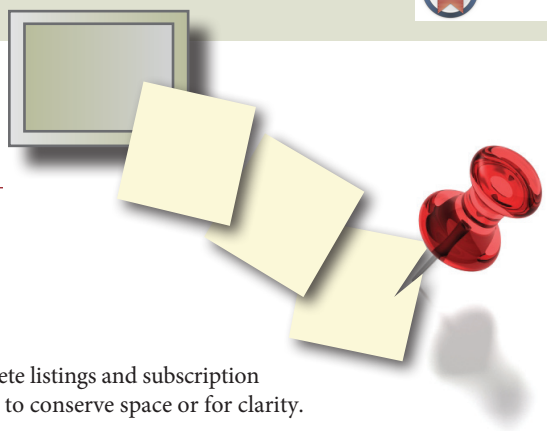
https://doi.org/10.1017/S1551929517000967 Published online by Cambridge University Press



NetNotes

Edited by Thomas E. Phillips
University of Missouri

phillipst@missouri.edu

Selected postings from the Microscopy Listserver from July 1, 2017 to August 31, 2017. 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:
bubbles in resin

I usually cure my EMBED 812 resin in a vacuum oven at 15 mm Hg at 60°C. The oven had failed; facilitating a new purchase. After balancing the temperature, I put some test blocks in without tissue to test the polymerization and cutting post cure. To my dismay, I have been having bubbles form in the capsules (BEEM 00), and they occur along the edges from the bottom to the top of capsule. It does not matter where I put the trays, or the number of capsules to polymerize. I have varied the vacuum from 10mmHg (bubbles are finer and worse) to 20 mm Hg (bubbles form at the bevel to the tip and are slightly larger in size). I have yet to vary the temperature. I've been using vacuum ovens for over 16 years and have never had any problems before and I'm a bit stumped. ***Mike Ganger mtg2003@med.cornell.edu Sat Jul 8***

I have not had to use the vacuum part of my vacuum embedding oven for the past couple of decades when I remember to do one simple thing: I preheat (degas? de-water?) my molds, capsules, and paper labels in the oven for several hours or, preferably, overnight before use. It may be a Hawaiian humidity thing, but I have not had any bubbles at all whenever I've done this. Give it a try! Tina (Weatherby) Carvalho tina@pbrc.hawaii.edu Mon Jul 10

No need for vacuum here either. We preheat the beems and the samples while in resin for 5–10 minutes before embedding and make sure the oven is at a stable 64 degrees. So far no serious issues. Nick Madary joseph.n.madary.civ@mail.mil Sun Jul 16

Specimen Preparation
resin expanding on water

I'm having a specific problem while trying to section a Flat embedded sample (in glass bottom Petri dishes) in epoxy resin. When the section hits the water, the resin expands, and keeps expanding until it reaches almost 3-4 times the size of the block face. In addition, the sections are quite sticky as well, making it impossible to collect them (they stay glued to the eyelash).I have already processed samples before without a problem, but for the past months it has been the same problem. I do not think it's the resin because cell pellets embedded in the same resin can be sectioned normally. ***Leandro Lemgruber leandro.lemgrubersoares@glasgow.ac.uk Tue Aug 1***

It sounds like an incomplete infiltration of your resin. Normally for cells on coverslips, we do an overnight in pure Epon (after complete a dehydration series), no ethanol: Epon parts. The next day we do three 2-hour changes of fresh resin (rock, 15 psi vacuum, rock), then cure the resin/coverslip to a pre-filled BEEM capsules and bake 60°C overnight. The following morning we detach the coverslip from the polymerized block by submerging in liquid nitrogen. The coverslips fall off within 5-10 min. Cells are at the BEEM capsule surface. Michael Delannoy mdelann1@jhmi.edu Tue Aug 1

Specimen Preparation:
molecular sieves for dehydration

Are any labs out there that use molecular sieved 200 proof ethanol for dehydrating TEM samples? Do you prewash them in ethanol and then bake out before use? I used to do this but switched to the pint-sized bottles (opening up a new one for each new experiment). The problem of course is generating too many bottles of unused ethanol. All comments are welcome. If you do use it please forward a catalog number and vendor. ***Michael Delannoy mdelann1@jhmi.edu Thu Jul 20***

I have used molecular sieve in 100% ethanol for years, it works well. I use mSorb sieves, and they are packaged to be used as received - that is, no pre-baking before use, nor do they need washing in ethanol before use. Which is good, because unless the ethanol is absolutely anhydrous, some of the water capacity of the sieve would be used up in the ethanol wash, and then you would have to pre-bake. Just watch the dust - be careful getting ethanol out of the bottle with sieve in it, or put the sieve in dialysis tubing. I ordered these from Delta Absorbents, Cat #MS3AEDG05 in 5 gal pails (much cheaper than buying from lab supply or EM supply companies) and MSBI4A4801 for the blue indicating sieve (1 lb. packets). The indicating sieve is *much* more expensive, so buy a separate packet and mix it 1:5 or 1:10 with the non-indicating sieve. Phil Oshel oshel1pe@cmich.edu Thu Jul 20

