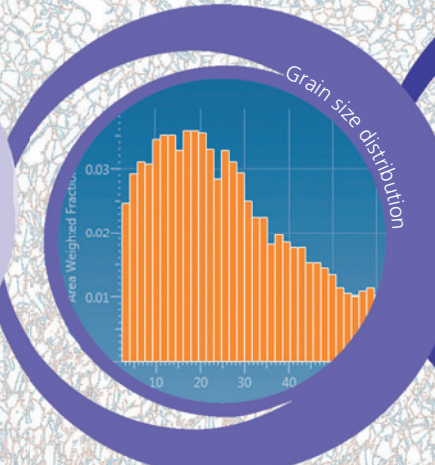
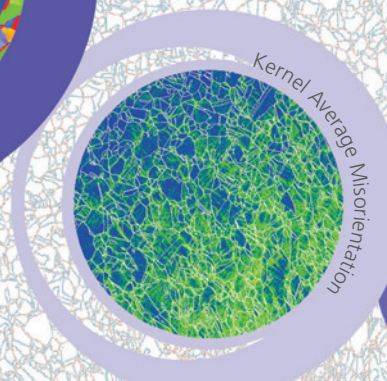
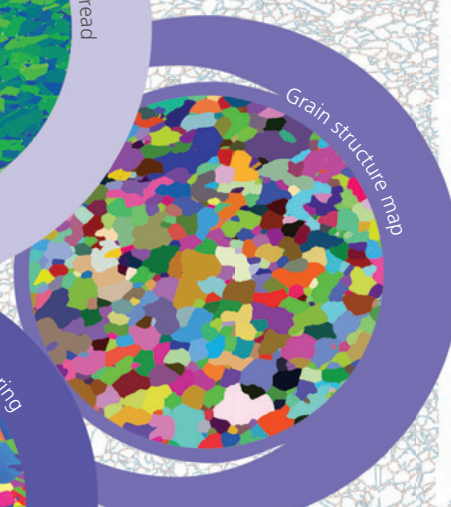
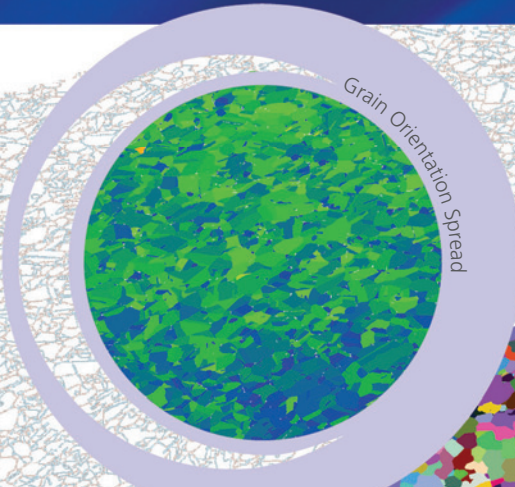
LaB₆ enthusiasts! This afternoon I am looking at 4 Kimball filaments in the FIB. Some had a dignified normal death, others an untimely and less dignified death. What we are going to look at is the elemental differences between them, from the La right to the base. We are going to do slice and views and look for defects. Is there anything else you guys want us to investigate? Let me know and your dreams will come true, we are the

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Measurement	Minimum	Maximum
Area (μm^2)	3.60	20,326.3
Aspect Ratio	1.09	1803
Breadth (μm)	0.60	184.11
ECD (μm)	2.14	160.87
GOS ($^\circ$)	0.22	19.14
Length (μm)	2.68	367.28
MOS ($^\circ$)	0.37	43.56
Perimeter (μm)	6.63	2,350.6
Slope ($^\circ$)	0.00	178

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Disneyland of microscopy. I have been contacted by one of the manufacturers; they too are interested in the data we get. So things are moving and hopefully we will be getting good product in the near future. Next on my agenda will be the quality and performance of FEG tips. How have your FEG tips been lately?? We at Cornell have tales to tell. Send me your tales of woe or post them, but let's just say our experience lately has been similar to our issues with LaB₆. Thanks for interacting, and if we don't keep the suppliers on their toes, our down time because of poor product will just be expected rather than a surprise. **John Grazul jlg98@cornell.edu Fri Apr 29**

