Monitoring Electron Microscope Performance

Steve Chapman, Protrain, UK
protrain@compuserve.com

It is most important that any test specimens used to audit an instrument are from a traceable source and are certified by a recognised body.

Resolution Test Specimens

Scanning Electron Microscopes

Traditional methods for the evaluation of resolution in the scanning electron microscope rely upon a high-density particle upon a low-density substrate, the most popular method being evaporated gold on a carbon substrate. Although most manufacturers use this method, it is subject to abuse as there is no inbuilt magnification standard and therefore the evaluation of the image may only be made through measurement. To simply judge the specimen by appearance as most scientists do, could lead to a misleading result.

An ideal specimen for the evaluation of scanning electron microscope performance is dried polystyrene latex sputter coated with gold, or gold palladium for the evaluation of higher performance (*e.g.*, field emission instruments) (Figure1). The specimen requires very pure polystyrene latex that is allowed to settle and dry down over a period of time sufficient for it to form a solid block. The block is fixed to a specimen stub with silver Dag and the adhesive allowed to dry. The dry block of latex is pricked with a pin to open up the internal structure. The specimen is then sputter coated a number of times, for one minute at 1,000 V, 20 mA, with a specimen target distance of 5 cm. At least one minute is allowed between coatings and the procedure repeated for a total of 5 to 9 coatings.

This procedure produces a specimen that contains hexagonally packed latex spheres with gold structures and cracks on their surfaces. If the spheres are aligned to place the fracture in a flat plane, the image of the hexagonal packed areas anywhere on the specimen may be compared. In the opinion of the author, there is no need to measure the structures on the specimen, as a careful visual evaluation will display an improvement or degradation of the instrument's resolution, and the capabilities of the operator.

The specimen, unlike gold on carbon or gold on magnetic tape, has an inbuilt dimensional standard in that the latex particles are of a specific size (0.25 µm is ideal). This feature in its self simplifies the evaluation of test micrographs and is unique amongst scanning electron microscope test specimens. There is no doubt in the size of the structures because the spheres act as the magnification standard.

Transmission Electron Microscopes

In order to determine the performance of an operator there is no point using a crystal lattice test as used by the manufacturers to present their instrument capabilities. A lattice only tells if a certain resolution level has been reached, it does not tell how far short of that resolution one is, or even if a higher resolution is attained. The Fresnel fringe around a hole, however, provides a resolution figure from 5 nm down to 0.45 nm (Haine), and it is therefore this specimen that is ideal for a resolution test under the circumstances of this paper. In order to measure performance, the instrument will also require a magnification calibration at the same level. The test should be made in excess of 100,000X, and that will require a crysatile or similar crystal lattice specimen for calibration. The test specimens mentioned here are available from any electron microscope accessory organisation.

Energy Dispersive X-Ray Analytical Systems

A simple aluminium/copper or aluminium/cobalt standard, which is available from most accessory organisations, is sufficient for resolution testing. The aluminium offers a low electron volt calibration and resolution point with either copper or cobalt being used for middle range performance.

Performance Monitoring Procedures

It is not a criticism of electron microscopists if they are unable to take high resolution micrographs. Whilst many people are able to drive cars, few are able to race them successfully without prac-

