

Microscopy Horror Stories

Recently, on the Microscopy Listserv, participants provided summaries of "horror stories" they had experienced in their microscopy laboratories. Those stories have been supplemented by readers of this publication. As follows is the completed series—for your hopeful enjoyment!

A bright fellow from a senior University (I would embarrass them) had a dim TEM image, so he rigged up an Anglepoise lamp to try and throw some more light on it.

Keith Ryan, Marine Biological Association

The respected PhD I once worked with on our old JEOL JSM-U3 scanner was having trouble making out the details of the image on the monitor, so grabbed a flashlight and shined it at the screen so he could see the image a little better.

Larry Allard, Oak Ridge National Laboratories

Many years ago a colleague from a friendly lab visited me with a great project. The idea was simply and beautiful. She argues that because the image in the scope (TEM) is green (green fluorescence of the screen), she wants to modify the sample (I forget what, some protein, I believe) by red-fluorescent dye to be able to see on the screen the "double-staining" red on the green background. No comment!

Sergey Ryazantsev, UCLA School of Medicine

A previous "ham-fisted user" reference made us chuckle/ cringe with the memory of a guest "microscopist" in our lab who hauled himself (220 pounds or so) out of his seat by pulling on the half-inserted specimen rod, bending it about 20 degrees or so! Henceforth, when we saw him in the hall (he never came into the scope room again), we referred to him as "Conan the Microscopist"!

Ron Anderson, IBM Corporation

We had a cousin of Conan, who in his addled age, after inserting the injector tip into the stage of our EM400 could not find his grid in the microscope; he had dropped it on the floor. Of course, the tip must have fallen off in the stage, so he promptly took a second tip and properly inserted the second tip on top of the first. Still no grid could be found. Ah ha, I will leave a polite note explaining the problem. It read as follows: "Please check the microscope, I had great difficulty inserting my specimens last evening." Philips kindly replaced the bulk of the stage just so they could keep the original for the museum of what not to do.

David Patton, University of the West of England

I heard this one from the Philip's engineers: A brand new Philips microscope was being installed in a government laboratory. It was to be in a state of the art, brand-new building. Everything was there, house nitrogen, chilled water, etc. Even the darkroom was state of the art. Well, when the in-house plumbers hooked up the water to the microscope they weren't being very careful in their reading of the blueprint designs and they had D-19 developer running through the EM instead of water. I'm not sure, but I think they got a new microscope out of that blunder!

Margaret E. Bisher, NEC Research Institute

Well, after reading Peggy Bisher's story, I couldn't help but add another along similar lines.

About 20-odd years ago, a prestigious institution purchased a state of the art new TEM. The main body came in a very large wooden container and was unloaded onto the load-

ing dock at the back of the building. It sat there for quite a while, because it was too heavy to move with the regular forklift, and I think the lab still needed a few final things to be finished off. Anyway, one day, someone decided that they were going to move the TEM in, and loaded it on the forklift. About half way to the lab, the TEM started oscillating back and forth on the forklift - it wasn't strapped on securely, and an eyewitness said he just stood there and watched this thing slowly crash to the floor on its side. Not much use rushing in and getting crushed by a few 100 kilos of metal.

I'm not sure it ever worked properly, the camera was smashed and a few other things too. The workshop had to retool all the smashed bits as best they could.

Amazing how many ways there are to destroy precision instruments.

Rosemary White, Microscopy Centre, CSIRO, Australia

When I was a grad student, I did a CPD run with a fellow grad student JT. I worked on plant pathogens and JT worked on chick embryo hearts so we had little pieces of leaf tissue and tiny chick hearts to dry. When the run was finished I couldn't get the lid off the CPD (it was a twist type). JT volunteered to muscle it off and when she did the lid shot passed her head with a boom and hit the ceiling. She had a grazing wound on her forehead but was otherwise ok. We both burst into fits of nervous laughter...we both knew she was so lucky not to be seriously injured. Then we looked in the CPD and saw that all the lids had blown off the little white sample containers. We howled with laughter when we got down on our hands and knees to search the floor for the tiny hearts and leaf pieces. The CPD was fine, the samples were fine, and surprisingly we both graduated.

Beth Richardson, University of Georgia

I worked for Humberto Fernandez-Moran at the University of Chicano many years ago. For those of you who are not familiar with the name, he was instrumental in developing the first diamond knives, producing the first pointed filaments for routine use and construction of first cryo-TEM using liquid helium cooled lenses. It was a very interesting place for a young budding microscopist at the time!

Dr. Moran had a large scar on his nose. He said it was from the removal of a cancerous skin area. He claimed to have gotten the malignancy in that location due to using electron microscopes in the late 40's without the benefit of lead glass windows. They used to press their noses against the window when concentrating on the relatively dim image projected by those early instruments. I wonder if other early EM researchers eventually developed cancer which might be related to similar research experiences.

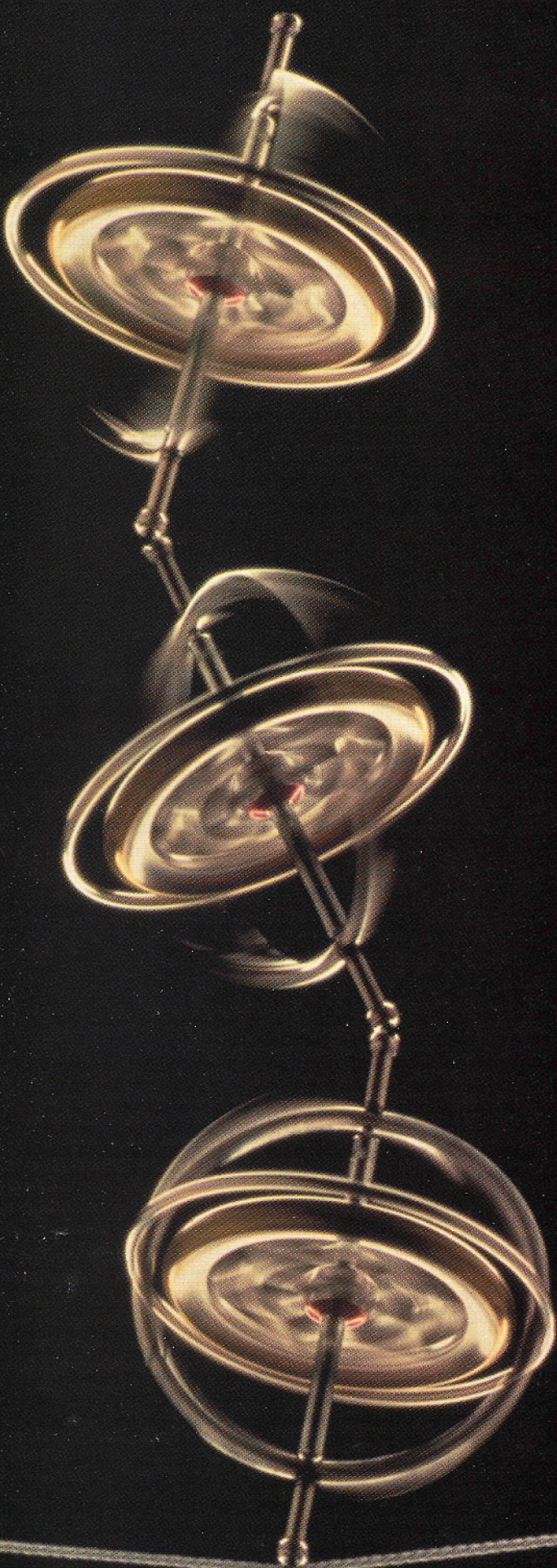
As it turned out, Dr. Moran lived a long life, although not without controversy through the years. He had a very unusual life history and was also a brilliant but erratic person to interact with... somewhat akin to what Bobby Knight is to basketball!