Many thanks to all your quick responses to my molecular sieve questions. I think we will give Phil Oshel's protocol a shot: Order a 5 1b sieve and 1 lb. indicator sieve, mix 1:5. From Delta adsorbents - Ready to use, no washing or baking required (I like that). Michael Delannoy mdelann1@jhmi.edu Thu Jul 20

Residual molecular sieves will kill your diamond knife if it makes it into your block. Known problem with using this to dry ethanol. Al Coritz acoritz@emsdiasum.com Thu Jul 20

Not a problem I've had, but then it's just a matter of being careful to not stir up the sieve when in ethanol - or of putting the sieve in dialysis tubing. Phil Oshel oshel1pe@cmich.edu Thu Jul 20

Specimen Preparation:
HRTEM cross section imaging of plate-like nanoparticles

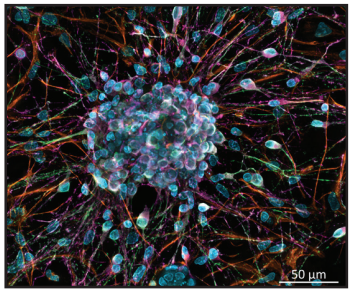
Plate-like nanoparticles (2–5 micron diameter, 20–100 nm thick) have been prepared. Plan view was examined by the usual powder dispersion drop cast on a holey-C film method. But, we now would like to look at the cross sections of these particles to study how the atomic layers stack and grow into a plate. The sample comes in water suspension. Two questions: 1. Sample prep for cross sections HREM. How do we prepare TEM specimens for high-resolution atomic imaging of the cross sections of these thin nano-plates? 2. Good statistics. How can we maximize the number (~30 or more) of the nano-plates in one specimen? ***Z Zhou z.zhou@lboro.ac.uk Wed Aug 30***

You may consider JEMS from P. Stadelmann. Have a look to http://www.jems-saas.ch/ that also contain the access to the limited

Adding efficiency to your fluorescence imaging.

ZEISS Celldiscoverer 7

// INNOVATION
MADE BY ZEISS



Rat cortical primary culture. Sample courtesy of Dr. Holger Braun, LSM Bioanalytik GmbH.

Your automated platform for live cell imaging

Imagine the ease of use and automation of a boxed microscope – combined with the image quality and flexibility of a classical research microscope. Imagine this system calibrates itself, detects and focusses your samples and adaptive optics adjust themselves automatically. ZEISS Celldiscoverer 7 is your reliable automated research platform. No matter if you work with 2D or 3D cell culture, tissue sections or small model organisms.

www.zeiss.com/celldiscoverer

ZEISS

"Student version" for demo on Mac, PC and Linux. This software is a comprehensive program for (HR) TEM+STEM image simulation and electron diffraction (SAED, nano-diff, CBED, Kikuchi, precession, powder patterns) interpretation and simulation based on Bloch waves and multi-slice approaches. In the present case, feeding JEMS with files containing the crystal parameters for all suspected phases allows an automatic match with the experimental patterns for phase identification. Disclaimer: P. Stadelmann and myself were working in the same laboratory for long though on different subjects. Philippe Buffat philippe.buffat@epfl.ch Fri Aug 18

We faced a similar problem trying to look at the cross-sectional structure of hard nanowire heterostructures (See Jiang et al., *Nanoletters* 13 (2013) 5135 and Zheng et al., *Nanoletters* 13 (2013) 3742). In the end we borrowed a page from our biological colleagues and embedded and microtomed them! Surprisingly successful, you will have to do this very carefully at liquid nitrogen temperatures with a diamond knife. If you want the details of microtoming hard materials look up the papers by Selwyn Glanville. The biggest struggle you will have is getting your plates to line up and disperse for the embedding (easy with epitaxially grown samples). This will also give you many plates in cross-section, so plenty of statistics. Matthew Weyland matthew.weyland@monash.edu Wed Aug 30

Microscopy:

data generation trends

I'm looking for a good reference that describes the rapid increase in data being generated in the microscopy field. With the advent of direct detection, we are now generating vast amounts of data that require new approaches to handle them. I am wondering, is there an article that describes these trends over time? ***Steven Spurgeon steven.spurgeon@pnnl.gov Tue Aug 15***