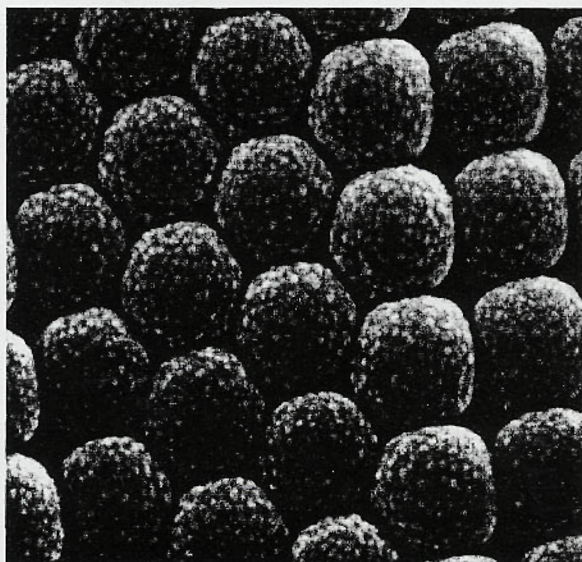


Figure 1: SEM Resolution Standard

IMAGE CONTROL INC.
"Your Image Is Our Concern"
P.O. Box 1858
New Port Richey, FL 34656
Phone: 727 844-3744 or 703 369-0021
Fax: 703 257-6321
Web: www.imagecontrolinc.com
TOPCON/ISI HITACHI
JEOL AMRAY
Service Contracts available at reasonable cost:
Unlimited Calls
Fixed Number of Calls
Hourly Rates
- Servicing Electron Microscopes since 1966
- Engineers located strategically thru the U.S.
- Prompt and economical service
- Move units room to room or state to state
- Quality prime used equipment for sale

CrossMark
https://doi.org/10.1017/S155192950005757X Published online by Cambridge University Press

tice! Therefore we may assume that if a microscopist is forced to take performance test pictures on a regular basis, their operating technique will improve.

Fitting a form of instant micrograph feature to the microscopes may make a further step in the direction of improving the quality of micrographs produced in a laboratory. In this way the microscopist will be able to assess each micrograph and make corrections to their procedure if necessary. In the case of scanning electron microscopes this requires the use of Polaroid or electronic image reproduction procedures. In the transmission electron microscope, the addition of a TV display facility will also offer the use of electronic image production procedures.

Determining Performance in Scanning Electron Microscopes

Resolution Test

Before testing the machine the instrument should be clean and set up in such a condition that the full potential of the instrument may be realised. In an instrument using a tungsten hairpin filament, the gun geometry required would not be that one would use for extended filament life. It requires the filament to be placed in such a position within the cathode that the emission current will be:

1) For a Japanese instrument around 90 to 110 µA above the standing current for that kilovoltage whilst the bias or emission current setting is at its half way position.

2) For a Camscan, around 120 to 150 µA (indicated as 1.2 to 1.5) above the standing current for that kilovoltage whilst the bias or emission current setting is at its half way position.

3) For a Philips, around 40 to 50 µA whilst the emission current setting is at its half way position.

4) Field emission instruments require an emission current of at least 10 µA.

Place the specimen in the microscope and select the accelerating voltage that is the subject of the test. Switch on the accelerating voltage and leave the instrument in this condition for one and a half hours to allow the high voltage tank to stabilise. Not until the heat gained by the components within the tank equals the heat lost through its walls will the high voltage reach maximum stability and offer maximum performance.

Investigate the specimen at the working distance selected for the test, less than 5 mm being ideal for a modern conical final lens. Look for areas on the specimen which display hexagonal packing in the perfectly flat form that is ideal for a resolution comparison. The magnification of the test micrograph should be at a level to test both instrument and the operator. On an instrument with a tungsten hairpin filament originally guaranteed for around 10 nm resolution, a magnification of 50,000 X is appropriate at 10 kV or more, where as 25,000X is more suited to accelerating voltages of less than 10 kV. For instruments able to attain better than 5 nm resolution, it is more appropriate to double these figures, and for field emission instruments only magnifications of four times these levels would offer a good test.

Take pictures of a fresh area within one screen width of that which was used to focus and correct the astigmatism. Move between these positions with the electrical deflection often known as "image shift", as a stage movement will unsettle the specimen and possibly change the focus. Do not dwell on the area of interest, as this will contaminate the specimen and soften the image. Instruments vary the size of the probe through the use of their condenser lenses. The appropriate control may be called spot size,

Continued on following page

STOP HASSLING WITH MULTIPLE SERVICE CONTRACTS!

