Initial Instrument Evaluation And Performance Monitoring For SEMs

Steve Chapman, Protrain

Microscopes in an electron microscope laboratory are built to be within specific tolerances in relation to resolution and magnification. The final specification from the manufacturer should include detail of magnification accuracy, drift rate, contamination rate and resolution. On delivery, the onus is on the installation engineer to confirm that the microscope meets specification. However, the final responsibility for microscope acceptance to specifications is the responsibility of the lab management.. Energy dispersive instrumentation is also specified as to specific resolution; and should also be confirmed on installation.

In some cases, those with final responsibility for equipment performance might not resolve with the manufacturer's representee, at installation, that the instrumentation meets all specifications.

Scanning Electron Microscopes

Resolution Test Specimens

Most instruments are set up incorrectly. The electron gun is always in economy mode, *i.e.*, the filament is too far from the cathode to enable specification resolution to be attained. Correct this problem or tune the gun further, and see how many old instruments are capable of beating their specification resolution. I use a test specimen we have developed for this.

Traditional methods for the evaluation of resolution in the scanning electron microscope rely on high-density particles 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 built-in 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 for the evaluation of higher performance (*e.g.,* field emission instruments) gold palladium. The specimen requires very pure polystyrene latex, which is allowed to settle and dry over a sufficient period of time for it to form a solid block. The block is fixed to a specimen stub with silver Dag and the adhesive is 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 volts, 20 mA, with a specimen target distance of 5 cms. At least one minute is allowed between coatings and the procedure is repeated for a total of 5 to 9 coatings.

This procedure produces a specimen that contains hexagonal packed latex spheres with gold structures and cracks on their surface. 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 authors, there is no need to measure the structures on the specimen as a careful visual evaluation will display 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 a built-in dimensional standard, in that the latex particles are of a specific size. This feature simplifies the evaluation of test micrographs and is unique among scanning electron microscope resolution test specimens. There is no doubt in the size of the structures because the spheres act as the magnification standard.

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 practice! We may assume that if microscopists

Continued on Page 32

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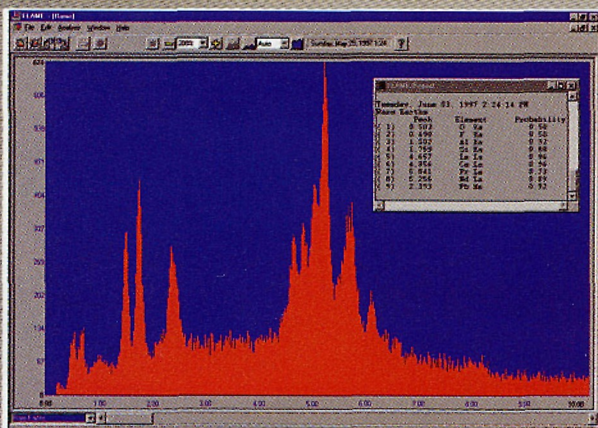

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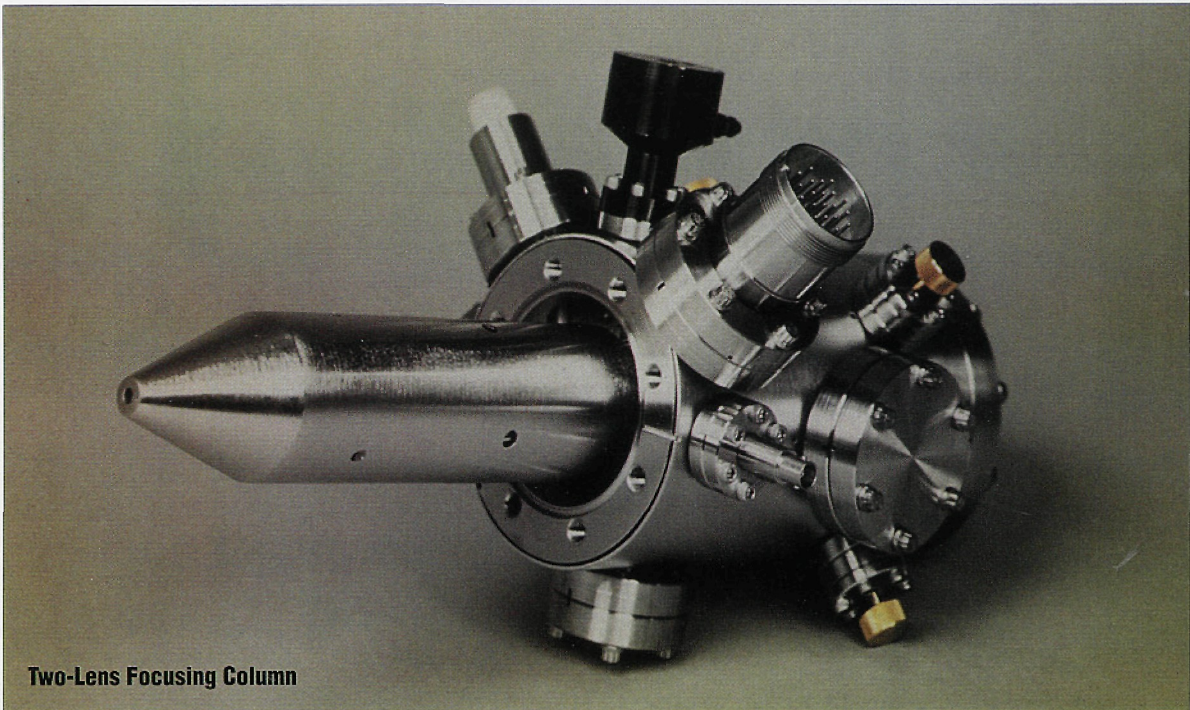
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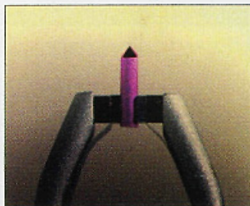