SEM: desktop instruments

Do any of you have experience with the newer benchtop or desktop SEM's? They seem quite capable and their cost to purchase and maintain, compared to the full scale SEM's, looks attractive. Their advertised performance is appealing. Do they maintain that performance? Do they require much service? Should I have a service contract? Do the accessories function well (e.g. rotating and tilting stage/holder, charge reduction holder)? Is the provided EDS system comprehensive and reliable? Your replies would be greatly appreciated. **Tom Kremer tkremer@ipstesting.com Wed Feb 17**

I have a 100 × 100 mm stage in a FEI Quanta ESEM. EDS works—with some problems from Oxygen at 15 Torr with water vapor. If I did not need to serve a large group of users, I could likely do just fine with powders, particles on 12 mm circular stubs or even 7-8 mm lengths of curtain rod (Older small SEM's from a very reputable manufacturer) if the specimen was constant in dimensions—as in Quality Control. Service for us is justified by the patterns of use and the criticality of the work that is done. We have service contracts on both the ESEM and the EDS (this is unusual for an EDS). The reason is that faculty in biology, chemistry, and geology with laboratories have included individual student contact with the ESEM/EDS tool as part of laboratory schedule. For the EDS the service contract reduces a loss of detector from an 8-week turnaround to 1. Considering a SEM to be a tool, a careful list of requirements will likely lead to an easy answer to your question. Everything about a tool on a bench is about compromise. We have a 250 lb. “Benchtop” X-Ray Diffractometer with a strip-CCD detector that can provide 60K peaks in a 30 min scan over 20–125 degrees 2Theta—perfect for student laboratories. (~\$100–150k) There is no service contract even though we use it for student laboratories. In the XRD everything inside is modular: computer, controller and goniometer, source and detector. We also have a 250lb. benchtop vacuum coater that can't be put on a bench against a wall, because 75% of its maintenance is done thru the rear of the unit. For our uses—no contract. Cleaning and servicing the TMP is relatively easy. The control system is modular and can easily be diagnosed over the phone and countered by exchange by overnight mail and screw driver and cable changes. What I have seen of the small SEM with EDS is that data are the same - % by weight and atomic % + a couple others estimates. Currently, my only real consideration would be whether the benchtop I purchased had a SDD detector instead of the Si(Li) that all of us users want to replace with one. **Fred Monson fmonson@wcupa.edu Wed Feb 17**

FESEM: data logger

I have a user who wants to document everything he does while using our FEI Quanta 450 FEGSEM. A call to FEI Tech Support said they can log error messages, but they can't log other actions such as moving sample etc. My question is, does anyone have an app, or know a company that might sell such an app, that will enable my user to document all his actions on the scope? **Steve Barlow sbarlow@mail.sdsu.edu Mon Mar 21**

What you are asking for is called “FIB Assist” and was/is made by Fibics in Ottawa, but I doubt it would be commercially available for Quanta. For documenting FIB process development work or circuit edits I use stand-alone video recorder, configured to capture FIB screen at about three frames per second. Separate PC with Epiphan PCIe is my favorite solution, but you can get USB-pluggable version from the same guys or elsewhere: <http://www.epiphan.com/products/compare-pcie-capture-cards/>. No any affiliation with either Fibics or Epiphan. **Valery Ray vray@partbeamsystech.com Tue Mar 22**

Did he explain what his goal was in doing this? That would certainly help to know. Indeed, I do not go far in supporting unusual requests unless and until the user is willing to explain them. I have had too many occasions where what they wanted to do was actually quite available with another technique that they did not know about. The Quanta affords a movie option that can be programmed at whatever capture rate they want. Stage X & Y is one of the options for the databar so it could be used to track moves. I suppose there are some screen capture utilities that could render a movie of the whole screen showing the changes to other parameters. I hope it has a good level of compression. Most of the screen would not be changing from frame to frame and should not take up much of the data stream. **Warren Straszheim wesai@iastate.edu Wed Mar 23**