Debby Sherman, Purdue University

A friend had just finished cleaning his TEM column parts with acetone, as he had been instructed. Being an impatient young man, he quickly reassembled everything and, as he was lowering the column back into place, noticed a few drops of acetone had fallen into the viewing chamber and onto the phosphorous screen. I guess he had the chamber open for cleaning as well, because he said he quickly grabbed the canned air and aimed it into the chamber to blow off the drops. I walked in just then to see a cloud of yellow dust settling all over the walls and floor of the EM lab... He cleaned out the viewing chamber as best he could, I suppose, but there was dust in the column and pumping system for months to come. Then there were the phosphorescent footprints and fingerprints that appeared all over the lab for weeks! Now every time I open my viewing chamber I hold my breath.

Tina (Weatherby) Carvalho, University of Hawaii at Manoa

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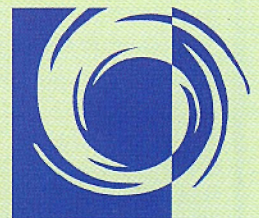
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Many years ago we had a Siemens EM-101. This was a relatively new microscope and beautifully machined with German precision. The camera chamber was a work of art. Each film plate (we used glass back then) was encased in it's own light-tight cassette. The camera would move a film cassette into position for exposure and pull off the cassette cover. The operator would then expose the film and move the cassette into a drop box. The cassette cover would be pushed closed in the process.

This camera worked almost flawlessly. On occasion, an operator, when loading the camera, would put the cassettes in on an angle. When you went to take the first image, the cassette would jam and not move into position. All that was necessary to unjam was to break vacuum, giggle the cassette with your hand to straighten it and then repump the camera chamber and get back to work...a 10 minute process at most.

We had one operator who thought she knew it all. She was working on the microscope one evening and the camera jammed. Her solution was to take out the camera and take it apart...screw by screw and spring by spring. She ended up with an incredible heap of parts and, of course, had no clue as to how to put it back together. Neither did the Siemens service engineers. They had never seen one apart! They had to find another camera (not easy)

In those days (with relatively few of these instruments around), one should take it apart (carefully), marking where each piece came from...and then reassemble each camera in order. It took hours!!

Moral of the story...NO ONE fixed ANYTHING without permission of the lab personnel!!

Debbie Sherman, Purdue University

My story is not as much of a horror as it is embarrassing. Several years back I received a package of samples from a regular client without any paper work describing what the samples were. This is not unusual since we frequently receive unknowns. I proceeded to open the package and was abruptly assaulted with an extremely strong odor of bananas. Apparently one of the samples was a vial of concentrated banana flavoring. It was months (almost a year) before the odor completely disappeared from my office, and I was cajoled about it frequently. I by the way, hate bananas.....

Lou Solebello

Once, over a decade back, I was working EM and Med Tech at the same time at a local hospital. We had a CAP inspection, the officers were from another hospital in the state. The question I was expected to reason and answer with a straight face was: "What other protective measures besides standing behind a lead wall do you take when you put the glass slide into the TEM, to protect yourself from the flying electrons???"

I was rather stunned, and started looking from the wall socket to him and back, but he didn't catch the connection. I then tried to tactfully explain that vacuum was required, and that we really didn't need that extra protection!

It was explained to me that he did too know all about EM because he had a one day workshop.

Lou Ann Miller, College of Veterinary Medicine

I am enjoying the "horror stories" (don't we all enjoy hearing about someone else's stupid mistake) and thought I would share one from the "software" category.

I had just joined Northern Scientific (later Tracor Northern, now Noran) and was writing EDS software for our first computerized analyzer (the venerable NS-880) -- the year was 1972, and my software, like all of the software being produced in

those years, was written on paper tape for machines with 4K memories. The software was not all that "user friendly", so it was not unusual to have to walk people through difficult command sequences. Then too, computers were pretty new to everyone and people sometimes made silly-sounding mistakes out of pure inexperience (one highly intelligent customer, when instructed to 'type a space', dutifully typed "A SPACE" on the keyboard). One particular customer, however, defied all attempts at instruction and had passed well beyond "complaining" into the realm of "abusive" and "insulting". He insisted that he was following the instructions to the letter, but that our miserable software consistently malfunctioned. We were unable to duplicate the problem and finally several of us drove a considerable distance to his lab to get to the bottom of this. We went through the operations with him watching intently and the software performed flawlessly, which he acknowledged. We then asked him to operate the software himself and midway through, observed that he was responding to one of the dialog questions with a decidedly inappropriate response. We had him back out of this and repeat it with the correct response and the software then ran perfectly. To which his response was to draw himself up and state with great authority and indignation: "but when you run it THE WAY I DO, it doesn't work!"

Fred Schamber, RJ Lee Instruments Limited

Here's another one. A customer on the west coast had a bottle of N2 that was used to vent their TEM. They also used the same bottle to agitate their developer. One day someone left the N2 on in the developer and it ran out. You can guess what happened next, the TEM was vented with D-19.

Dave Harrison

I fielded a phone call from a distraught SEM lab manager who told me that some months ago he put Dow Corning fluid into his diffusion pump "to save money". And now that he has had an accident, the silicone fluid of course, has contaminated his system, so he was asking us, "what organic solvent will easily remove it."

He was especially upset when I told him that his EDS detector will see Si everywhere also, because they do a lot of analyses for Si! I won't repeat what he told me when I tried to explain the reality of his situation.....

But it does go to show that there are a lot of new people entering our profession, some with less training and experience with vacuum than others, so such stories are very well worth repeating.

But just out of curiosity, is there some "recommended procedure" for removing silicone fluid from the internal parts of a column, and also removing it from the window of an EDS detector? I presume one can always call in an outside service provider with experienced people but a lot of users out there just don't have the budget for something like that. But they do have a good supply of student manpower.

While we are on the subject of silicone, a few weeks ago a well known TEM user got me on the phone to say "hello" and commented that he had just placed an order for silicone grease and, yes, he said, he was going to be using it on the o rings of his column. I told him I thought that he should be using other greases and his response was "don't listen to dogma, I thought you read the listserver!". Am I correct, namely that one should not be using silicone grease on the o rings of a column instrument?

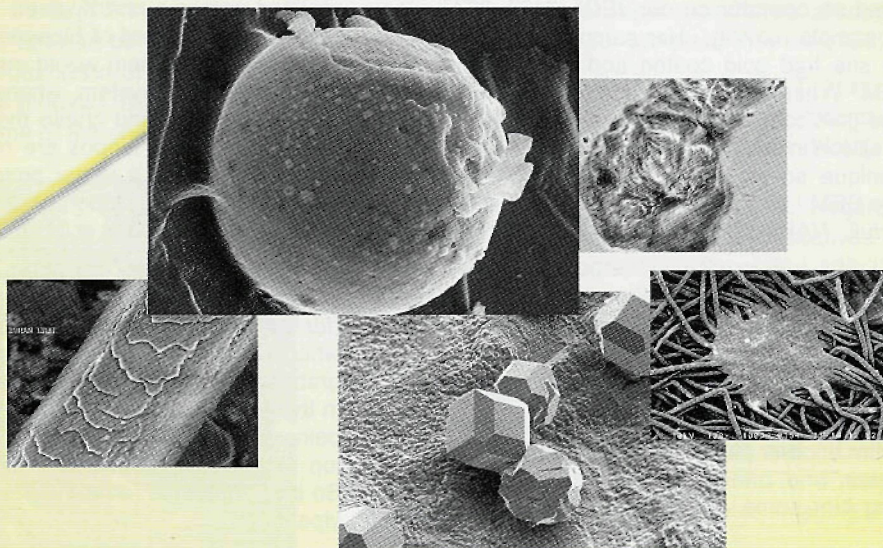
Charles Garber, SPI Supplies

I heard that one years ago. Also one that supposedly happened at University of Wisconsin. Someone apparently cleaned a scope's internal parts in acetone and started pumping. Before it was fully pumped down, they turned on the filament. Bang! Nice story but could it possibly happen? I would think that any acetone residue would evaporate very quickly or is there some other phenomenon here relating to ratio of gases?