START by putting all of your instruments under one service contract with MAS (regardless of make or model). Our expert **EM SERVICE GROUP** has the knowledge and skills to keep your instrument working at its best.

TEM'S / SEM'S	PREP EQUIPMENT	SPECIAL SERVICES
HITACHI, TOPCON, JEOL, ISI, AMRAY, CAMBRIDGE	VACUUM COATERS, PLASMA ASHERS, SPUTTER COATERS, ION MILLS	STAGES, BEAM BLANKERS, CUSTOM DEVICES

NEW NEW NEW

Authorized service representatives for Gatan preparation equipment and Topcon TEM'S.

Contracts and On-Demand Emergency Service at Reasonable Rates from Factory Trained Specialists.

800-421-8451

3945 Lakefield Court Suwanee, Georgia 30024 770-866-3200 FAX 770-866-3259
616 Hutton Street Suite 101 Raleigh, North Carolina 27606 919-829-7041 FAX 919-829-5518
ADVANCED ANALYTICAL PRODUCTS AND SERVICES

https://doi.org/10.1017/S155192950005731X Published online by Cambridge University Press

Monitoring Electron Microscope Performance

Continued from preceding page

condenser lens (c-lens) resolution or probe current; in this manuscript the term spot size will be used. Expect to use a spot size between 60% and the limit of the system when attempting to attain the highest resolution for a particular microscope. It is not possible to determine the ultimate resolution of the instrument other than by taking a series of pictures over a range of spot sizes, the changes are usually too subtle to observe on a conventional CRT. One would expect the image on the viewing CRT to be very noisy under the conditions required for maximum resolution, focus and astigmatism correction being made only by looking for maximum contrast. Under these conditions it is very unlikely that a clear image will be displayed, noise will dominate.

$$\text{Resolution} = \frac{\text{Measurement}}{\text{Calibrated Magnification}}$$

Magnification Calibration

Low magnification calibration standards are available in the form of a metal grating or the more abundant transmission electron microscope specimen support grids. The latter are usually well documented in accessory catalogues and offer very low cost but very accurate test specimens. There are two styles of standard that are applicable to the scanning electron microscope at medium to high magnifications, either a metal grating or a carbon replica of a grating (the transmission microscope test specimen) both 2160 lines per mm or 0.4629 µm. The difference is cost.

The test specimen should be placed in the microscope taking great care to ensure that it is sitting flat on the stage. Preliminary investigations in this area using a bubble spirit level are advised if one is not confident that the stage is truly flat when indicating zero degrees.

Switch off any accessories that interfere with the conventional scanning process and may lead to irregularities in magnification, for example scan rotation, dynamic focus and tilt correction. It is important that imaging media is fully understood. Most scanning electron microscopes offer a 1 to 1 image recording on 4" X 5" Polaroid film, but with any other media a change in this ratio is almost certain to take place. Full details of any ratio changes should be available in the microscope's instruction manual or from the supplier.

Accelerating voltage, working distance and spot size all play a role in the level of magnification being attained. Be aware that a spot size change will almost always give a magnification change, unless the manufacturer attempts to compensate changes of this type. A simple test of this feature is to focus at one spot size and, after switching to the next position, recheck the focus. Any change in focus indicates a need to make a focus correction that in turn will change the imaging magnification.

With any magnification standard the procedures are similar, only at magnifications in excess of 20,000X is the stability of the accelerating voltage likely to require consideration. Always focus and correct astigmatism at double the photographic magnification, switching back to the desired photographic magnification for setting the image intensity and taking the photograph. If possible, orient the specimen structure to sit up-down, left-right, so as to enable measurements of magnification in both X (left-right) and Y (up-down) directions. The specimen should be measured in each direction taking into account as many calibration units as possible.

$$\text{Magnification} = \frac{\text{Measurement}}{(\text{Number of units}) \times (\text{Size of one unit})}$$

Results should be within plus or minus 10% of the readout, with an X to Y comparison of no more than plus or minus 5%. The service engineer should correct errors in excess of this range.

