SEM

Understanding & Optimising Scanning Electron Microscope Performance

Whilst the scanning electron microscope with the help of modern computing and sophisticated imaging systems has developed into an instrument capable of imaging almost any structure, the techniques used by operators have not always progressed at the same rate. Sophisticated computers and the user friendly interfaces may appear to simplify operation, but a basic understanding of the beam-specimen reaction is frequently ignored, possibly spoiling the information content of the image. For example the selection of accelerating voltage is extremely critical, it determines the level of penetration and the signal balance between the surface and the sub surface. The signal contributions that most satisfy the operator's demands will also be related to the working distance selected; this will vary the mix of signals changing the information displayed. Microscopists are scientists, who are defined as being people who experiment; therefore we should experiment with the parameters within the SEM. Once the instrument is optimised, adjustments to display the data with appropriate grey levels will further maximise the information.

15kV X2,000 10µm 13 40 SEI

The scanning electron microscope was first made available commercially by Cambridge Scientific Instruments in 1966, with a performance level of around 100nm. The SEM, as it became known, was the first instrument developed as a scanning microscope. In scanning microscopy an illuminating probe is scanned or rastered across a specimen synchronous with the beam scanning across a cathode ray tube, the signals generated being used to drive the CRTs illumination; this is known as intensity modulation. Magnification is a simple relationship between the length of the scan line on the specimen and the length of the scan line on the display or recording media.

More than 40 years on the SEM has developed into an extremely important scientific instrument being used world wide to study all things from a fly's eye, through the development of medical wipes, to failure analysis, the results of which for example keep our new generation of passenger planes in the sky.

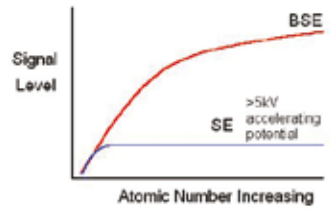

Fig. 1. Coefficients of secondary and backscattered emission at accelerating voltages typically used in SEM.

Whilst the early instruments were analogue, with images on cathode ray tubes and knobs as the controlling device, the instrument has benefited from the developments in computing. All modern SEM are digital, being controlled by computers, which in turn are controlled by the operator through a mouse or a desk panel. The computing not only enables all of the instruments facilities to be varied and memorised but it also has a considerable effect upon the final image quality.

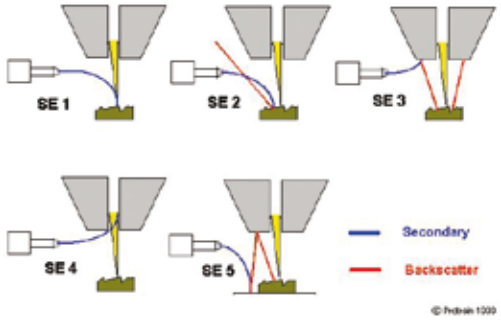

Fig. 2. The 5 major sources of SE signals from materials in a SEM.

In the early days of SEM the image was recorded on film, 35mm or 120 formats, before Polaroid became the preferred media for many investigations. The major problem with the SEM has been trying to relate the quality of the recorded image to the inferior image presented on the viewing cathode ray tube. Polaroid enabled the operator to obtain an instant image and therefore its true quality could be assessed and adjustments made to correct any inadequacies. Today the instruments rarely display a raw image, almost all images being displayed with a degree of computer processing and enhancement.

> *As most operators run their instrument in the, so called, SE mode, we should consider what happens to the signals leaving the specimen.*

Thus we see scanning electron microscopy in the twenty first century as computer controlled, image enhanced and based on the world enveloping Windows criteria. But one thing has gone wrong! Just because an operator understands Windows it does not mean that they are automatically able to obtain high quality information from their materials in the SEM. All the computing in the world cannot make the critical decisions that an operator needs to make, which accelerating voltage to use, at what working distance, with which spot size, using which detector, does the microscope model itself make a difference and at what magnification will the images in a report be?