Damian Neuberger, Baxter Healthcare, Inc.

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Is there any truth in the story I was told about a lab in London that replaced the leaded glass on the microscope chamber with regular glass? Apparently the mistake was discovered when all the film on the shelf behind the operator became fogged!

Paul Webster, House Ear Institute

We once had an operator on our JEOL 840A SEM complain about her sample moving. Her sample was samples of vacuum grease she had gold coated and was attempting to image in the SEM. When the electron beam heated the grease it cracked up the gold coating and charged. She was using the SEM to look for silicon in various greases. I suggested she use a different technique so we did not end up with vaporized grease inside the SEM.

David R. Hull, NASA Glenn Research Center at Lewis Field

I got in an argument with an engineering prof. that disputed that the inverse square law applied to photo flash units.

Gordon Couger,

And one from us...

The guy who asked how long to wash between dehydration steps and from another place a long time ago:

The Professor who offered to pay the electron microscopist by giving him some oil immersion objectives for the TEM.

Sally Stowe, ANU EMU, Australia

Back during the asbestos craze I was the last "little indian" doing TEM analysis, the other two left for bigger and better paying jobs elsewhere. My business managers (who knew the EM was big and needed electricity, but that was the extent of their knowledge) suggested cross-training some in-house employees to assist me with my work. The first guy walked into the scope room chewing gum (not like a normal human being, more like a horse). I started showing him the scope (JEOL 100SX) while he sat in the chair in front of the scope. The phone rang so I turned to answer it. Meanwhile that cute little handle on the camera door caught the new guy's attention. "what's this?" I heard, and then that all too familiar hiss followed by valves closing and pumps and power switches clicking off. He just reached out and turned the handle, venting the column. The scope handled the shock better than I did. I sent the guy to lunch, and locked the door behind him.

Jon Ekman, University of Wisconsin Milwaukee

A couple decades + ago, while providing TEM services for the NIH neurology group, the neurosurgeons were impatiently awaiting the biopsy results of their surgical patient. The frozen section was not conclusive, and so they needed a TEM result which would confirm their findings. Not being satisfied with my answer that the results will take a couple days, or one day at the very earliest, the surgeon sent his first resident up to my lab and he began rummaging through my supply cabinets so he can prepare the sample quickly himself. This is before microwave embedding and fixation. He told me that he was instructed (by my boss) to cut a very thin slice of tissue (use a very sharp razor), coat it with a lot of glutaraldehyde (straight out of the bottle) and stick it into the scope (a JEOL 100 at that time). He would be waiting in the OR with the patient until he got the result. He declared that he did not need any training. ...

I was speechless and could not believe this attempt. I watched the "procedure" with amusement—and voluntarily moved on to safer grounds thereafter.

Thomas Baginski, Uniformed Services University of the Health Sciences

This is a story that was told to me by Dr. Audrey Glauert of Cambridge University in the U.K. Even though it's not my own, it is so amusing that I can't help relaying it to you.

It seems that a number of years ago, Dr. Glauert spent several months working in an electron microscopy laboratory in Africa. Every now and then the water supply to the laboratory would go off making it necessary to shut the electron microscope down for an extended period of time. Investigation eventually revealed that the problem arose because the town involved was getting it's water from a pond that was formed behind a dam that had been constructed across a nearby river. It seems that this pond was a favorite site for a herd of hippopotamuses to bathe, and every now and then one of them would manage to plug up the water inlet to the village water system, whereupon it was necessary for the villagers to go out and chase the hippos away and then open the inlet again. Since hippos are not very easy to chase, this could sometimes cause a rather prolonged period without water in the electron microscopy laboratory.

Wilbur C. Bigelow, University of Michigan

I hope this scary one helps some of you!

We are a research and teaching lab (thankfully!) A very lucky (for us) student was working on a weekend when she noticed white smoke pouring out of the lab into the lecture room, then grabbed the extinguisher, and put out the electrical fire that started in the wall due to our specimen rotator motor shorting out (and not being properly wired). The fire was directly underneath the 1 gallon storage tanks for 100% ethanol, and pure Xylene.....<yipe>! So bad, that it melted but did not break through the spigots...<big yipe>!

I also learned that when you are filling a liquid nitrogen tank, and it gets full, and it begins to shoot up a little underneath a temperature sensor, you get to meet the fire chief!

Life is good!

Tracey Pepper, Iowa State University

Another department on campus was kind enough to give us a TEM, a JEOL 100 CXII. It, of course, had minor vacuum leak which put it out of service, but besides that it was in mint condition. I had planned to move it myself but was overruled by my boss. There was concern for injury to myself or others in case it fell. I begged, whined, pleaded, and lost the discussion. A moving company would transport. I talked to the administrative person arranging the move and was told that the moving company said it had all the information it needed. Bells, whistles, lights, and red flags went off in my mind. I finally got hold of the moving company supervisor and was told he had done things like this before and knew what had to be done. I told him several times, NOT to use the lift-gate on his moving truck, to use a forklift. The column was too heavy, and balanced wrong for liftgates to work well. He assured me HIS truck could easily lift the 3,000 lbs. More flags waved! I told him one last time to use a forklift to avoid problems. The day for moving came and on this day, as well as the next two appointments, the movers gave the excuse the truck clutch was out, more red flags. When finally they did get the clutch repaired, I unfortunately (fortunately?) was not there to see the fireworks. They tried to use the lift gate, got the column several inches off the ground before both hydraulic cylinders for the truck lift gate sheared off, dropping the gate and giving the column a good bounce. As good luck would have it, the microscope didn't tip over, just wobbled a lot. Had it been higher, a lot of damage could have occurred as well as someone could have gotten badly hurt. To top this story off, the moving company had the audacity to try to charge the University for the cost of repairing their truck. The damage to the microscope was limited to knocking the intermediate and projector lenses out of alignment and the instrument is working wonderfully in its new home. "Takes a lickin' and keeps on tickin'."

David L Bentley, The University of Arizona

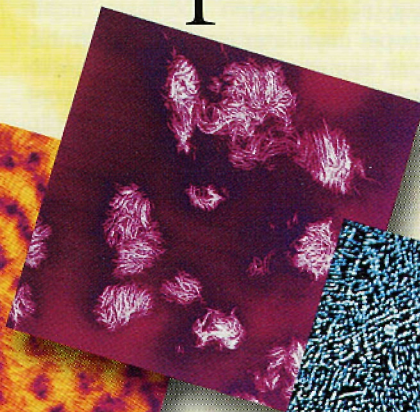
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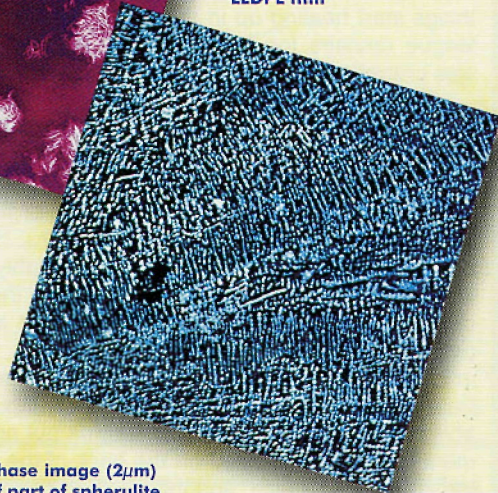
Phase image (14 μ m) showing banded spherulite of PE