This would indeed be a very timely topic. For scanning probe microscopy, there are papers: Big, Deep, and Smart Data in Scanning Probe Microscopy (2016) ACS Nano 10(10):9068–9086 and Big data and deep data in scanning and electron microscopies: deriving functionality from multidimensional data sets in (2015) Advances for Structural and Chemical imaging 1:6. There are also some DOE documents, for example: https://science.energy.gov/~/media/ascr/pdf/programdocuments/docs/ascr-eod-workshop-2015-report_160524.pdf. They partially address data generation in physical sciences including electron microscopy. Sergei Kalinin sergei2@ornl.gov Tue Aug 15

LM:

comment on resolution

From the lectures and discussions at the recent Three Dimensional Electron Microscopy Gordon Research Conference (Les Diablerets, 2017), I noticed there are still serious misunderstandings – even among distinguished professors in Physics and in Biology - on what "resolution" actually means. So, allow me to go over the basic principles: 1) The instrumental resolution of an imaging system is given by the physical properties of the microscope, telescope, photographic camera or whatever your favorite 1D-, 2D-, 3D-, 4D-imaging device is. The classical case would be that of a light microscope where the numerical aperture (NA) of the objective lens (https://en.wikipedia.org/wiki/Numerical_aperture) determines the "instrumental resolution" of the microscope (https://en.wikipedia.org/wiki/Angular_resolution). In the case of a diffraction-limited telescope it is the diameter of the main lens that determines the instrumental resolution. In the old days of electron microscopy, one would often see the first zero of the CTF being used to define the instrumental resolution of the microscope. 2) The resolution achieved for the results based on the images collected is a very different issue! Suppose, for example, you forget to switch on the illumination of your light microscope! What good will then the high-resolution (high NA) properties of your expensive instrument do you? If, on the other hand, you do switch on the illumination but only use a very low dose of ~ 10,000 photons to generate an image, that image will be very noisy. How much better will the image of your object be if the image is created accumulating a total of 10,000,000,000 photons? The underlying question is: How do I define a results-oriented quality metric that reflects the image information I have collected in an experiment rather than what a specific instrument can potentially collect? The basic idea is to take TWO images of the same object rather than just one. Both images will contain the same signal (the object) but a different realization of the random noise so we can then compare the two images to each other in Fourier space using the FRC (Fourier Ring Correlation). This suggestion first emerged in single-particle EM in the early 1980s (https://en.wikipedia.org/wiki/Fourier_shell_correlation). Strangely enough, it took decades for the rest of the imaging scientists to realize what they were missing. Only very recently "everybody" suddenly started using the results-oriented FRC and FSC metrics in many other imaging fields, including X-ray microscopy, X-ray crystallography, light-microscopy, X-ray tomography, scanning microscopy, astronomical imaging, etc. Instead of claiming "super resolution" by showing some nice images from a given microscope, one can now just prove it through an FRC/FSC curve. I never understood why it took everybody so long to adapt to this straightforward gold-standard metric. Take home lesson: the "instrumental resolution" is the intrinsic resolution that the instrument is capable of, whether you actually use it or whether you leave it in the cupboard. The statistically significant "results resolution," on the other hand, reflects the quality of the final results achieved within a given data-collection experiment. These are two very different concepts! ***Marin van Heel marin.vanheel@googlemail.com Sun Jun 25***

TEM:

electron beam brightness

We have an electron beam brightness issue with our JEOL JEM-1010 after filament change. The electron microscope filament beam, which is not sharp/bright enough (shadow of the fixed aperture) after I replaced the fused filament with a new one. In the past, we have done this practice multiple times without any issue. I have done all the possible filament distance-adjusting options with the Wehnelt cap/cover but no major difference in results. Furthermore, obtaining the filament saturation point did not yield a converged single very sharp point. When we desaturate the filament by turning the filament knob counter clockwise, it is not producing the typical image of two balanced half crescents circling a round spot inside. Without engaging any lens aperture, the shadow of the fixed aperture in causing a hollow zone like double spot with slightly missing the full overlap. Is it possible to post an image or video clip of the same for better understanding? ***Muhammad Javed Iqbal mj_iqbal@yahoo.com Fri Aug 11***

The main thing is to adjust gun tilt to get good beam image emission. With filament at 100 kV, it must be around 20 μA add to 66 μA of HT. Set filament knob around 9 o'clock with bias adjusted around 5 to 7; adjust gun tilt *x* and *y* to get maximum brightness. Michel Ribardière m.ribardiere@jeol.fr Fri Aug 11

I see your problem which you described very clearly. The error that you are making is due to making a standard error when aligning an instrument. When we start learning to operate a TEM we worry about the gun alignment and we often make our own problems.