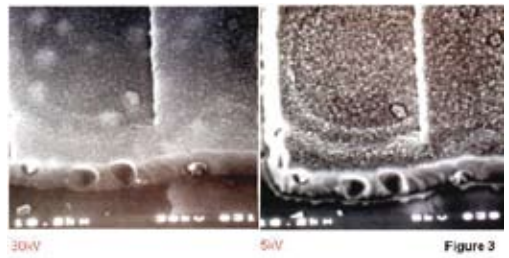

Fig. 3. A Microcircuit viewed at different accelerating voltages demonstrating on the left a high degree of penetration bringing signals from dense areas (bright spheres) deep within the material. On the right at a lower accelerating voltage sub surface detail is lost and the true surface information enhanced.

Whilst in many laboratories the transmission electron microscope is worshipped due to its apparent complexity and dominating size, in truth the scanning electron microscope is by far the more complex instrument. Why does a manufacturer of a SEM provide the operator with 100volts to 30kV in 100volt steps, surely it makes the instrument far more complicated? It would be easier to provide the operator with far less steps, just like the TEM, but the problem is that to obtain images that truly describe the specimen you may need the extensive accelerating voltage range provided.

Fig. 4. A shell exhibiting strong line of sight BSE contributions to the image when its surface pointed towards the top right of the display and darker areas shadowed from direct BSE contribution.

In order to fully understand the SEM and the electron beam's reaction with our specimens, the signals emitted from the specimen need to be considered.

Fig. 5. Fractured polystyrene latex surface in an instrument where the SE detector was placed at the top centre of the display. The topography causes shadows to be created due to the inability of BSE to contribute line of sight signal from those areas.

Most operators are aware that the incident beam liberates low energy secondary electrons (SE) from the specimen surface and that higher energy backscattered electrons (BSE) are also liberated. In my experience the problems arise when an operator fails to understand that, whist they may have the SE detector switched on, there is usually quite a large signal contribution from the BSE. The two signals contribute in relation to image contrast in different ways, thus their contribution to the data we derive

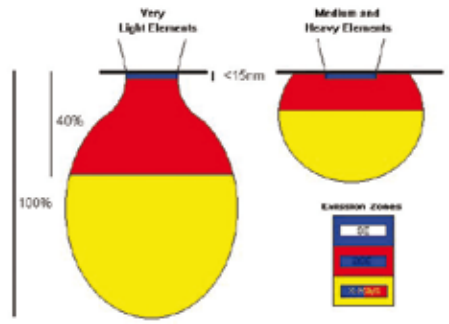

Fig. 6. Examples of penetration depth and signal zones in light and heavy materials.

from the material under investigation may be critical. Figure 1 shows the relationship between SE and BSE signals at the accelerating voltages typically used in SEM. SE emission hardly changes with material density but BSE offer atomic number contrast, an increase in signal with material density.

As most operators run their instrument in the, so called, SE mode, we should consider what happens to the signals leaving the specimen. Figure 2 demonstrates the five routes by which SE may be generated and be attracted into the "so called" SE detector.

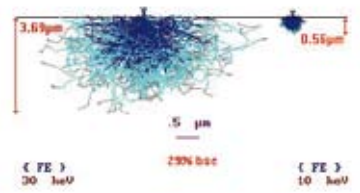

Fig. 7. Monte Carlo simulations for Iron at 30 and 10kV.

SE1 are the electrons everyone expects to collect, electrons liberated by the incident electron beam entering the material. SE2 are the electrons liberated by BSE exiting the material. BSE have energies up to the values of the incident beam and thus may act on surfaces as they exit in the same way as the incident beam as it enters. SE3 are electrons that have been generated by the BSE striking the components of the chamber, converting the BSE signal into SE, which then display areas of high BSE within the specimen. The geometry of the chamber in a particular instrument does contribute to the image form. Variables like; is the BSE detector fixed on the base of the lens, is the specimen tilted, what is the working distance, how many other units fit into the specimen chamber space, each of these features have an effect on the signals contributing or detracting from the image quality. The left hand micrograph in Figure 3 demonstrates the SE3 contribution at the higher accelerating voltage. Dense materials within the microcircuit show up as bright areas due to the converted BSE from the dense materials contributing a high level of SE.