Phase image (5 μ m) of lamellar aggregates in sub-surface layer of LLDPE film



Phase image (2 μ m) of part of spherulite in melt-crystallized isotactic PP



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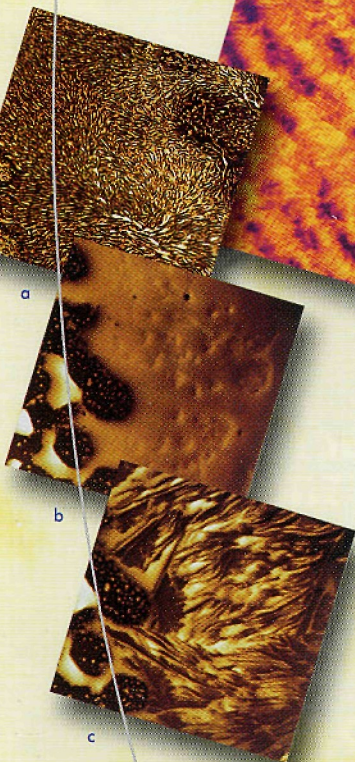
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Phase images (10 μ m) of 20nm thin film of LLDPE at (a) 25°C, (b) 126°C, and (c) after crystallization at 102°C



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My horror story is pretty recent: Just last January we had a local SEM service guy come in to do a semi-annual tune-up on our ESEM, and he did a great job. He did notice, though, that our ion pump wasn't giving us quite as good a vacuum as it should. When he left he said "All you need to do is bake it off a bit with some heat for a few hours - that'll refresh the active ingredient (or parts) inside and it'll work better for you. I've got a heater that's designed for just such a job."

True to his word, a couple days later he dropped off this device for me. Just a bigish aluminum box in two parts, with luggage clips to lock it together and a 1500 W heater element inside. Simplicity itself to use, just turn off your ion pump, clip this box over/around it, and plug it in for a while. The heater element rests right against the back of the ion pump. So I mounted it on there, plugged it in, waited a bit, and sure enough, things start heating up in there. OK, I think, I'll go see my colleague upstairs for a few minutes about those samples she was preparing.....

I'm back down in the lab ten minutes later and go back behind the scope to see how things are doing. There's a small hole in the back of the heater apparatus and when I happen to glance in there, I see a small wall of blue flame. "This can't be good", thinks I....then "Now which type of fire extinguisher do you use on a burning ESEM? Water?.....No, probably not. Powder?.....uhhhhh.....no. CO₂? Probably..." Meanwhile, I unplug the heater and mostly just stand there....thinking how close I've come to pensionable retirement age, only to lose it like this.

But, anyway, the flame started to die back a bit as soon as I unplugged the thing, and with some damp towels I was soon able to unclip it and remove it. It turns out our particular instrument had a clip mounted on the back of the ion pump to retain the HT cable in place, and this clip had been mounted on piece of black plastic, which I swear to God I thought was anodized aluminum. The plastic had melted with all that intense heat, of course, and dripped right down onto the heater element where it had burst into flame.

I had our machine shop make me a metal replacement for the lost plastic bit and there was no other damage. By God, the ion pump now works great and I figure local management doesn't need to hear about every little detail of life in the SEM lab, now do they?

Frank Thomas, Geological Survey of Canada (Atlantic)

I set up an lab in a hospital with a Siemens microscope-a wonderful instrument for resolution but a beast to align. The lenses were physically moved during alignment so that it did look odd to see different components of the column a few centimeters off. One morning I came in and a pathologist proudly told me that he had aligned the column for me. He had straightened it out very nicely and it took a full day for me to get it back in alignment. It did look nicer the way he did it but of course it was impossible to use.

Joyce Craig, Chicago State University

Here's one that involved the fire department:

Early one evening when the only people still in the building were us students, a guy from down the corridor came into our room saying that he could smell something downstairs. We all banded together and went down to investigate. After a while we noticed this sort of thick foggy smoke beginning to form below the ceiling along the length of the corridor and in the adjacent large teaching lab. We noticed it wafting down through some light fittings and gradually getting lower down and thicker. "Has to be electrical!" "Looks like the whole place could go up any minute!" It started looking pretty serious. When the fire brigade turned up we were told to get out as they proceeded to

rip out chunks of the ceiling trying to locate the source. It was a very diffuse source and there was a very large area of ceiling to rip chunks out of. After a while their investigation moved upstairs -- where they found our carbon coater happily chugging away with it's bell jar off and the exhaust line feeding down into the ceiling cavity! You learn something new every day.

I think we remembered to say thanks....

Arthur Day, Ansto Materials Division

My horror story happened many years ago when I came to work one morning to find the Philips 200 had been left running for a long period of time with no water coursing through its veins. The column was hot to touch so my immediate reaction was to turn on the water supply to try to cool it down. But hoses and o-rings had deteriorated from the heat, so instead of cooling the instrument down I now had water gushing from everywhere and filling up the column's viewing area so that it looked like a fish bowl. I called my Philips service engineer and he spent the next 3 days repairing and cleaning up the scope. He said the stage had come very close to a melt down and if that had happened I could have just dug a hole and buried the scope in the back yard. The scope survived to give the lab many years of service, thanks to the Philips engineer, John Braunagel, whose expert service was given without a grumble or complaint.

Betty Loraamm, University of South Florida

At only 23 years of age and only 18 months of intermittent "user experience" I found my self in charge of a small EM Lab with a Phillips 100C as the center piece. An interesting TEM, with the column in the near horizontal position and a transmission viewing screen. There was some minor leak problem with the specimen holder. That TEM design prepumped around the holder on partial insertion, while a spring loaded ball sealed that space from the column. The holder was then pushed in completely and that pushed that ball aside. Simple and effective.

To fix the ball seal I had to open the column and so I also cleaned various bits and pieces and re-assembled. Pumped and realigned, I then pulled the specimen holder out, whereupon the TEM inhaled very deeply. I later found that that spring loaded ball assembly would fit 180 degrees reversed quite well, but in that position the ball would not roll back to cover the seal when the specimen holder was withdrawn.

The main damage was a clear center on that lovely transmission viewing screen, the coating had been vacuumed. It cost \$300 then, which is more like \$2000 today and I had another opportunity to hone my maintenance skills, cleaning column, oil and Hg pumps.

It was a very lonely job, with so little experience running that lab without any other assistance, but I never learned so much as during those 18 months in that lab.

Jim Darley, ProSciTech Microscopy PLUS

Waaay back, when I was an instructor at MIT in Cambridge, Mass., I was helping out in the physical metallurgy laboratory course when we were teaching the students how to develop glass metallographic plates (I told you it was a long time ago). One of my students, the son of a famous metallurgist, placed his fully developed plate on the belt of a machine that he didn't notice was running. We never let him forget the day he tried to dry his plate in the cylindrical print drier. I am trying to forget that everyone used mercury ice-point reference junctions in their thermocouples in the heat-treating lab in those days ... and how many got tipped.