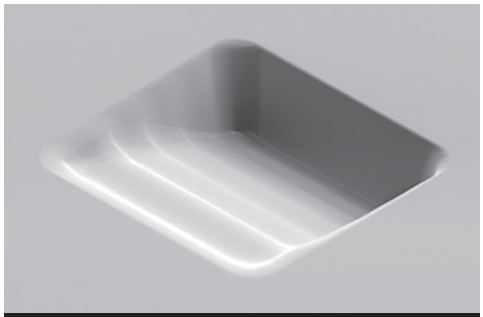
TESCAN S8000G
and novel Orage™ FIB column

TESCAN
PERFORMANCE IN NANOSPACE

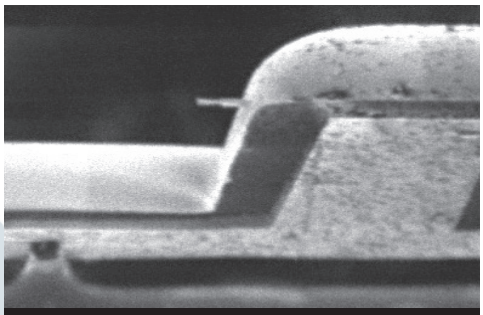
Anticipating the future, expanding your possibilities today

Orage™ FIB column: Prepare for the new level in sample preparation

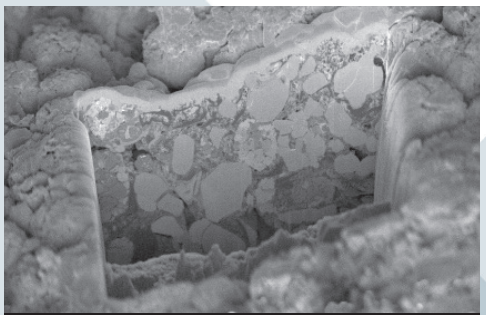
- Next generation of Ga source FIB column and a guarantee of world-class quality in sample preparation
- Cutting-edge ion beam optics delivering improved resolution at low beam energies for damage-free ultra-thin TEM specimens
- High ion beam currents up to 100 nA enabling fast sputtering rates for maximum throughput and minimum time-to-result



Cross-section 50 µm wide prepared with an ion beam current of 85 nA.



1 keV FIB image of a lamella on the TEM grid.



50 µm-long cross-section prepared in a Li-ion battery cathode

The tendency is to see a spot of light and to work on that spot of light, but if it does not respond correctly, this is a shadow! You need to forget the spot of light that does not behave correctly and find the true beam which will behave correctly; your gun is out of alignment when it displays this shadow. Use your gun alignment shift to find the beam and then gun alignment tilt to bring up the spot and halo that you are familiar with. I suggest you reset all of your gun alignment controls and start again. Steve Chapman protrain@emcourses.com Sat Aug 12

SEM:
using instrument in a glovebox with argon

Looking for guidance on the use of an SEM, or any electronic instrument for that matter, in a glovebox having an argon atmosphere. After hours of extensive searching, I have not located any experience with two reported concerns: 1) that there can be issues with arcing of circuit boards or exposed electrical components. 2) That motors are subject to overheating - probably only for open motors that are cooled by the air, but how would a sealed Turbo Molecular Pump fare? 3) Argon's breakdown voltage is 20% of that of Air. What effect would this have inside an electronic device like an instrument or SEM? ***Mike Toalson miketoalson@gmail.com Thu Aug 3***

More problems: 1) Heat transfer - argon has about 67% of the thermal conductivity of air at normal pressure making overheating of electrical components quite possible. Watch your thermocouple and Pirani vacuum gauges for starts. Forced cooling (as in motors with fans) won't help much because specific heat of Ar is about half of that of air. 2) Breakdown voltage of argon is 0.2 of air. Circuits with voltages above low ones (5 / 12 / 24 V) will be at risk. More points? Vitaly Feingold vitalylazar@att.net Thu Aug 3

There would definitely be problems with high voltages in an argon setting, but the question I've got is why put the SEM in an argon glove box, instead of using a gas-chamber transfer system between the glovebox and the SEM? The specimen itself is in a vacuum in the SEM, so the environmental sensitivity shouldn't be an issue. Justin Kraft kraftpiano@gmail.com Fri Aug 4

This will probably not end well if you try it. Anything high-voltage will not be happy. I don't think the motors will care too much but the turbo pump will probably need a water cooling loop added. I would look at getting/building a load lock for the SEM and only having that exposed to the inside of the glovebox. Jerry Biehler jerry.biehler@gmail.com Fri Aug 4

Did I miss the original post as to why this needs to be done? Does there need to be manipulation of the sample in the glove box whilst imaging? Why add an SEM to a glove box? My thoughts are heading towards using an ESEM with argon gas in the chamber. There are nano manipulators too. Chris Gilpin gilpin@purdue.edu Fri Aug 4