The right hand image displays only the surface detail through reducing the accelerating voltage and therefore the beam penetration depth; it is made up of SE with a minimal contribution from converted BSE. SE4 and SE5, whilst of limited contribution, may carry most surprises as they may come from areas well away from the specimen under investigation. As well as these SE contributions the so called SE detector is unable to distinguish between SE and BSE signals, thus it does process both if they are present. The result of this lack of discrimination is often an excess of signal on faces pointing towards the detector and shadows cast on faces pointing away from the detector. In theory the image from a so called SE detector should be of even intensity as the signal is actually attracted into the detector. It is the presence of BSE data, direct or converted to SE, which provides us with the strong contrasts and apparent third dimension within the image. Figure 4 demonstrates the strong BSE contribution to the image when the material surface pointed towards the top right of the display; the position of the SE detector. The figure also displays the apparent third dimension due to the image probably containing as much as 40% of its information from BSE or converted BSE. Figure 5 demonstrates the "shadow casting" from areas devoid of BSE line of sight contribution due to the surface contours.

The signal contributions that most satisfy the operator's demands must also be related to the working distance selected; this will also vary the levels of each signal outlined above. Thus as scientists, people who experiment, we should vary the parameters within the SEM to optimise information and then display this information with appropriate grey levels to maximise that information.

Figure 3 clearly demonstrates the effect of accelerating voltage on image form. Few operators fully understand that the first decision that they will make when using a SEM could probably be the most vital: which accelerating voltage to use?

> *When trying to understand the ideal conditions for the investigation of a specimen a number of points are critical.*

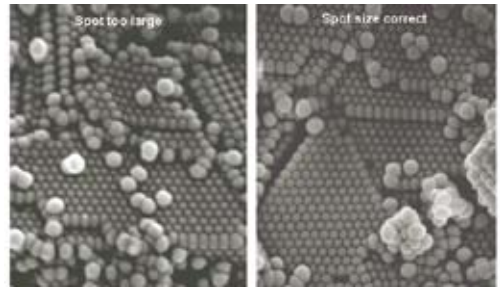

Fig. 8. The micrographs demonstrate that when a spot is too large the transition from one grey level to another is not well defined giving the impression that the image is not in focus. The left hand image is spot size limited but is rich in the apparent third dimension due to a high level of converted BSE being present in the SE image. In contrast the right hand image looks rather flat as it is being made up of SE which as they only emitted from the very surface of the material do not exhibit the apparent third dimension.

When trying to understand the ideal conditions for the investigation of a specimen a number of points are critical.

(1) What are you looking for, true surface, sub surface, or a controlled mix of the two? Imaging signals in terms of electrons are collected from approximately 40% of the total beam penetration (Figure 6). Using a simple Monte Carlo simulation you may derive the relationship between accelerating voltage, penetration and signal source. Using such a calculation carbon at 30kV with a total penetration of 9.25µm provides imaging signals up to 3.6µm into the specimen, but with iron at 30kV the beam only provides imaging signals from 1.4µm into the material (Figure 7). Lowering the accelerating voltage considerably reduces sub surface information due to a dramatic decrease in the volume from which BSE are produced.

(2) Having answered question (1) the operator must select the accelerating voltage. Light elements will not prevent beam penetration as well as heavy elements therefore true surface evaluation of light element materials should be constrained to low voltages. Biological material and aluminium alloys are best investigated at less than 5kV, but as you will learn later this figure should also relate to the structure of the material.

(3) Once the accelerating voltage has been selected a decision on the level of electron emission from the gun must be made (using the bias control). If the specimen is fragile, or prone to charge, or you do not know what will happen, it is best to start at a low emission current at a low accelerating voltage, perhaps 20 to 40µA at 2kV with a tungsten hairpin source. However if you intend to attain the highest resolution possible for the selected accelerating voltage you will need at least 80 to 120µA with a tungsten hairpin source. If you are unable to reach these high current settings the filament is positioned too far back in the cathode assembly.

(4) Where will you place the specimen within the microscope, what working distance (final lens to specimen distance)? A starting point if you have to find the specimen is the longest working distance possible; this will provide you with the lowest magnification. Once the desired area is selected move to a more reasonable general operating position of 15mm working distance, moving closer if resolution becomes a problem.