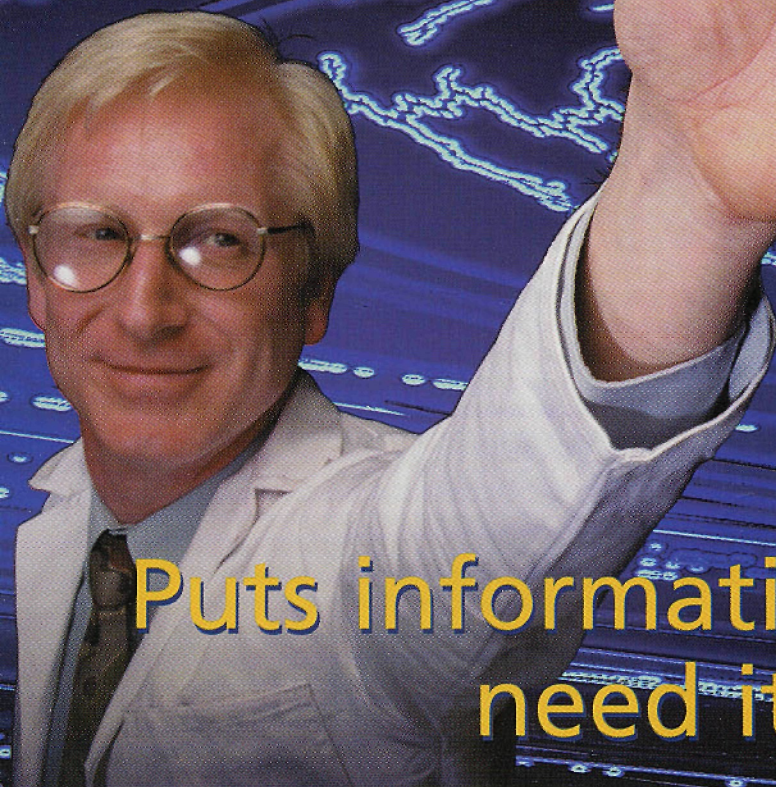
George Langford

We once had a student twist the condenser control knob off a TEM. When asked to turn the knob clockwise, he just kept turning, until he handed me the knob.

William McManus, Utah State University

Continued on page 16

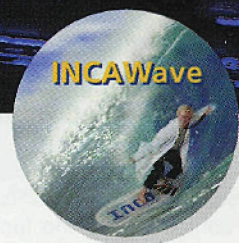
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Microscopy Horror Stories

Continued from page 14

A year ago (recent history!), I was teaching a lab section in Electron Microscopy, and although overloaded with students, things were cruising along reasonably well. One day, however, in a fit of brainlessness, I instructed a student who was doing cell cultures in how to dehydrate samples for TEM. We were processing his samples in a culture plate. He was going to embed in Spurr's, so I took him through the ethanol dehydrations up to 100%, then moved on to, you guessed it, propylene oxide. The plastic cell culture plate he was using was not amused. Right in front of both of us, it melted and "embedded" his samples in a resin that I was not prepared to risk a diamond knife on. His comment was something like, "Is it supposed to do that?"

He finished the semester with flying colors and presented a very respectable project. Even better, we're still friends.

Randy Tindall, University of Missouri

We had a technician here who I taught how to develop film for our JEOL 2000FX. It had been awhile since he replaced the film, so after he left, I wanted to make sure that the film was loaded into the cassettes with the emulsion side up. When I opened the film cassette box, I couldn't take out the cassettes. He had put them in around 180 degrees and the slot in the film holders didn't match up with the alignment bar going up along the side of the box. The sides of the film box were actually bulging. I had to pry each cassette out of the box with a screwdriver. I left most of them for him to do the next day. Before showing him this, I innocently asked him if he had trouble putting the holders into the box. He said that the last couple were a little hard to put in. The good thing: he had loaded the cassettes with the emulsion side up.

Scott D. Walck, PPG Industries, Inc.

About a month after starting my post doc in the lab of Professor Ruhle in Stuttgart, Germany, I was working on the TEM late one Sunday night. I was using a double-tilt, analytical, double-specimen holder (expensive) and was just putting it away. The holder was gripped in a Gatan holder stand and when I pushed the protective end sheath onto the tip the stage moved back so the front stand grip moved onto the narrower part of the holder where it doesn't grip. The motorized back end was heavy and the holder tipped backwards while I was still holding the protective end piece firm. What resulted is a holder that greatly resembled a Concord airplane coming in for a landing. It bent the holder at about a 30 degree angle right where the hinge was for the back specimen cup.

I sat and looked at it, beads of sweat forming on my forehead, contemplating the fact that I had an open ended return ticket and wondering if I could pack my stuff up and be gone back to the U.S. before anyone noticed. Of course I stayed and Professor Ruhle was very good about it, basically saying that mistakes happen but don't do it again! I didn't.

Lessons Learned: Be very careful when handling expensive specimen holders; think twice before you do anything.

I hope you all have a good weekend and don't mind my confessions!

John Vetrano, Pacific Northwest National Laboratory

As a field engineer, I have always told my customers, "The only stupid question is the one that you don't ask."

I will also say that occasionally it can be very difficult to hold one's temper when someone has done something stupid on an instrument covered by one's own service contract and then they try to lie about it. Users, don't add insult to injury. Tell us what you did so that we can more rapidly repair the damage by looking in the right direction.

Field engineers have horror stories, to:

I became an expert on the ETEC Autospec WDS as an ETEC field engineer while being impatient in looking for vacuum leaks. The Autospec about doubles the volume of the system and therefore takes a lot longer to vent. I had put a 13-1/2 stopper in the port for the secondary detector to see if the SED was the source of the leak. When it was about half vented, I pulled the stopper. Without the WDS, this wouldn't have caused any problem, but I drove the columnator/electron trap into the 4 crystal turret, broke the tape drive and blew the thin window detector, along with destroying 2 crystals and loosening all 4. The process of fixing all this was a three week intensive course in WDS alignment and operation.

The moral: Don't rush. Take it easy and (God forbid) THINK before you act. It was only milliseconds to create three weeks of work and some \$4k in damaged parts.

Moral #2: Learn from the mistakes of others, because you'll never live long enough to make them all yourself.

Ken Converse, Quality Images

Several years back I was looking something up in the Balzers 400 freeze fracture manual, and noticed it recommended strongly that the turbo pump be sent in for reconditioning every 50,000 hours of use (or some such number). I had no previous experience with turbo pumps, and the consequences sounded pretty dire, so I was concerned. Since it had run 24 hours per day, 7 days a week, for several years, and then off and on for several more, it easily had run those kind of hours. The facility director was getting ready for a big project utilizing the instrument and, although he was reluctant, I convinced him that we should send the turbo pump in before he started.

It came back a few weeks later, and it was clearly not our pump, but another reconditioned one. As I lifted this large pump out of the box two small ball bearings bounced onto the floor and rolled away. Still holding the pump, I watched them go. Then I turned the pump all around in my arms, looking for any signs of damage or loose parts, but all looked fine. I considered calling the company, but it was Friday and with the time difference, it would be days before I got an answer. I figured what the heck, either it is going to work or not! So I installed it and turned it on, standing as far away as I could. It started up fine and achieved a reasonable vacuum, so I left it running over the weekend. Monday afternoon I decided it was OK, and turned it off.

My first thought was that a jet had crashed into the wall behind me and that I was going to die. And from the look on the faces of the others in the lab, they clearly thought they were going to, as well. The horrible screeching actually stops really suddenly as those pumps seize up, and then the quiet is deafening.

I opened the chamber to find it full of aluminum glitter.

The pump was replaced.

When I talked to some EM service people who had a lot more experience with turbo pumps, they all seemed to think that one should never overhaul a TP, but just wait until it crashes. Sure, it's a lot more expensive to repair it then, but apparently few do fail within the normal lifetime of the instruments they are on. Sigh.

Tina (Weatherby) Carvalho, University of Hawaii

For those of you that don't know the instrument, the XL30 ESEM, like most modern SEM's, displays its image as 256 intensity levels on a computer monitor. Presumably the colour could be set as the user chooses, but we use the default black-and-white.

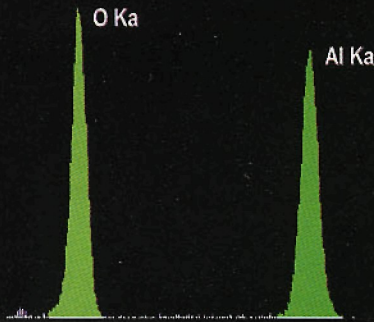
I had a young photographer working for a prestigious magazine who needed an SEM photograph of a human hair. I had the microscope set up before he arrived, so there was an image on the screen as he walked in. After a few minutes, he asked "I thought you said this was one of your own hairs". "Yes", I replied. He looked puzzled, looking closely at my head, then said "Did you take one of the gray ones?"