Thank you so much to all in the community for providing some great comments and suggestions. Some have asked why this was being asked about. Our company is a distributor of tabletop SEMs and we have a project where a customer with another type of SEM wishes to have a system inside an argon filled glovebox for analyzing samples that cannot be exposed to air. Like many of you, my first thought was "why not use a transfer device like the Quorum accessory?" However the type of SEM being considered (not ours) has no external port so the solution for them is to put the entire compact SEM in the glovebox with a special modification kit. Our system has 2 "boxes" with extendable cables to the electronics box so only the column and HV supply would be in the glovebox. Thus, I was curious about experiences with this. I do like the one idea of using a cold plate to keep the components from overheating. More study is needed though and the use of a transfer system seems less complicated for sure however, our system only has one side port that is normally used for an EDS but we are looking into modifications. Curious if any of you have looked at using something like the small transfer device shown here with the "rupture" film? My first concern would be does the film stay intact after splitting open but the paper describes using elastic films that are not prone to this. It seems like a simple and elegant way to move a sample from glovebox to SEM without the need of an Airlock or complex shuttle.www.creol.ucf.edu/research/publications/5296.pdf<http://www.creol.ucf.edu/research/publications/5296.pdf> Mike Toalson miketoalson@gmail.com Mon Aug 7

Here are two other alternatives to a SEM in a glovebox: http://www.int.kit.edu/downloads/INT_Research/Flyervacushut.pdf and https://www.kammrath-weiss.com/en/products/materials/transfer-module.html Jim Quinn jquinn11733@gmail.com Mon Aug 7

SEM:
tungsten filament

Please help me formulate a stance on a debate about the effects of an SEM's Tungsten filament breaking when they fail or "burn out". One vendor is raising the alarm that if a user waits until a tungsten filament breaks or fails prior to replacing it, that they run the risk of experiencing a much bigger cleanup issue and even having "shards" from the filament passing over to the Turbo Pump turbines and causing damage. I have seen plenty of burned out Tungsten filaments but never experienced these issues. Of course, the Wehnelt or Anode can often need a good cleaning, which is why most labs have some Pikal or Wenol paste handy. Other than the interruption to replace a filament, has anybody with a Thermonic tungsten filament SEM ever have a major problem from the filament burning out? ***Mike Toalson miketoalson@gmail.com Thu Aug 3***

Tungsten filament won't cause any problems beyond periodic routine maintenance (cleaning) of gun components unless electrical malfunction turns it into a photo-flash all the time, which is extremely unlikely. Would not worry about that. Vitaly Feingold vitalylazar@att.net Thu Aug 3

Would this vendor by any chance be a vendor of filaments that he wants to sell you? This sounds like a scam. Tungsten evaporates continuously from the filament during its operation. If it is overheated, there can be some melting, but the liquid coalesces into little balls on the ends of the 'gap' in the filament. There is nothing that would migrate from the gun to the turbo pump that would cause turbo failure. Pieces of specimen falling off your mounts are the bigger concern. John Mardinly John.Mardinly@asu.edu Thu Aug 3

Filaments are tiny and they tend to "burn out" because tungsten has evaporated away. Only if something catastrophic happened would a filament blow with a lot of debris. None of that debris would ever get to the turbo, and even if it did it is so small it would pass right through. Your vendor just wants to sell you a lot of consumables. Jerry Biehler jerry.biehler@gmail.com Fri Aug 4

Would this vendor happen to have any real estate interest in bridges? Maybe the one in Brooklyn? More seriously, unless the person who is spreading "alternate facts" is the owner of the company, you might check with the company to see if they know he/she is saying this. I doubt they'd be happy about it. Phil Oshel oshel1pe@cmich.edu Fri Aug 4

I've never heard of this. Certainly, my JEOL service engineer doesn't recommend anything but replacing the filament after it fails, and our 'scope has a turbopump. As for "shards"—I doubt it, but don't

Join us at the

19TH INTERNATIONAL MICROSCOPY CONGRESS

IMC19

Sydney 9-14 September 2018
International Convention Centre

BRIDGING THE SCIENCES

ARGYLE DIAMOND PARTNER

ThermoFisher SCIENTIFIC

OPAL PARTNERS

JEOL Solutions for Innovation

ZEISS

GOLD PARTNERS

GATAN

HITACHI Inspire the Next

WHY ATTEND IMC19?

- Be part of an outstanding scientific program led by world-renowned plenary speakers
- Network and share experiences and ideas with over 2000 colleagues and peers from across the globe
- Keep up to date with the latest advancements and developments in microscopy and visit our interactive trade exhibition
- Experience the stunning new International Convention Centre right in the heart of Sydney

KEY DATES

Call for Abstracts - 1 November 2017
Registration Opens - 1 November 2017
Abstract Submission Deadline - 1 February 2018
Early Bird Registration Closes - 10 May 2018
Author Registration Deadline - 10 May 2018

Download our Program at a Glance at www.imc19.com

VISIT SYDNEY - A WORLD CLASS DESTINATION

Sydney is renowned for its stunning harbour setting, temperate climate, and dining scenes. Whilst at IMC19 explore the iconic Opera House and Harbour Bridge and experience a myriad of other unique Australian experiences.