Contamination Rate

The assessment of contamination rate in a scanning electron microscope is, unlike that of the transmission electron microscope, not a traditional test of performance. The main reason for this lack of procedure is that with the scanning electron microscope, the specimen itself is the biggest source of contamination. With the wide variety of specimens used within the scanning electron microscope, comparison of rates from instrument to instrument are rarely relevant. However in auditing a particular laboratory the author believes that contamination rate is a valid test of the instrument(s).

The transmission electron microscope test for contamination relies upon the build up of the contamination within a hole in a carbon film decreasing the size of the hole over a known time (Chapman). For the scanning electron microscope, the hole is substituted by a gold coated latex particle, in this case the contamination increasing the size of the particle over a known time.

Having inserted the specimen, wait for the instrument to stabilise. Because of the influence of the specimen on its environment, it should be pumped within the microscope for a specific time before commencing the test. One and a half hours, the period required to allow the instrument to stabilise, is ideal. Find an individual latex particle within the test specimen described earlier

Modern Coating Solutions for Today's EM Sample Prep Needs

Compact modern desktop systems with fast cycle times. Carbon and sputtering systems for all SEM, FE-SEM and TEM applications.

Cressington Scientific Instruments, Inc.
508 Thomson Park Drive, Cranberry Twp., PA 16066
TEL 800-755-1781 / 724-772-0220 FAX 724-772-0219
Website: www.cressington.com

and increase the magnification to similar levels as suggested for a resolution test of the instrument. Use the appropriate spot size and working distance that was used for the highest resolution. Take a photograph and note the time. Repeat the photograph having refocused and corrected the astigmatism after exactly twenty minutes.

$$\text{Contamination Rate} = \frac{\text{(Diameter of Large Sphere) - (Diameter of Small Sphere)}}{\text{(Time in Minutes) X 2 X (Calibrated Magnification)}}$$

The increase in the size of the sphere is determined within the calculation, then the rate of contamination, remembering that the contamination has deposited on both sides of the sphere requiring division by 2 to calculate the actual contamination rate on one edge in nm per minute. Expect a result between 2.5 nm/min and 10 nm/min depending upon the age of the instrument, state of the vacuum system and the type of specimen routinely used within the instrument.

Drift Rate

Another test not associated with the scanning electron microscope, the drift rate test is important at determining the loss of stability, hence performance, as an instrument ages.

The isolated sphere is again the subject of this test, the set up procedures being identical to that when taking a contamination rate picture. Once the instrument is stable and the specimen has had time to out-gas, the same high resolution conditions are used, with particular care being taken to ensure that the "final" aperture is well aligned. In this case, pictures are required at the commencement and at the end of the test but super imposed; a double exposure is required. Set up the instrument and take the first picture. Leave the instrument in exactly the same condition as set up for the photograph taking care not to touch any image movement controls. Repeat the photograph after 20 minutes. The problem here is that excessive focus change will change the magnification and any aperture misalignment will change the position of the sphere. Expose the second picture on the same piece of film and process the film.

Remember that most of the drift we see on a scanning electron microscope is due to specimen charge deflecting the beam. This results in apparent specimen movement, not true specimen movement!

$$\text{Drift rate} = \frac{\text{Distance moved by the sphere}}{\text{Magnification X (Time Minutes)}}$$

In our experience, the drift rate on an instrument in good condition is less than the resolution of the instrument which means even over a 20 minute period there should not be a discernible shift in the image. If image movement detected, find another area and perform the test again. Repeated evidence of movement suggests either a poor specimen or stage earth, or problems with the stage movement, all areas that need attention in a well run EM unit.

Determining Performance in Transmission Electron Microscopes

Prior to making any test of the machine, the instrument should be clean and set up in such a condition that the full potential of the instrument may be realised. This requires the filament to

Continued on following page

Crystal Clear.