Anthony J. Garratt-Reed, MIT

Continued on page 18

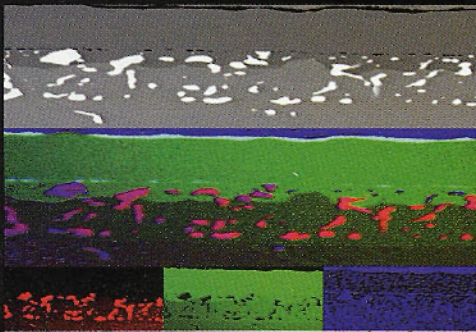


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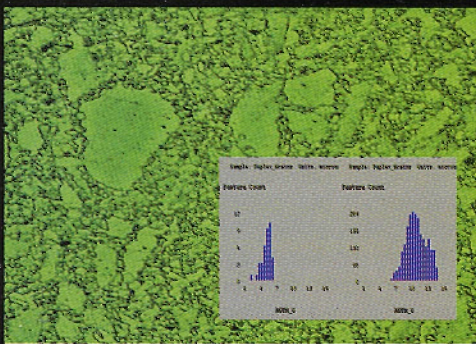
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Microscopy Horror Stories

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When I took over managing the EM facility here, I ordered a new cylinder of CO₂ for the CPD. When it arrived, I pointed out to the delivery person that he made a mistake and had not supplied a tank with a siphon tube. He replied that he delivered what he had always delivered. I checked the shipping records, and, indeed, my predecessor had used CO₂ from a tank without a siphon tube--in other words, for a decade he never critically point dried a single specimen, since only CO₂ gas would have entered the chamber! Perhaps more than a few people may want check their cylinders. (In the US, the proper cylinder typically has a red band painted at the top of the cylinder and the words "w/ dip tube" stenciled on the side.)

Donald L. Lovett, *The College of New Jersey*

A former colleague spent approximately six hours (over three separate days) analyzing metal wear particles on the SEM. He was doing the work with a French engineer who had prepared the samples. She told which particles to analyze and he dutifully analyzed them. I happened to pass and glanced at the screen and asked why they were analyzing paper fibers. They both insisted that I was wrong (unfortunately they didn't take my bet!), until I suggested that they try using the BSE detector. Strangely, they had lower contrast than the Al stub. The engineer has almost finished her PhD now and my colleague has moved on to become a forensic scientist. Just goes to show the customer is always right?

Colin Reid, *Trinity College Dublin, Ireland.*

At Martin Marietta Labs (1977), Harry O. and myself were responsible for training, use, and maintenance of an ISI Mini-SEM. One morning we found the SEM with a blown filament after late-night operation by user or users unknown. Further investigation revealed the remains of a house fly affixed with Aquadag to a specimen stub -- and dispersed throughout the microscope. The innards of the house fly in the microscope prevented the SEM from reaching operating vacuum. It took two days to adequately clean the column and the vacuum system. Apparently the guilty party wanted to examine a housefly in the SEM, but did not think about it exploding in high vacuum. The fact that the fly was not sputter coated suggested the likely guilty party. He later confessed and was denied further access without close supervision.

Steve Stokowski, *Stone Products Consultants*

It seems everyone has one of these. I had a person who one day complained of a burning smell while she was at the scope. I went to investigate and when I arrived in the room there was no smell...burning or otherwise. Several more times that session she came and got me to investigate this burning smell in the TEM room. This continued for a couple of sessions. I did go and investigate because after all, who wants their TEM on fire? I honestly didn't know what to do about it and was beginning to seriously question her sanity. Finally she convinced me to sit with her while she operated the scope and sure enough there was this faint smell of something burning! I jumped up and tried to find it, but it had gone away. So...she sat down again and began to work and the smell, like plastic kitchenware burning in a dishwasher, came back. Again I jump up and look around....to no avail! I was curious now. The burning smell only happens when she is sitting at the scope. Not being a big believer in spontaneous combustion, there had to be an answer! There was. As part of the TEM course I take the kick panel off the Zeiss 10 to show the students the guts of the vacuum system. The kick panel is right below the desk area where you sit up to the microscope. What she was doing was placing her tennis shoe on the heater of the diffusion pump and the plastic/rubber would melt and burn a bit -- just enough to

give the room a burning smell. She never did notice her foot getting warm. When I pointed it out she did admit that her shoe was warm and had some ugly scars on it to boot! She never returned after that semester - I guess that she had enough of EM! Now I carefully point out the hazard and replace the cover quickly when we are finished with the class.

Greg Strout, *University of Oklahoma*

I was working with an investigator who wanted to see matrix vesicles. Well, we were so happy that we had a nicely fixed sample with "lots" of vesicles. Nice whole round ones. Well, when the negatives were developed, my director and I discovered that what we really had were nicely fixed, beautiful bacteria!!!! No vesicles! Another time, in school, I was walking down the hallway late in the afternoon when I saw the scope room door slightly open. I popped my head in to see who was there and to ask if they wanted the door closed. When I looked inside, I found a student sound asleep, with hands still holding the knobs of the microscope!!!!!! Funny huh?

Ron Anderson, *IBM Analytical Services*

I had a student using my JEOL 100 CX II and her loading and looking around went well. When she went to remove the holder, she got it half way out and yanked it toward her. This action put a 70 degree bend in the holder. I could never get the Z height to behave after that for some odd reason.

The other disaster was with my Philips 300, complete with Mercury Dif pump at that time. I needed to do some anode cleaning, so without deflating the air table I got on the console, finished my work and got off. When the scope lurched to the right it dumped the contents of the mercury pump into the oil pump. This sent a cloud of mercury up the column creating an alloy wherever it went. Believe it or not, the scope is in fine working order with not a trace of mercury left in in the column. How did I do it? If you can figure it out I will send the winner a Lucent/Bell Labs brief case...at my cost of course.

John Grazul, *Lucent Technologies*

I had one faculty operator several years ago who after a SINGLE lesson on the Phillips 200, decided to come back late at night and look some more. After taking some images, he couldn't get the door to the camera chamber off, since he had failed to vent it. After rummaging through the lab, he found a large screwdriver and commenced to pry the camera door off, unconcerned about the marks and gouges that he was putting in the brass. Needless to say, when the vacuum was finally broken, the mercury diffusion pump became very unhappy. Eventually we were able to replace the instrument and the user finally left after being denied tenure.

W. L. Steffens, *University of Georgia*

One day a user of our TEM complained that she could see the beam with the specimen out, but as soon as she put in a grid the screen went black. After playing with it awhile and ruling out magnetism or something on the grid holder, I decided that there must be some small magnetic particle in the stage area that was being pushed around by the grid holder. Of course the service technician I got by phone wanted me to begin taking the column apart from the gun on down, but I grew impatient with this and soon went straight for the stage. I figured I would be looking for some small metal sliver. Imagine my surprise when I found an entire plastic pipette tip lying across the bore through the anticontamination plate in the column! It seems the previous user had been using these tips as forceps covers, and mysteriously lost one the day before. It had gone into the column through the airlock with her grid, not impossible with the Zeiss 10 top-loading system. Although not the only strange thing I have found in that microscope's column, at about an inch and a half long, but it is the largest.