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are forced to take performance test pictures on a regular basis, their operating technique will improve.

Fitting a form of instant micrograph feature to the microscopes may be 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 procedure, if necessary. The instant feature is backed by the conventional photographic emulsion, once the ideal conditions for the micrograph have been achieved. In the case of scanning electron microscopes, this requires the use of Polaroid or electronic image reproduction procedures.

Determining Performance in Scanning Electron Microscopes

Resolution Test

Before testing the machine, the instrument should be clean and set up so that the full potential of the instrument may be realized. In an instrument using a tungsten hairpin filament, this will not be gun geometry for extended filament life. It requires the filament be placed within the cathode so that the emission current will be with the bias or emission control in the center of its range, as follows:

1) For a Japanese instrument, around 90 to 100 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 μA whilst the emission current selling 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 one half hours to allow the high voltage tank to stabilize. 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, they are 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 50,000X 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 4 nm resolution, it is more appropriate to double these magnifications, 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 you used to focus and correct the astigmatism. Move between these positions with the electrical deflection often known as "image shift", as 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, and the appropriate control may be called spot size, condenser lens (c-lens), resolution or probe current. In this manuscript the term spot size/probe current will be used. You should expect to use a spot size/probe current near to the limit of the system when attempting to attain the highest resolution for a

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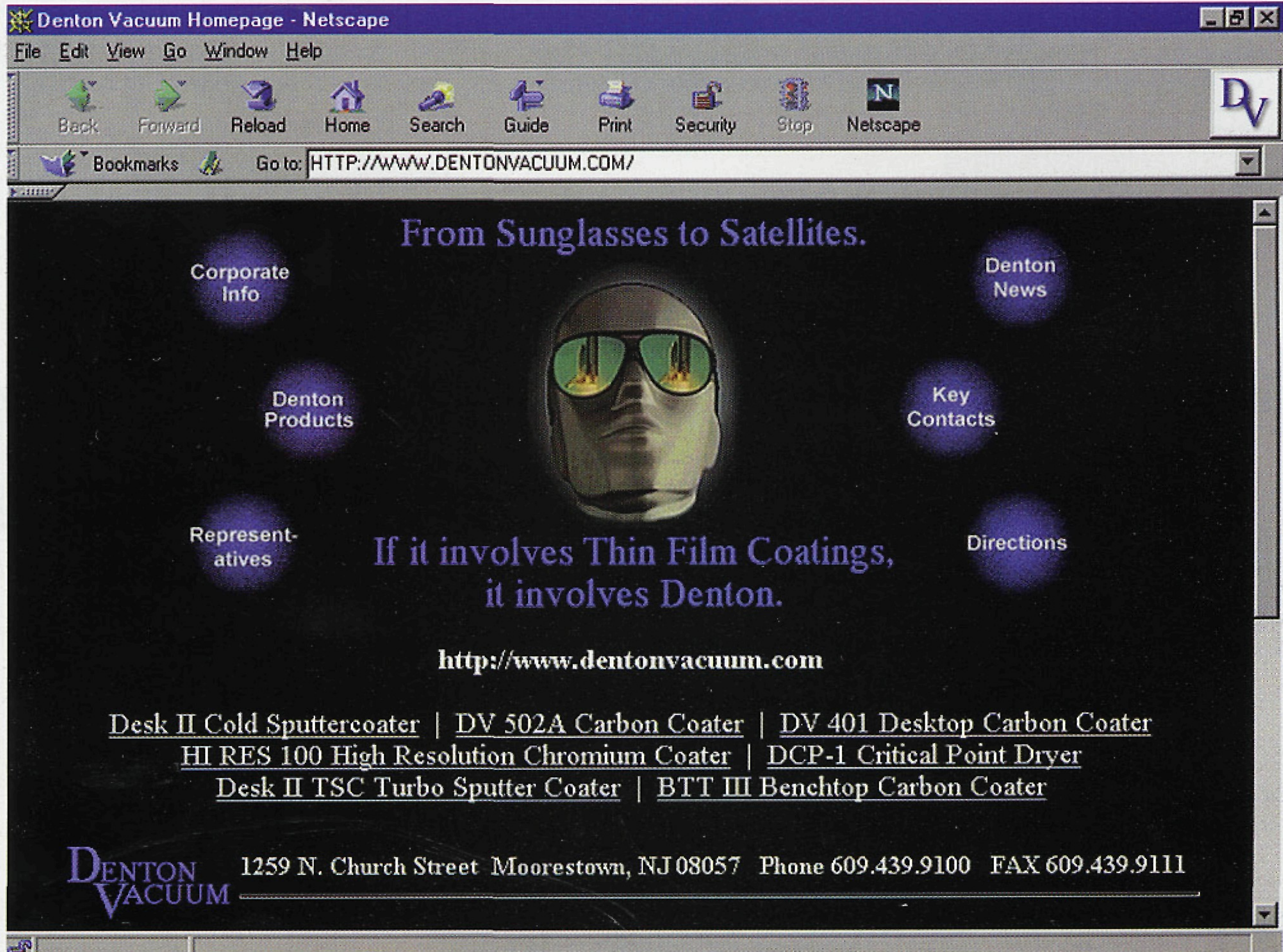

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Initial Instrument Evaluation And Performance Monitoring for SEMs - Continued from Page 32

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 size/probe currents, as 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, and noise will dominate.

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 catalogs and offer very low cost, but very accurate test specimens. There are two styles of standards 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). The difference is cost.

I use an SPI TEM carbon grating replica, a cross grating of 2,160 lines per millimeter. I prefer this specimen as it also makes a very good demonstration specimen on the effect of kV on image form (see *Working With a SEM* by S.K Chapman ISBN 0-850770-93-9).

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 you are not confident that the stage is truly flat when indicating zero degrees.

Switch off any accessories that interfere with the conventional scanning process and that may lead to irregularities in magnification, for example scan rotation, dynamic focus and tilt correction. It is important that you fully understand your imaging media. 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 instruction manual or from the supplier.

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

With any magnification standard the procedures are similar; only at magnifications in excess of 20,000 X 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.

Mag = (length max. No. squares in field) / (No. squares in field X size one square)

For 2,160 lines per mm, the size of one square is 1 mm divided by 2,160 which equals 0.4629 µm.

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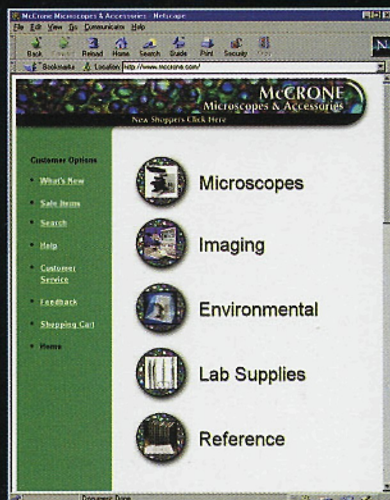