Position your brand at the frontline with other global leaders in microscopy.

Be represented at the premier global microscopy Congress. IMC19 sponsors and exhibitors will achieve superior brand exposure and direct networking opportunities with professionals from all disciplines of microscopy.

For more information or a copy of the Prospectus, contact Drew Whait on:

Email: sponsorship@arinex.com.au
Phone: +61 2 9265 0700

Find us on Facebook: IMC

Find out more at www.imc19.com

HOSTED BY

AMMS Australian Microscopy & Microanalysis Society

IFSM

CONGRESS MANAGERS

Arinex Pty Ltd
Level 10, 51 Druitt Street
Sydney, NSW 2000
Phone: +61 2 9265 0700
Fax: +61 2 9267 5443
Email: imc19@arinex.com.au

know. As for "more cleanup"—well yes, I suppose in that final "bye, cruel world" flash, you get more deposited tungsten. But you're going to clean the Wehnelt and anode anyway at each filament change. Julian Smith smithj@winthrop.edu Sun Aug 6

I might get a different vendor. That said, I have heard of and seen this. While visiting a lab running a TEM, I was shown a filament with the top blown off and told it happened because there had been a high voltage surge to the instrument, and tungsten filament debris was found in the scope. TEM, not SEM. I had just talked about this with a lab mate the night before this question was posted on the list server. Never otherwise heard of it. Kleo Pullin kleopullin@email.arizona.edu Mon Aug 7

In my 17 years with EBS, I've never seen or heard of a situation where the failure of one of our tungsten filaments has had a catastrophic effect on a customer's SEM or TEM. This is not say it is impossible, but given the hundreds of thousands of filaments we have manufactured during my tenure, one would think that if it were truly a problem that users need to be concerned about, we would have heard about it happening once or twice. Mike Nesta mnesta.ebs@gmail.com Sun Aug 13

SEM:
unexpected shutdown

We recently had a power blip/surge/loss? Last Friday. We noticed late Monday afternoon our SEM was shut down. It would not turn back on with the key, so we checked the power box in the other room and the reset was tripped. We flipped it back on and tried the key again, and it fired right up...for about 25–30 secs. In the manual it says after about 30 secs the DP power supply will turn on. I reset the instrument and tried to fire it up again and watched the light panel, sure enough as soon as the diffusion pump (DP) light turns on the SEM shuts down. Bad power supply? Maybe shorted or fried? If so where is this power supply and how could I check it in house? Any other ideas of what it could be? ***Gary Castelow gary@cermet-materials.com Wed Jul 19***

Did you check all the fuses at the power supply (the big yellowish block)? If you suspect the heater plate of the diffusion pump, take away the panels, unplug the heater and measure the resistance. I think it should be ca. 30–50 Ohms. When the SEM shuts down at start of DP heating, you might have a faulty heater plate. You will need to contact JEOL to get a fitting one (ca. 400 €...).You can try unplugging the heater and starting the SEM. If it starts then you know; but: it will switch off later since the thermo-switch at the DP will not close after a certain time (normally 20 minutes). Other reasons for the JEOL to switch off is not reaching the pre-vacuum trip point (check your pre-vacuum pump suction pressure), faulty vacuum lines (I once had a leak just in between the vibration dampening weight), and not sufficient air pressure at the SEM (4 bar). Stefan Diller diller@stefan-diller.com Wed Jul 19

It sounds like DP heater problem. There are two diffusion pumps under the column of your SEM, a small one and a bigger one. The heaters are at the bottom and are connected to 200V by a ceramic plug. Shutdown the SEM, unplug the ceramic plug and check if the two pins are still isolated from the ground. If not, there is a short circuit and you may buy another heater. Such problem appears often when the SEM shutdown but the water still flows in the pump hoses. Humidity of the room and the temperature gap between the water and air act together to condense water on the body of the pumps. This water can fall on the heater. Nicolas Stephant nicolas.stephant@univ-nantes.fr Thu Jul 20

Most likely your diffusion pumps are full of water due to condensation from the cooling water. If you dry them out, they may work but most likely need replacing. Bill Mushock wim5@lehigh.edu Thu Jul 20