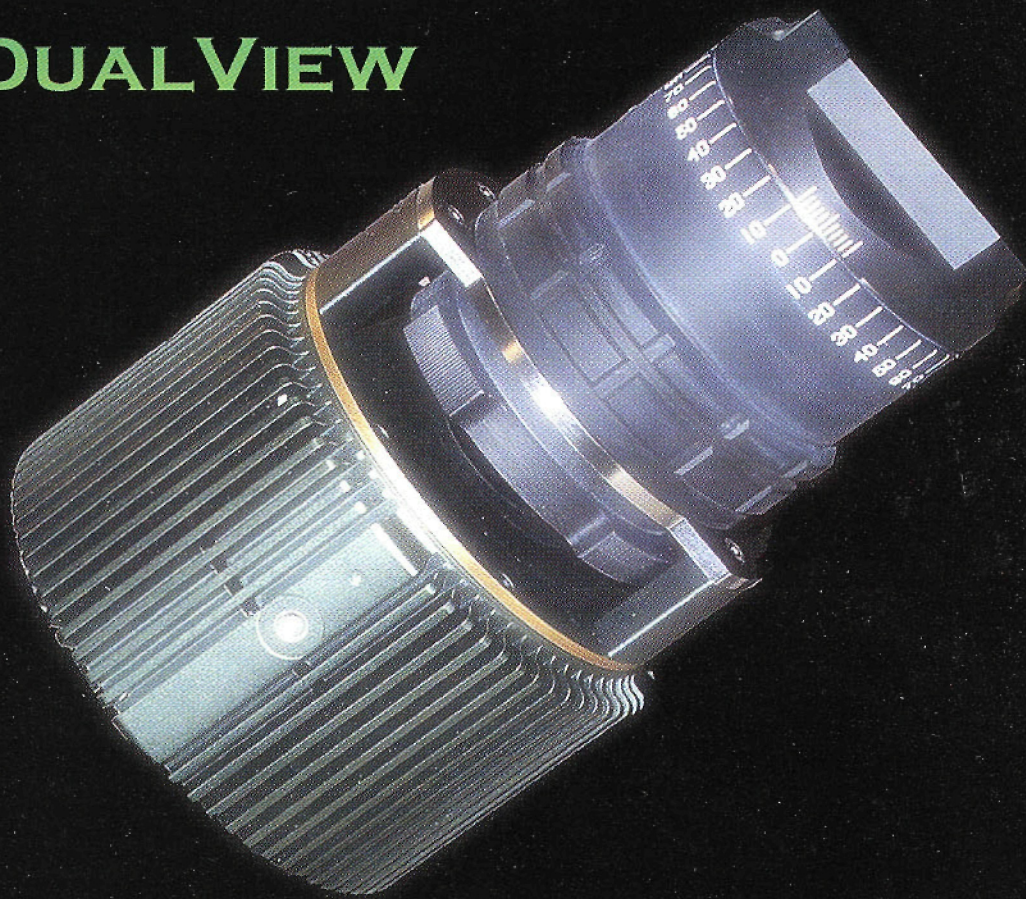
Tina (Weatherby) Carvalho, *University of Hawaii*

Continued on page 20

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Microscopy Horror Stories

Continued from page 18

There once were two eager graduate students at Florida in the Materials Science and Engineering Department (one has the initials MK and now works for JEOL) that decided that they wanted learn how to align the Philips 300 TEM. Armed with the user manual, they proceeded to align the column, but couldn't do it. They had to call in the service engineer. The first thing that the service engineer did was to use a framing square to help to take the visible bow out of the column. It seems that the user manual they were working from was different from one for a microscope with a STEM attachment. (Not witnessed by me, but heard from reliable sources.)

A professor in the same department was escorting a visitor around the EM lab and the two were discussing possible experiments that might be performed in the Philips 300. They asked the EM technician to give them a hand opening up the microscope so they could look inside. The technician was busy and said that he would be with them in about 5 minutes. Well, busy professors can't wait for lowly technicians so they decided to open the chamber themselves. The professor cranked on the handle to open the gun chamber while it was under vacuum and the voltage was on. Bang, pop, whoosh, whistles, bells brought the technician running to see what had happened. After all, they only wanted to look inside. But, there was a happy ending to the story. The vacuum and diffusion pump were OK and the apertures were cleaned by the supersonic air. (I was around when this one happened.)

Scott D. Walck, PPG Industries, Inc.

Several years ago, one of my research students was using the Cambridge S250 SEM in an early solo session. The room was hushed, in almost in total darkness, and he was giving his total concentration to the screen while he adjusted the image. As he moved his hand to the specimen stage controls the turbo-molecular pump disintegrated without warning, making a crash that sounded like a metal tray full of spanners being dropped from a great height, followed immediately by the wailing of alarms. The poor chap was literally green with shock - he thought he had caused it!

When the column was opened up a glittering cloud of aluminum alloy powder drifted out. The turbo pump - a double-ended model—had its rotors and stators intertwined so forcibly that there was no free play. Presumably it had come to rest from 60,000 rpm in less than a single rotation. That works out at a damage rate of almost \$1 billion per second!

Chris Jeffree, University of Edinburgh

Three short "horror stories"

[1] Eons ago (actually, maybe 25 years) I was a service engineer with Philips and went to perform routine maintenance on a very early EM-300. I always cleaned the windows and in this instance, I found the smaller, left, projection window to be Plexiglas! No one would tell me how long the ersatz window had been in place but I really made my point when I demonstrated, with a Geiger Counter, that lots of x-rays were getting sprayed into the room. They quickly got as new window. I also had a similar experience when a customer stuck the Aluminum shipping plate in place of a broken window. They also bought a nice new leaded glass window. See, the window stories are true.

[2] Some of the older microscopes used Mercury Diffusion Pumps to increase pumping speed. They were usually operated in tandem with the Oil Diffusion Pump. In one of the not terribly uncommon disasters where a blast of Mercury up the column turns all the beautifully Gold plated brass pieces into a amalgam covered mess, a service engineer who was particularly resourceful found that Iodine readily combines with Mer-

cury. He sprinkled Iodine crystals all over the contaminated parts of the column. Not long later, there was an explosion! No one was physically hurt, and I heard the microscope company cleaned up the mess.

[3] Our laboratory was below the level of a creek so when the sump pump fails, water would seep onto the floor, fairly quickly. A lovely and dedicated graduate student was working late a night when one of these pump failures occurred and she ended up with her feet under water while running our old JEOL 35C Scanner. I found her and said she would have to stop, as I yanked the wall switch. She was angry with me for interrupting her research until I was finally able to convince her she was very close to electrocuting herself.

Alex Greene, Scientific Instrumentation Services, Inc.

Many, many years ago... I was asked to evaluate particle size of a powder. The powder was dried sludge from a radioactive waste storage tank. My downfall was permitting a co-worker to prepare the sample. When I received it, rather than a thin layer on carbon tape, it was caked on. Furthermore, the powder turned out to be sub-micron in size and almost pure Sr-90! Static and air currents began to spread contamination, even before I got it in the chamber. I quickly left the area before I became "hot" too.

I did finish the exam... In anti-contamination dress with a full face respirator at the console! The job took a few hours. It took two weeks for me and my service engineer to disassemble the SEM, clean (and the room) it to reasonable levels and reassemble. The vacuum system was rather "warm" from that time on... Nevermore! Nevermore!

Moral: Always insist on making input and final approval if someone else wants to prepare your samples.

Woody White, McDermott Technology

This one is from grad school days. I was doing my thesis on the petrography and geochemistry of Miocene andesites as part of an international study on PreHellenic arc volcanism. Since I did all my own thin section preparations, I became the unpaid "volunteer" to keep the prep lab in good running condition, and to assist other students. Nothing compares to being an indentured servant. One day, a coastal sedimentary graduate student approached me. He was working on temporal barrier islands formed off the Gulf coast of Florida that formed as a result of Hurricane Helena in 1986. His major advisor thought it would be good for him make some impregnated thin sections from core samples, and stain them for carbonates and feldspars, map the distribution of them. I instructed him on all the staining procedures including safety procedures.....emphasizing safety procedure since he would be dealing with concentrated HF to etch the sections. I decided to stay in the lab to do some maintenance, and to keep an eye on him.