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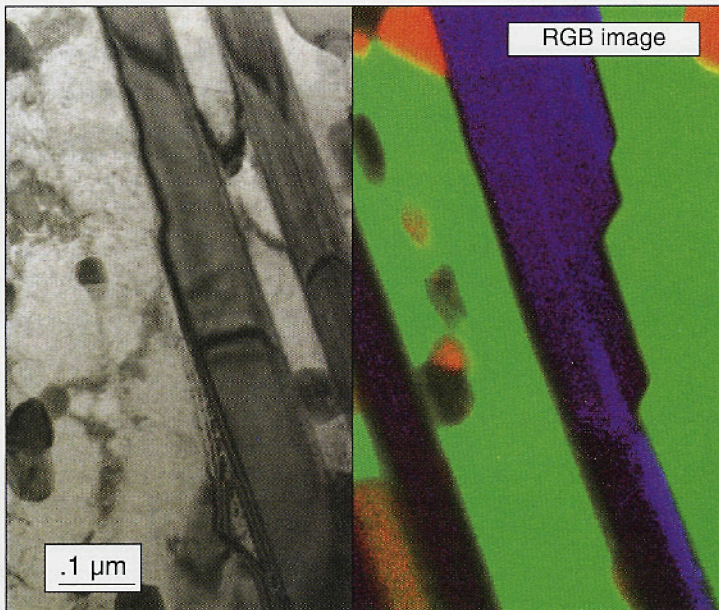
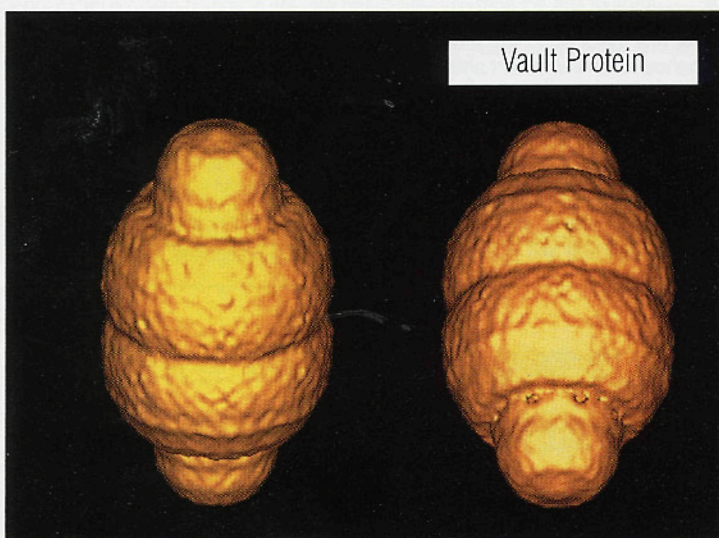
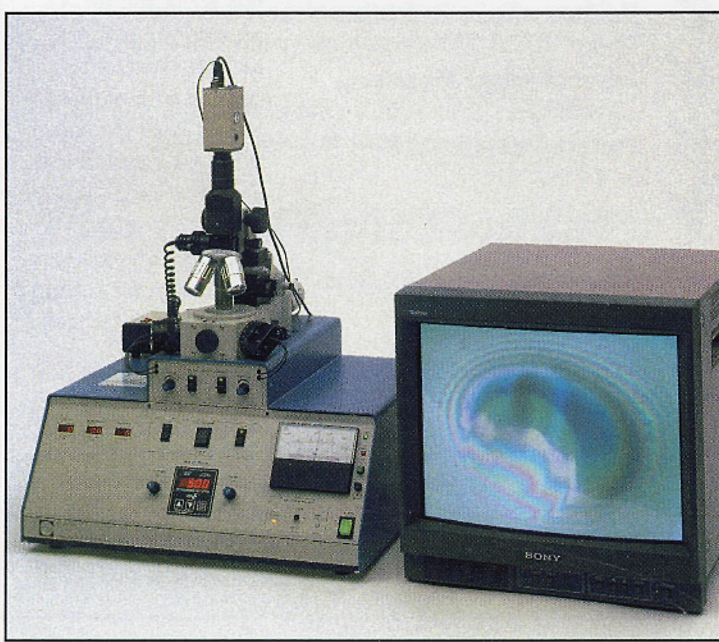


Color composite maps constructed with Gatan EFTEM software make it easy to distinguish between different kinds of carbides in high speed steel. Images were taken with a GIF 200 at FELMI TU-Graz, Austria.





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Initial Instrument Evaluation And Performance Monitoring for SEMs - Continued from Page 34

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%. Errors in excess of this range should be corrected by the service engineer.

It is my experience that on some SEMs the magnification calibration is very good at certain kV, but bad at other kVs. Machines also seem good at certain working distances, but not at others. In courses, each student measures each picture and we have a spread of 4 to 7% amongst them! It is not that easy to calibrate a SEM!

New tests on instruments less than 5 years old are very good. except they fall off in accuracy at longer working distances.

Contamination

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 SEM, the specimen itself is the biggest source of contamination. With the wide variety of specimens used within the SEM, comparison of rates from instrument to instrument are rarely relevant. However in auditing a particular laboratory, I believe that contamination rate is a valid test of the instrument(s).