The Meiji EM Series of Modular Stereo Microscopes.

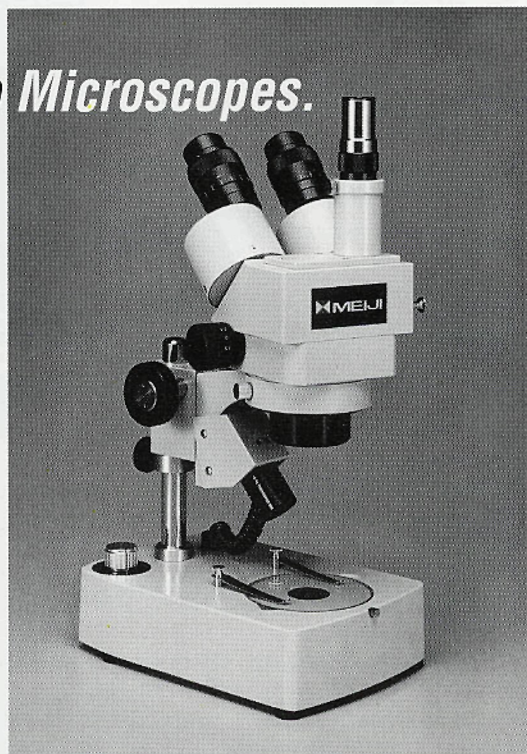
If you are looking for precision, durability, quality and value in a stereo microscope, we invite you to take a closer look at Meiji's EM Series of Stereo Microscopes.

The modular design (A wide variety of bodies, single magnification or zoom— rotatable 360°, auxiliary lenses, eyepieces, stands, holders, etc.) gives you the freedom to create the ideal instrument for your specific needs or application, and Meiji stands behind every instrument with its limited ***Lifetime Warranty.***

For more information on these economically priced stereo microscopes, please call, FAX or write us today.

MEIJI TECHNO AMERICA

2186 Bering Drive, San Jose, CA 95131, ***Toll Free Telephone: 800.832.0060***
FAX: 408.428.0472, Tel: 408.428.9654



https://doi.org/10.1017/S155192950005731X Published online by Cambridge University Press

Monitoring Electron Microscope Performance

Continued from preceding page

be placed in such a position, within the cathode, that at the selected accelerating voltage the emission current will be 30 to 50 μA (at 100 kV) with the bias or emission control approximately in the centre of its range.

Resolution Test

Place a holey carbon film in the microscope and select the accelerating voltage that is the subject of the test. Switch on the accelerating voltage and leave the instrument in this condition for at least one and a half hours to allow an oil filled high voltage tank, and the specimen rod, to stabilise. A gas filled high voltage tank will stabilise in a shorter time, probably within one hour. Not until the heat gained by the components within the tank equals the heat lost will the instrument reach maximum stability. During this period the specimen rod will be brought to exactly the same temperature as the surrounding components within the instrument, providing maximum stage stability.

Investigate the specimen at the eucentric position, with a spot size of around half to one micrometer and a magnification greater than 200,000X. Work with a hole about 2 cm across at this magnification; remember that only half a hole needs to be seen, as the other half will mimic what is seen in the field of view. Use an overfocus condenser setting for the pictures of a fresh hole having previously set the instrument up on another hole. Do not dwell on the area of interest as this will contaminate the specimen and soften the image. Expect to use a two to four second exposure. We believe it is not possible to obtain the highest resolution micrograph other than by taking a through focal series. The magnification should be calibrated at the same time by imaging one of the crysatile or carbon lattices.

Focus and correct the astigmatism making the fringe finer and finer until it can only just be detected all round the hole. The final focus should make the overfocus fringe only just visible, using this as the first exposure in a through focus series turning back towards underfocus. To judge the range required, count the number of steps between a just visible overfocus fringe (black) back to a just visible underfocus fringe (white). Divide this step figure by 5 and use this result as the number of focal steps between each of 6 exposures. The fringes should be measured in each direction. If the astigmatism is not correctly compensated, do not count the resolution test. A simple guide to performance is that 0.1 mm at 200,000X is equal to 0.5 nm resolution.