SEM:
silicon oil leak

I am the owner of a very nice Philips model 525M SEM. It has a LaB_6 source and EDAX. It has been leaking silicon oil from the rubber sealed box containing capacitors that is attached to the side panel inside the instrument for many years. The instrument ran fine until recently. The detector signal started displaying severe noise, and then there was a popping sound. The noise went pop, pop, pop, at about 2 or 3 times a second coming from under the instrument. I assumed the noise is coming from the HV box under the instrument and not coming from the capacitor box. I shut the instrument down. The ion pump is still running to keep the vacuum up near the source. Your guidance, recommendations, instructions, questions, etc., for helping me bring back to life this wonderful workhorse of an instrument are appreciated. One additional detail, there are a few small holes on the exterior of the metal around the column. These are starting to show some kind of foam being extruded from them. The foam is bulging out of the holes by 2 or 3 mm. Not sure if that is significant or not. ***Tim Thomas tim_thomas@tkd-inc.com Sun Aug 6***

The leaking of silicon-oil in older versions of the high voltage parts (like gun, HV main supply and the side-mounted HV box to deliver the HV needed for the cathode tubes for viewing / recording) is a well-known issue at 5×5 electronics. It even happens at the HV cascade to produce the lower voltages needed for the SE detector cage (used in the SE module on the operator console). The best you can do is looking for a 5×5 SEM to break down for parts and hope you get a newer (black, not white) version of silicon used in those parts. If your monitors are still working showing an undisturbed image, the box sitting left-side back down in the electronics might still be OK. If at small acceleration voltages (ca. 5 kV) you get a stable image and the image gets noisy going up in voltage (happens mostly at 20 to 30 kV) your main HV supply (shoebox-size) is shortening and you need a replacement. You can try cleaning the cascades from silicon residue and use a new, high-voltage isolating silicon to newly isolate the parts but if there had been a lot of discharging happening in the past in the cascades you might have burned a carbon layer which makes it impossible to use / refurbish these parts. The "foam" you mentioned is coming from the upper part of the column (containing the cathode assembly)? There is also silicon used for the heating transformer isolation. If it's the old version (whitish), it will come out with time. You can only try cleaning the holes with petroleum ether / acetone and gluing them shut with two-component resin. The problem will get worse, since you need to tilt the gun 90° when changing the filament (or you dismount the upper column part and keep it upright all the times during filament change). The best way would be to look around for parts and exchange the faulty ones. Ask here at the listserver. Stefan Diller diller@stefan-diller.com Mon Aug 7

SEM:
vacuum issues

Our JEOL SEM JSM5600LV does not get vacuum ready. Normally pre-evacuation and evacuation phases last 2 min each, now pre-evacuation lasts 2 min but evacuation phase seems endless. After 1 hour, no vacuum ready appeared nor any message from the software. Can it be mechanical or electronic failure or both? ***Yorgos Nikas eikonika@otenet.gr Tue Aug 22***

Personally I do not have any experiences with SEM JSM5600LV. However, a long time ago we had a similar problem on Balzers BAF 301 freeze etching device. The problem was in a Pirani gauge. It was heavily contaminated. Therefore the vacuum level readout was completely wrong. In the Balzers manual, there was a procedure how to clean the Pirani gauge and it was working well. The cleaning of the Pirani gauge solved our problem. So, I would suggest to check the Pirani gauge in your SEM. Oldřich Benada benada@biomed.cas.cz Tue Aug 22

Thanks for your responses. As you suggested, I checked the diffusion pump heater and the pirani gauge; both looked fine. It turned out that the problem was a tiny bundle of dust fibers situated at the o ring sealing the specimen chamber. After some cleaning the scope goes vacuum ready but needs considerably more time, so I guess more cleaning may help further. Yorgos Nikas eikonika@otenet.gr Wed Aug 23

EDS:

elemental analysis

I have a few questions for EDS analysis using an electron microscope. I did one soil sample for one user; the composition of soil was unknown. Therefore, they did SEM-EDS to know the composition of sample #1. What is the difference between weight% & atomic% value in EDS report? ***Ravi Thakar ravi.thakkar369@gmail.com Fri Jul 14***

The answer to your stated question is simple. The answer to your unstated question is more complicated. Mass fraction or weight fraction is just that. How many grams of that element would you find in 100 grams of sample. Atomic fraction converts that over to moles so you might determine stoichiometry and the formula, if appropriate. For example, pyrite should be 46.6 wt% Fe and 53.4 wt% S. If convert that to moles, you would find it is 33.3 at% Fe and 66.7 at% S which tells you there are two atoms of S for every atom of Fe. The formula is FeS_2. Now the unstated questions should be "Was the analysis done properly?" and "How accurate was the analysis?" The analysis was probably done on a rough powder preparation. How well did that represent the original sample? What effect did that have on the accuracy of the analysis? The best analyses are from flat, homogenous samples. I don't suppose the soil was only one phase. How did you handle oxygen in the sample; how well does your detector measure oxygen? In short, the analysis will give a ballpark figure at best. I would not necessarily expect the same answer from two different preparations of the same sample. Caution your user about pushing the results too far. Warren Straszheim wesaia@iastate.edu Fri Jul 14