The transmission electron microscope test for contamination relies upon the build-up of contamination within a hole in a carbon film, decreasing the size of the hole over a known time (Chapman). For the SEM, the hole is substituted with 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 stabilize. 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 one half hours, the period required to allow the instrument to stabilize, is ideal. Find an individual latex particle within the test specimen described earlier, 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 you 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.

Contamination rate in nm/min = $(D_L - D_S) / (T_{min} \times 2 \times \text{calibrated magnification})$

D_L = Diameter of Large Sphere (sphere at end of test)

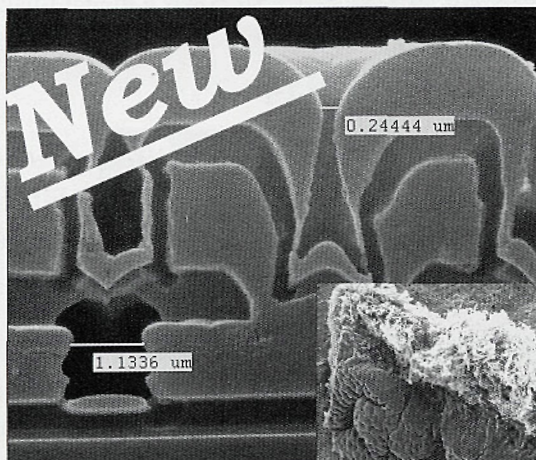
D_S= Diameter of Small Sphere (sphere at start of test)

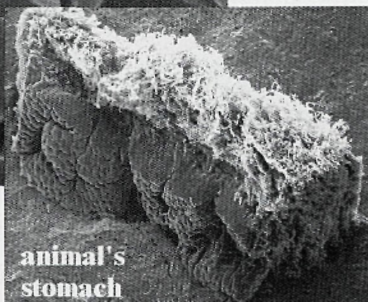
The increase in the size of the sphere is determined within the calculation, then the rate of contamination, remembering that the contamination has deposited on both sides of the sphere requiring division by 2 to calculate the actual contamination rate on one edge in nm per minute. Expect a result between 2.5 nm/minute and 10 nm/minute, depending upon the age of the instrument, state of the vacuum system and the type of specimen routinely used within the instrument. Add an airlock and a cold finger around the final lens, similar to that used in a cryo system, and the rate decreases to <1.5 nm/minute.

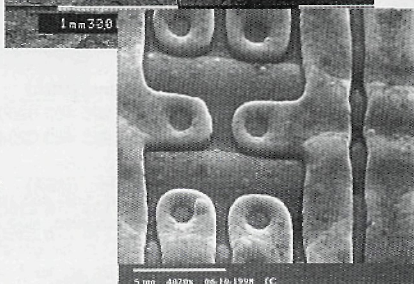
Drift Rate

Another test not normally associated with the scanning electron microscope, the drift rate test is important in 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 outgas 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







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https://doi.org/10.1017/S1551929500067821 Published online by Cambridge University Press

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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 also 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!

Drift rate in nm/min = (distance moved by sphere) / (calibrated magnification X time in minutes)

In our experience, the drift rate on an instrument in good condition is less than the resolution of the instrument. This means that even over a 20 minute period there should not be a discernible shift in the image. If you do detect image movement, 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..

References:

Chapman S. K. 1986. Checking the performance of the microscope. In: *Maintaining and Monitoring The Transmission Electron Microscope* (Royal Microscopical Society Handbook 08). Oxford University Press. ISBN 0-19-856407-4.

Important note: Specimen Selection

It is most important that any test specimens used to audit an instrument *within a quality regime* are from a traceable source and are certified by a recognized body.

In this Protrain guide to improving a laboratory performance, we are suggesting that staff and instruments are assessed using non-traceable standards, but only as a starting point in determining the performance of instruments and staff.

This article is part of "Quality in Electron Microscopy" by Steve Chapman, Tony Bruton and Paul Harding, a guideline booklet produced by Protrain. ■

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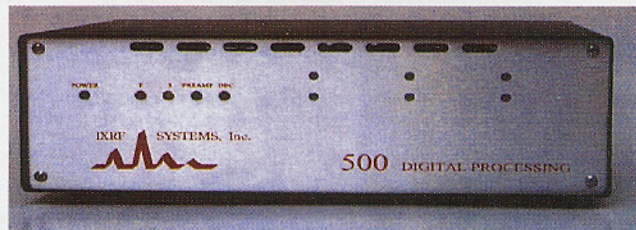

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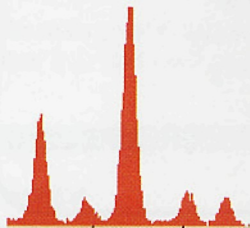

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