Should one desire to determine the highest possible resolution of the instrument, lowering the specimen further in the lens, increasing the lens strength and reducing the aberrations, will attain higher performance. This is achieved by adjusting the side entry rod until it requires a higher lens strength to focus (clockwise). Most eucentric systems allow the specimen to be lowered from that level by at least 1 mm.

$$\text{Resolution} = \frac{\text{Measurement (center black fringe to center white fringe}}{\text{Magnification}}$$

Magnification Calibration

Low magnification calibration standards are available in the form of the transmission electron microscope specimen support grids. The grids are usually well documented in accessory catalogues and offer low cost, but very accurate test specimens. There is only one standard that is applicable to the transmission electron microscope at medium to high magnifications: a carbon replica of a line grating usually 2160 lines per millimeter. At magnifications in excess of 80,000 X, it is better to use one of the crystal lattice test specimens which will be found in most accessory catalogues. Advice on obtaining the lattice resolution, and the lattices dimensions that may be imaged, are usually found with each specimen purchased.

Accelerating voltage and focal length play a role in the level of magnification being attained. Always set the specimen at the eucentric position to standardise the focal length. With any magnification standard the procedures are similar, only at magnifications in excess of 40,000X is the stability of the accelerating voltage likely to require consideration.

Always focus and correct astigmatism at double the photographic magnification switching back to the desired photographic magnification for setting the image intensity, setting the final focus and taking the photograph. The specimen should be measured in each direction taking into account as many calibration units as possible.

With crystal lattice observations, correct the astigmatism on the actual specimen structure and work just underfocus when looking for the lattice. Having found the lattice at higher levels of magnification drop down to the desired level, refocus, and then take the micrographs.

$$\text{Magnification} = \frac{\text{Measurement}}{\text{(Number of units) X (Size of one unit)}}$$

(for a grating 1 mm/2160= 0.4629 μm)

Results should be within plus or minus 5% of the readout; the service engineer should correct errors in excess of this range.

DIGITAL CAMERAS AND ACCESSORIES FOR MICROSCOPY!

Nikon. We take the world's greatest pictures.® Yours.

NATIONAL Graphic Supply

Nikon

800.223.7130 EXT.3109

226 North Allen Street, Albany, New York 12206 • Fax 800.832.2205
www.ngscorp.com • E-mail: scisales@ngscorp.com

https://doi.org/10.1017/S155192950005731X Published online by Cambridge University Press

Contamination Rate

The test for contamination relies upon the build up of the contamination within a hole in a carbon film decreasing the size of the hole over a known time. Having inserted the specimen, wait for the instrument to stabilise. Because of the influence of the specimen on its environment it should be pumped within the microscope for a specific time before commencing the test. One and a half-hours, the period required to allow the instrument to stabilise, is ideal. Find a hole within the test specimen that is about 1 to 2 cm across at the desired magnification. Work at similar levels to those suggested for a resolution test of the instrument. Use the appropriate spot size and working distance that was used for the highest resolution. Take a photograph and note the time. Repeat the photograph having refocused and corrected the astigmatism after exactly twenty minutes.

$$\text{Contamination Rate} = \frac{\text{(Diameter of Large Sphere) - (Diameter of Small Sphere)}}{\text{(Time in Minutes) x 2 X (Calibrated Magnification)}}$$

The decrease in the size of the hole is determined within the calculation, then the rate of contamination, remembering that the contamination has deposited on both sides of the hole requiring division by 2 to calculate the actual contamination rate on one edge in nm per minute. Expect a result between 6 nm/minute and 0.03 nm/min depending upon the age of the instrument, if liquid nitrogen traps are in use, the state of the vacuum system and the type of specimen routinely used within the instrument.