EDS: elemental analysis detection limit

I did EDS analysis by SEM and I was interested to find Pb, Cd and Zn elements but unfortunately my sample did not show peaks of any of these element. I used optimum parameters like 30 kV acceleration voltage and 10 mm working distance on S3500N Hitachi SEM with an Oxford EDS detector 10 mm² window. What is the detecting limit of SEM-EDS for Pb, Cd, and Zn in concentration? Is there a chart or any reference document where I can study the general detecting limit of EDS by TEM and SEM for all the elements? **Ravi Thakkar ravi.thakkar369@gmail.com Wed Mar 30**

Perhaps the easiest way to look at detection limits is to use one of the Monte Carlo spectrum simulation programs. There are a number of programs available but you might want to try: (i) Nicholas Ritchie (NIST) DTSA-II – <http://www.cstl.nist.gov/div837/837.02/epq/dtsa2/> or (ii) Raynaud Gauvin, Hendrix Demers (McGill Univ.) Win X-ray, MC X-ray - <http://montecarlomodeling.mcgill.ca/>. One caution about this software: they use the same models and databases as the standard-less quantification routines, so the same uncertainties apply to the simulation as to standard-less quantification. That said, they can be useful to get an idea of detection limits and experimental feasibility. **Henk Colijn colijn.1@osu.edu Wed Mar 30**

EDS is not the best to detect element in trace (if there is less than 1%). It's easier by fluorescence when the SEM has an X-ray source or by WDS. Anyway I think you can do better to optimize the conditions because 30KV is not suitable (except to see the L alpha of Pb). An empiric rule says that the best accelerating voltage is 2.5 times the energy of the peak you expect to analyze. This is because the ionization efficiency of an atom by an electron is nearly maximal when the energy of the electron is between 2 or 3 times the energy of ionization. Applying this rule you may get more photons on:

Pb M alpha near 5kV
Pb L alpha near 30kV
Cd L alpha near 9kV
Zn K alpha near 24kV

Zn L alpha near 3kV (but probably to low a voltage to get a good count rate. So you can try 5 kV). Once the accelerating voltage is

adjusted, increase the beam current to increase the count rate and acquire the spectrum over a long time (may be 300 seconds). **Nicolas Stephant** nicolas.stephant@univ-nantes.fr Thu Mar 31


I partially agree with Nicolas. XRF will be easier for detection since it won't have the same prominent background (Bremsstrahlung) that electron beam excitation has. Therefore, peaks will be more detectable. You should be using a lower voltage. The factor of 2.5 is a good rule of thumb. You should be able to see the Zn L line well enough with a thin window detector. I can hardly envision using a Be window detector anymore. I really like seeing the elements down to Be. I routinely depend on seeing the C and O lines. I spend most of my time at 10 kV anymore and sometimes drop it further. However, I think you should be able to see peaks corresponding to a tenth percent or so by EDS. The limit will be how well the background is defined. I routinely collect at 15–20kcps for a minute. I will count longer in critical situations where I need to improve the detection limit. **Warren Straszheim** wesaia@iastate.edu Thu Mar 31

Nice answers...but nobody asked Ravi what precisely is his sample, and this is essential information for a sound answer! First, the optimum X-ray line and beam energy will not be the same if the sample is a bulk homogeneous material or a thin (how thin) film on a bulk substrate or (micro/nano-) particles on a bulk substrate or even a thin foil like for TEM. To be sure that you analyze the volume containing the elements you are interested in and only that volume you should run a Monte-Carlo simulation. Second, the nature of the matrix may also influence the choice for X-ray line once you take the absorption consideration and possible overlap with adjacent lines from other elements. **Philippe Buffat** philippe.buffat@epfl.ch Fri Apr 1

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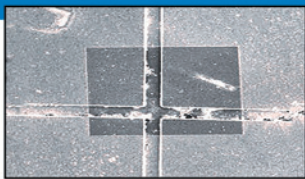


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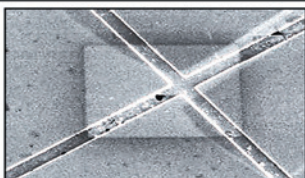
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