(5) Spot size or Probe Current adjustment is also critical. Too large a spot and you will find the image "spot size limited"; the spot is too large to clearly define contrast changes (Figure 8). However large spot sizes encourage a higher emission of BSE which, because of the depth from which they derive, contribute an apparent third dimension to the image. Take care with fragile specimens as too large a spot size may also damage a specimen. However if the specimen will take a large spot use this at low magnifications as this will provide you with a strong signal when looking for the important areas of your specimen.

Once the filament heating and bias are adjusted for the task in hand and the gun aligned the general operating procedures are basically repetitive. When you have found an area of interest, move to double the magnification that you intend to use to record

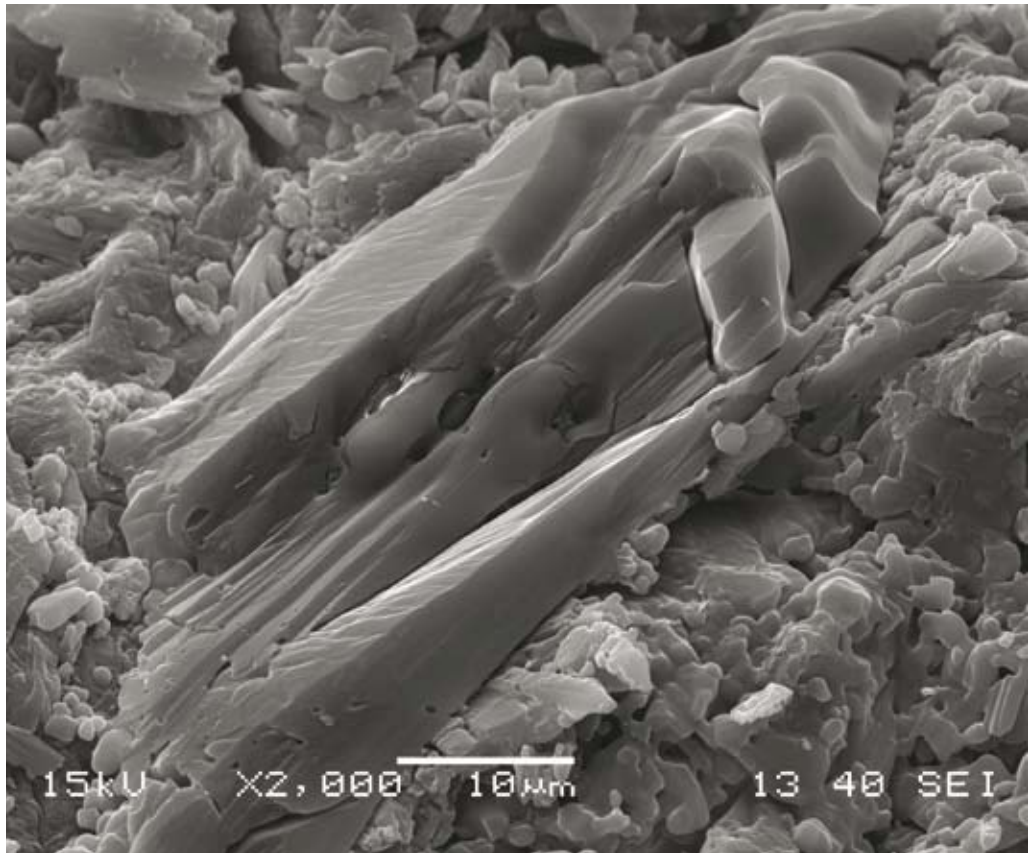

Fig. 9. A micrograph with a well presented "Grey Scale". There is a minimum of true white and a minimum of true black whilst containing a wide grey scale range.

the image: if that figure is less than 3,000X move to 3,000X. Doubling the magnification ensures that any errors in your adjustments will be minimised at the recording magnification. You need to be at or above 3,000X before you will see any astigmatism correction clearly. Focus and correct the astigmatism, always find focus first and do not try to correct astigmatism when you can see it, as this is an indication that you are out of focus; the result is prolonging the correction process! Check the quality of your correction by moving slightly out of focus, the image should blur evenly in all directions. Run a slow scan to check the image quality because fast scans hide information. If the image is good drop to the record magnification and move on. If the image is not good, reduce the spot size (probe current) and repeat the focus, astigmatism and slow scan process until the image viewed is satisfactory.