Drift Rate

The drift rate test, which once again uses a hole in a carbon film, is important at determining the loss of stability, hence performance, as an instrument ages.

Once the instrument is stable and the specimen has had time to out-gas, the same high resolution conditions are used as with a contamination rate test. In this case, pictures are required at the commencement and at the end of the test but if possible super imposed; a double exposure is required. Set up the instrument as discussed and take the first picture. Leave the instrument in exactly the same condition as it was for the photograph, taking care not to touch any image movement controls. Repeat the photograph after 20 minutes. Expose the second picture on the same piece of film and process the film.

$$\text{Drift Rate} = \frac{\text{Distance moved by the hole}}{\text{Magnification X (Time in Minutes)}}$$

Expect acceptable values to be in the range 12 nm/min to 0.6 nm/min. The higher the guaranteed performance of the instrument the greater the stability should be.

Determining Performance in Energy Dispersive X-Ray Analytical Systems

Resolution

Using a fixed kV, spot size, working distance (TEM focal length), tilt, counts per second, live time and processing time, the full width at half max for each peak is measured to determine the spectrometer resolution. The aluminium/manganese or aluminium/cobalt or aluminium/copper specimens check performance in the most commonly used areas of the spectrum. Aluminium with a windowed detector will record around 120 eV with manganese about 10 eV greater than the figure given for the spectrometer, cobalt and copper a little more. The reason for the manganese variation is that the EDX manufacturers use a manganese x-ray source to determine the detector resolution, not an electron beam induced signal.

Quantification

A standard specimen should be obtained which contains known quantities of material of similar levels to the unknown materials analysed in the laboratory. In this way the complete quantification procedure will be checked for accuracy. If rough surfaces or powders are routinely investigated, a standard in these forms should also be considered.

Conclusions

The author in the role of an electron microscopy consultant trains electron microscopists and uses microscopes in most English speaking countries of the world. It is clear from experiences in laboratories specialising in electron microscopy that those who oversee these laboratories rarely check the operating standards. Electron microscopy does not have a "quality" procedure; too many actions are carried out without criticism, which at best result in a very slow level of improvement in technique, if any!

Following the procedures outlined in this paper has been proven to improve the "quality" of results produced by a department. Also such a well-defined quality structure will place pressure upon internal and external (service) staff and should result in an improvement in the performance level of the instrumentation. If this is not the case, discussion must take place with the senior manager of the service organisation to improve the level of instrument service. The experience of the author as an electron microscope service engineer allows the judgment that an engineer will maintain an instrument to a level just above the capabilities of the laboratory in question. Improve the laboratory's capabilities and those of the instrument will improve also.

It is surprising how much commitment is given when people are being measured; can you imagine a golf course with no holes? When a measurement system is set up the audit team must possess a great deal of integrity and earn respect for fairness and consistency from the auditees. ■

**Electron Microscopy
Lehigh University**

Lehigh University seeks an Electron Microscopy Technician to perform duties in support of the Microscopy Center of the Materials Science and Engineering Department. The person appointed will work with other technical staff to instruct students in the operation of microscopes and other equipment; maintain and repair instruments; carry out upkeep of the lab; support research professors and students; analyze samples; give tours and demonstrations; maintain a safe environment and perform other assigned duties. A bachelor's degree in physical science and/or 4+ years related work experience required. Candidate should be familiar with electron microscopes, mechanical and electronic equipment, vacuum systems, computers (PC and/or Mac) and EDS/WDS systems. Experience with a microprobe would be especially valuable. Good communication and interpersonal skills are essential.

Lehigh University offers excellent benefits including medical, vision and tuition. Interested candidates should forward their resume to Jennifer Mohney, Human Resources, 428 Brodhead Avenue, Lehigh University, Bethlehem, PA 18015 EEO/AA

https://doi.org/10.1017/S155192950007731X Published online by Cambridge University Press