The difference between atomic and weight-percentage is, as the name suggests, that the at% is evaluating the number of atoms, while wt% is taking the weight of each atom into account. The wt% gives you approx. the "cooking recipe" to produce the investigated material (like steel for example: if it says it contains 55 wt% Fe, you need 55 kg in order to make 100 kg of this alloy). I know this is very easy spoken, but it may give you a hint of the meaning of that numbers. Furthermore you have to be very careful by interpreting EDS-analysis of minerals. It might be a good idea to review the excitation condition (overvoltage ratio of approx. 2.5). In almost all cases a HV of 15 kV might be a good starting point. If you are interested in elements with a very low Z (like Na), 5 kV might be a better choice. Furthermore, if you are interested in the highest accuracy/precision I would suggest using standard-based quantification algorithms. The usage of this approach may differ from supplier to supplier and it is not trivial. Therefore ask your supervisor and/or the applications/service team of your EDS equipment. Ferenc Molnar ferenc.l.molnar@googlemail.com Sun Jul 16

The basic difference between wt% and at% in the EDS report the way the software calculates the composition. In soil, there are several Si-, Al-, and Fe- (hydr)oxide phases in its composition. So, be careful with the EDS quantification report, as you cannot quantify light elements (Z<11) by using EDS. There's always delocalized electrons contributing to the X-ray emission for light elements. For oxides, it's better to quantify oxygen in such samples indirectly, by running a quantification model that takes into account the charge balance. Regarding the wt% of at%, it's up to you. Erico Freitas freitas.erico@gmail.com Sun Jul 16

For individual mineral grain analyses, the atomic % is useful for identifying some minerals based on their stoichiometry since it gives the number of atoms of a particular element per 100 atoms total. Calcite, $CaCO_3$ has 5 atoms, so it has 20 at% Ca, 20 at% C, and 60 at% O. Weight % is the mass of each element measured per the total mass. Calcite has molecular mass of (40.08 + 12.011+3*15.999) = 100.088g/mol, so it has 40.08/100.088 % Ca = 40.04 wt% Ca, and so on. For a soil or other mixture, the EDS spectrum should be collected over a large enough area that the results are representative of the bulk composition. Atomic % might not be very useful unless the numbers of each element are needed, but such results are often given in terms of wt%. Conversion from elemental wt% to atomic % or wt% as oxides (common for rock analyses) is relatively straightforward and described on most mineralogy textbooks. Jim Murowchick murowchickj@umkc.edu Sun Jul 16

Atomic percentage (or ratio) and weight percentage are two ways of describing the chemical composition of a compound. For example, for water, the atomic ratio is 2 H for each O (67% H and 33% O) the weight percentage is (total weight of water molecule is 18) 2/18= 11% H and 16/18= 89% O. As you can see, it is very important to know which one you are using because the percentage values are very different. You can find more information here: https://en.wikipedia.org/wiki/Atomic_ratiohttps://en.wikipedia.org/wiki/Mass_fraction_(chemistry). Please note that for an EDS analysis you also have the option to normalize the results (so that the sum of the element you are looking for is always 100%). Stefano Rubino stefano@soquelec.com Sun Jul 16

EBSD:

metallic samples

Regarding a metallic sample: I read that the sample should have the Normal direction facing the EBSD detector (at 70 degrees) for pole figures, but does the normal direction need to be facing the detector for Euler, Schmid, Normal, Transverse, and Rolling maps? ***Martin Taylor tayl1238@vandals.uidaho.edu Wed Aug 2***

The coordinate systems are a matter of convention rather than any particular technical need. A lot of samples that have EBSD performed on them aren't rolled. The important thing when making pole figures and maps is that you can relate the data supplied (and any anisotropy observed) back to the greater context of your sample in some way. If you don't care about how the maps relate to that greater context for whatever reason, then the directions won't really matter to you. Jacob Kabel jacob.kabel@ubc.ca Thu Aug 3

EELS:

plasmon peak analysis

I am looking for a good method in fitting the EELS plasmon peak for identification phases with subtle hydrogen concentration differences. The NLLS Gaussian fitting function in the Gatan GMS software seems not able to detect the very small energy shift. If any EELS expert can give suggestions in analyzing the plasmon peak, that will be great. ***Fei Long f.long@queensu.ca Mon Aug 21***

Hyperspy has an excellent library of EELS functions that can help you do this in an automated fashion. It's written in Python, so the syntax isn't too difficult to learn. Here is a link: http://hyperspy.org/hyperspy-doc/current/user_guide/eels.html. Steven Spurgeon steven.spurgeon@pnnl.gov Mon Aug 21

MT