If high resolution is required a major improvement in image quality will almost certainly be found by shortening the working distance.

> *Now that you are recording images with a good grey range it is time to be critical of the images.*

Recording an image correctly makes or breaks the image. A good micrograph should contain a little true white and a little true black with as many grey levels as possible. The more grey levels you have the more information you contain; a black and white image only has two levels of information!

Figure 9 demonstrates the range of grey levels desired for a nice image recording. How to obtain a consistent quality in your image recordings should not be down to guess work! Almost certainly your final image will be produced on an ink jet or laser printer; it is this that you need to calibrate for your images. All SEM have an ability to present one line of information in the form of an intensity trace; often called graph, line mode or wave form, figure 10. Using this facility the brightness and contrast of the image may be adjusted for each image, but this must be in relation to the final result on your printer, not necessarily the SEM display! A good starting point is a trace half way up the display with a peak to peak of 2 cms. Record an image and print this on the desired imaging printer. If the grey scale is not correct readjust the levels, record and check again. When you are satisfied with the image quality note the trace position on the display for future use. A piece of tape attached to the side of the monitor to mark the top and bottom of the peaks is the solution taken by many operators. However the ideal solution is to set the automated brightness and contrast system to the desired recording trace position.

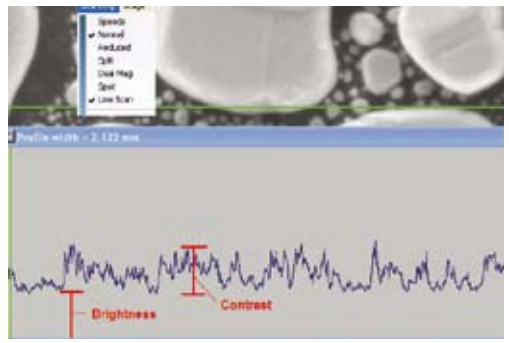

Fig. 10. A Line Scan and a signal "Intensity Display". The distance of a point on the trace from the base line is related to the brightness level, the peak to peak relates to the contrast level within the image.

Now that you are recording images with a good grey range it is time to be critical of the images. All of the images shown up to this point were produced by clients on Protrain courses; the clients are learning, so not all of the micrographs are perfect and none of them were prepared for publication.

As a general guide there should be no areas on the image that display as being smooth. Smooth means no information, which means that the accelerating voltage is too high and too much penetration. Only the smallest areas should be white as white means no more information; if possible the accelerating voltage should be tuned down to reduce white areas. Even with the nice grey scale in Figure 9 you will see that many areas are smooth and therefore do not provide the maximum amount of information; the accelerating voltage is a little too high!

Great care should be taken so as not to swamp the surface with sub surface information if it's the true surface that you require. Start at a low accelerating voltage (~2kV) and increase the voltage until the image changes in texture; now you are starting to look at the under surface. If you have not seen contamination you have yet to see the true surface of your image. Too high an accelerating voltage and you punch through the surface and the surface contamination visualising the under surface. However there may be information under the surface that would contribute to the investigation? It is most important if you wish to obtain the maximum information from the specimen that you vary the accelerating voltage and collect images to judge which contains the best display of the information that you desire.

Scanning electron microscopy is an exciting constantly changing challenge. New instruments continue to defy the original resolution barrier and developments in different types of detector are opening up even more exciting ways of extracting information from our specimens. But none of this effort is worth while if we do not try to maximise the information we obtain and the way we display it!

The next three figures relate to problems often visualised in SEM images. None of the micrographs were prepared for publication, they were derived from different clients on different instruments during training or consultancy visits.

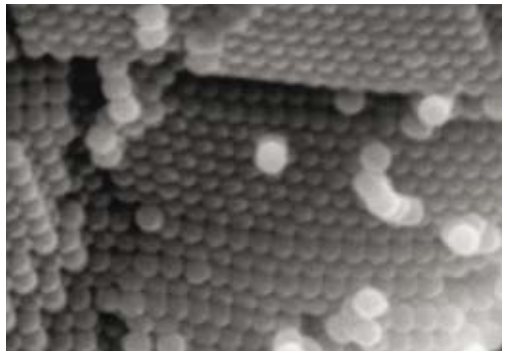

Fig. 11. The first problem image is made up of 0.24 µm Polystyrene Latex spheres. This image has two major errors; firstly the spot size (probe current) is too large making it "spot size limited". Secondly if surface detail is required the accelerating voltage is too high, note the glow from the bottom right of the picture, a typical indicator of the accelerating voltage being too high and note the smooth surfaces on the spheres.