Good thing I did, for what happened next could have turned into a real sad disaster. The student knocked over his slide drying rack into the HF bath. Before I could say or do anything, he immersed both of his hands into the concentrated HF to save his sections. Fortunately for me, I had been on a volunteer rescue squad in my teens, and was fully trained for all sorts of accidents. He was lucky.....didn't lose his fingers, but did lose his finger nails, and his hands were scarred for life. Moral of this story? I should have done the procedure myself. I took on a potentially grave situation under my responsibility for a position I was not getting paid for, or properly insured for by the department. I am glad the fellow didn't suffer worse for his lack of thought, but I learned a valuable lesson, and held myself accountable and responsible for what happened. The student didn't.....

Lou Solebello

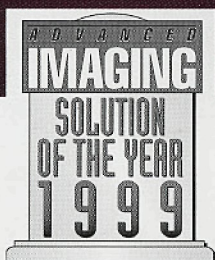
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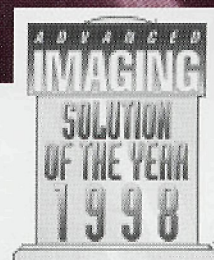


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Microscopy Horror Stories

Continued from page 20

Shortly after our laboratory purchased our first analytical electron microscope, the microscope developed a problem in the electronics. The microscopist telephoned the vendor's service department who requested he measure some voltages to help diagnose the problem. The microscopist dutifully attached clip-leads to the appropriate test points and was attaching them to the voltmeter when one of the clip-leads slipped off, fell into the electronics chassis, and shorted out much of the electronics. The vendor service engineers were in the lab for weeks repairing the problem. The rest of the lab teased him about this, giving the microscopist the nickname, "clip-lead". A few years later when he left the group for a promotion to a staff job, he was presented with a pair of clip-leads where the metallic ends had been coated with liquid-rubber.

John Minter, Eastman Kodak Company

Many years ago when communists were in power in Eastern Europe, thanks to a great person who knew how to deal with them, we got a beautiful piece of equipment, with all the possible stages. One of them - the heating stage - was especially impressive. All the people were amazed that *in situ* TEM heating experiment might be performed up to 1000 degrees centigrade. Among them was a young scientist who was investigating some processes in aluminum. Probably impressed by 1000 degrees centigrade, he forgot about melting temperature of metal. So, the new stage was required. At this time it was a real horror story since the price of the stage was almost equal the price of the small car - Fiat 126.

Witold Zielinski

My horror story is about 3 very competent service engineers, all from the same company, who each blew the window in the same EDX detector in their own turn. Engineer #1 was just unlucky, I believe. He was installing a STEM unit and the window just popped. Engineer #2 had disassembled the TEM goniometer and for some unknown reason turned on the roughing pumps. Evidently the inrush of air increased pressure on the window enough to cause it to pop. Engineer #3 just didn't listen to me. He had insisted that my detector bellows was causing a very small leak. He had taken the detector off 3 times, sure he would finally demonstrate that without the detector the TEM would hold vacuum. Each time, though, the vacuum would drift and he'd go find and solve another leak. After the third time, I told him not to mess with the detector anymore for fear he would break it. Two days later, after I returned from giving an out-of-town lecture, the detector was off the scope and the window was indeed blown...and the vacuum leak was still not solved. I ended up paying for a percentage of the last repair because the engineer had gotten permission to remove the detector from my trainee technician (2 months experience). The bellows was proved to be tight and a new butterfly valve solved the vacuum leak.

The companies and engineers remain nameless.

Chuck Bitterick, Engineered Carbons, Inc.

The End

Senior Research Technician

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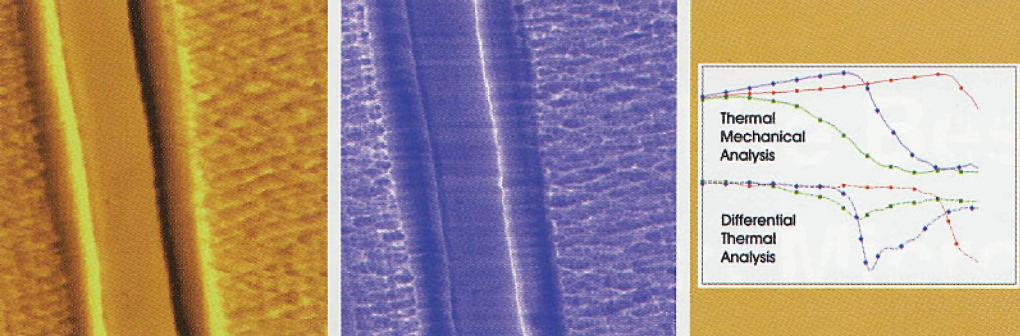
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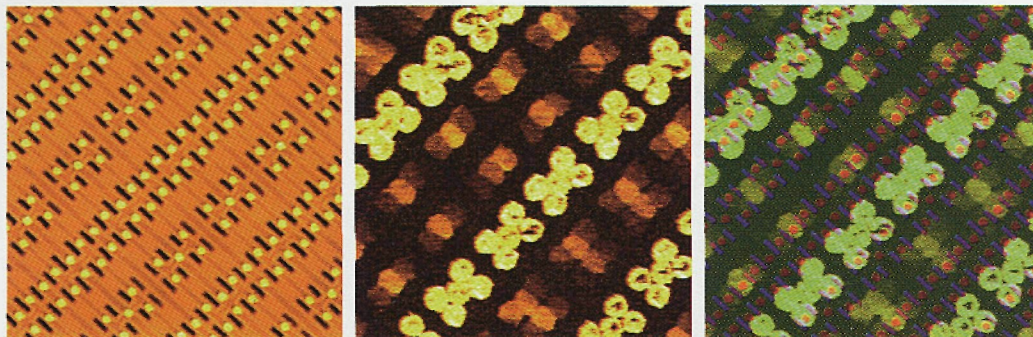
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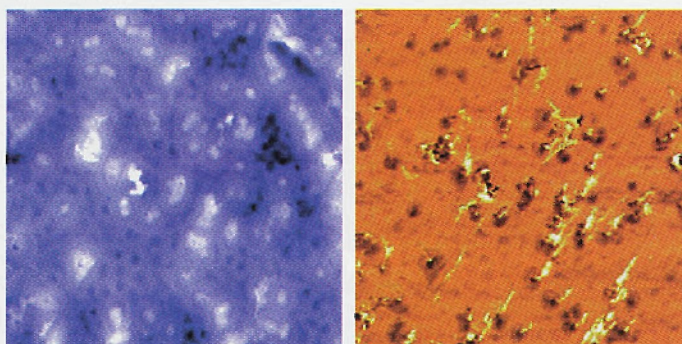


Using topography, thermal conductivity mapping and micro thermal analysis identifies layers in a polymer film. Courtesy of Duncan Price, Loughborough.



Topography and electrostatic force images combine to show misalignment of contacts and implants in SRAM.

When is an SPM



Topography and phase images show area and percent coverage of Teflon® and lubricant in support matrix. Courtesy of Steve Pratt, Kodak.

REGION GROUP	AREA	
	μm^2	%
Total area	67.69	100
Silicone lubricant	3.101	4.6
Teflon particles	7.007	10.4

more than a microscope?

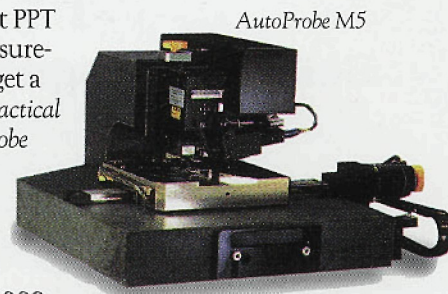
(When it has PPT.™)

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