Fig. 12. Here is an image of an engine casing failure demonstrating that the accelerating voltage is too high for most investigations. Note some smooth surfaces and a glow from others. The apparent third dimension indicates that there is a high degree of converted backscatter, not a problem if the data required is to be found sub surface.

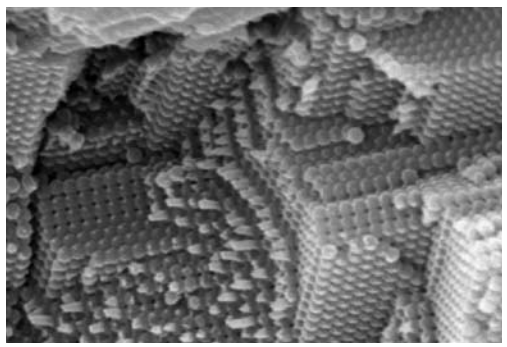

Fig. 13. The image is spot size limited and, if the surface detail is required, the accelerating voltage is too high. Being spot size limited the edges of structures look soft. The working distance is correct which combined with a small final aperture enables the overall focus to provide a very good depth of field. Penetration being too high results in the strands of material in the centre of the picture being detail free. The lack of detail is due to excessive signal with the secondary electron emission from the thin strands. The signal from the beam entered the material is being added to the signal from the lower surface as the beam exited the material. Lowering the accelerating voltage would prevent this total beam penetration and provide more true surface detail.

The following figures are BSE images of a polished aluminium-silicon-iron-manganese-copper alloy.

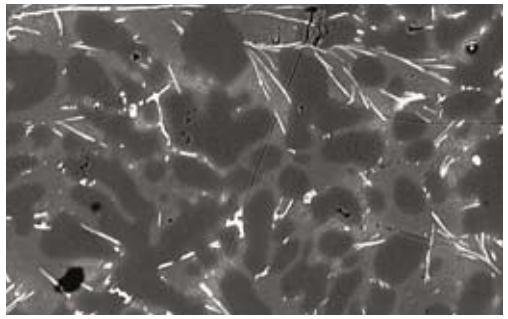

Fig. 14. This figure is produced at an accelerating voltage of 30kV which is too high; too much penetration softens the edges under the surface as the constituents build on top of each other. Total contributing volume for BSE calculated by a Monte Carlo simulation is from 3.6µm beneath the surface.

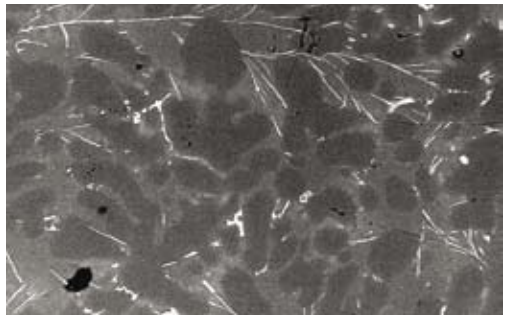

Fig. 15. Reducing the accelerating voltage to 20kV the image is more informative, sharper edges and a hint of the aluminium-silicon eutectic in the background between the dark aluminium phases. Note the dirt on the surface of the specimen is only just starting to be displayed emphasising the sub surface content is still dominating. Total contributing volume for BSE calculated by a Monte Carlo simulation is from 1.8µm beneath the surface.

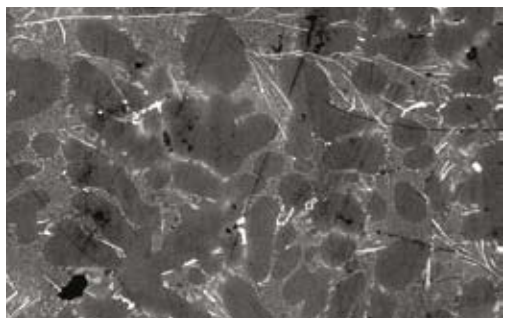

Fig. 16. With the accelerating voltage at 10kV there is an ideal balance of accelerating voltage and spot size, sharper edges and a strong presentation of the eutectic in the background. Too high an accelerating voltage in the earlier pictures involved too much eutectic within the reaction volume with structure overlap spoiling the detailed information. Note an even greater contribution from the surface including scratches. Total contributing volume for BSE calculated by a Monte Carlo simulation is from 0.5µm beneath the surface.

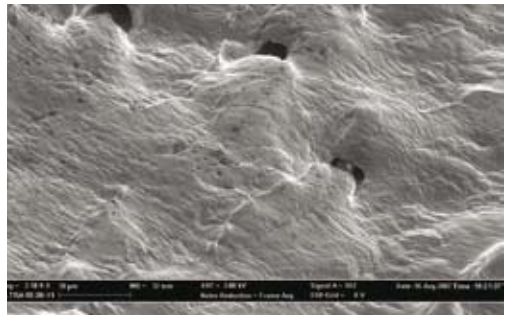

Fig. 17. Fatigue striations in a steel cylinder fractured under conditions of extremely high press. and vibration. The accelerating voltage has been optimised at 3kV to best display the structure, too high an accelerating voltage and the fatigue striations are overwhelmed by the sub structure.

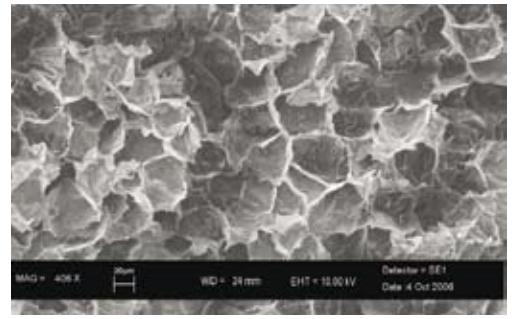

Fig. 18. A Peanut was fractured and had its fat dissolved overnight through immersion in acetone. The specimen was lightly sputter coated with gold but accelerating voltage, spot size and emission current were needed to be balanced to try to enable charge free images. The higher accelerating voltage adds converted BSE which give depth and character to the image. It is difficult to overcome the penetration affect on the thin edges of material and obtain the apparent third dimension. There is still evidence of charge in some areas.

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Steve Chapman formed Protrain in 1982 offering consultancy in electron microscopy worldwide.

Steve first became involved with electron microscopes in 1964 at the Department of Physical Metallurgy, University of Birmingham, England. Moving on to working as an electron microscope service engineer in the UK his service activities as a senior engineer eventually took him throughout Europe. He then moved into instrument sales and marketing where he worked with a number of electron microscope and accessory manufacturers in the demonstration and application fields. Steve has been involved with the design of cryo systems and sputter coaters for scanning electron microscopy, as well as the design of transmission and scanning electron microscopes themselves.

Steve runs his "in house" courses and consultancy in individual laboratories in many parts of the world, north and south of the equator. He also is the senior lecturer in a number of residential short courses in universities across Australia and South Africa. Steve also runs courses to train electron microscope service technicians across the range of manufacturers.

An advocate of the electronic book he has developed a range of interactive training CD with great success world wide.

Steve has written six books including those on the operation and maintenance of scanning and transmission electron microscopes as well as contributing to other author's works. His publications include papers on a range of electron microscopy subjects from instrument design through a very wide range of instrument applications and more recently on Quality in Electron Microscopy.

Steve Chapman teaching in a TEM Short Course at the University of Queensland, Brisbane, Australia.

Steve is also very active in his other love, that of motorsport. A designer of kart race circuits around the world he is also a Clerk of Course, the manager and referee at a motor race meeting, acting up to International level. He has written several books on kart racing and has an internationally successful karting interactive CD.

NOTE

The images in this article have been gathered over 27 years of teaching. Many were produced before data was printed on a micrograph and as they were obtained during courses to display SEM characteristics there is often little or no data on magnification or accelerating voltage.

References

I would suggest any paper by David C. Joy.

To obtain a copy of the Monte Carlo simulation used here go to: www.emcourses.com/montecarlo.htm