SEM Diaries - 42
MMC2025 and Low Vacuum Mode

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Fig. 1: Living the life - feet up on SEM desk in my TESCAN socks with a cup of tea in my TESCAN mug to hand.

Every two years in early July, microscopists from all over the world descend on Manchester Central conference centre for the Microscience Microscopy Congress (MMC). The year 2025 was one of those years. There are several parts to MMC. The main event for some is a conference (for which one has to pay), then there is the Trade Exhibition (which is free to attend), there is a poster session and there is also a Scientific Imaging Competition. Along with several other members of PMS I have been sometimes fortunate enough to have images down-selected for the competition, but unlike one of the others I have never been awarded a prize. Last, but not least, there is free coffee and tea available throughout the day and alcoholic refreshments (also free) during the last half hour or so of the event on the first two days.

I have been attending this event (or at least the free Trade Exhibition) for a number of years now, and in fact it has been pivotal in my journey to becoming an electron microscopist. Now that I have purchased an SEM, and equipment such as sputter coaters to go with it, one might think that I no longer have a need to attend. However, quite apart from it being an enjoyable event, it provides an excellent opportunity to catch up with (and sometimes moan at) suppliers. The electron microscopy community is sufficiently small that I am on first name terms with many of those I deal with. This does, of course, include TESCAN (who sold me my SEM). This year was my first opportunity to meet their new Sales Manager for the UK (and a few other countries). In fact, I spent quite a lot of time on their stand, not only having discussions with their service manager and another of the UK team whom I know quite well, but also being introduced to some of the staff from their head office in Brno in the Czech Republic.

Although I have no ambition to upgrade my current MIRA for a more advanced model, I did sit in on a demonstration of their new MIRA XD. This actually identified a couple of features of interest that could help with my current SEM. One was the use of an alternative Low Vacuum “gas” (namely water vapour), which is said to provide better images than the nitrogen gas I currently use. (Unfortunately, on further enquiry I was told that my MIRA could not be retrofitted with a water vapour system.)

The other is a software improvement that allows one to focus using the wheel on a mouse rather than the roller ball of the SEM that is normally used. This feature would be invaluable when controlling the SEM remotely using a laptop connected to the SEM via the Internet. As with almost all SEM manufacturers, TESCAN no longer transport their SEMs to the exhibition, preferring to demonstrate them remotely. It was while watching the demonstrator of the new MIRA XD using this feature to focus the beam that I realised that it had been added to the software used on my SEM.

The event is organised by the Royal Microscopical Society, and they have a large area set up as a “Learning Zone”. This included a small lecture hall, an exhibition of antique microscopes from their collection and various types of microscopes (including a desktop SEM) which can be demonstrated to visitors. In most cases the visitors were able to “play” with them. I spent quite a bit of time chatting with the demonstrator of the SEM, and managed to sell him a copy of my book in the process! I also manged to catch up with several PMS/QMC members, including a father/son duo who had travelled up for the day, by train, from South Wales!

Having visited MMC on numerous occasions now, I failed to take any photographs of the event this year. My main photo this time (Figure 1) shows off some TESCAN “merch” from the event; well not so much merchandise, more gifts for a valued customer. Either way the socks, at least, incorporate their new and catchy marketing phrase!

So much for my trip to Manchester, but what have I actually been imaging with my SEM?

Well, along with two others I have been studying the anatomy of a particular type of moth. This is a follow up project to one we carried out during Covid lockdowns to study the green shield bug. I shall not say much about the particular moth or the findings in this issue of the diary, but trying to image it on the SEM threw up some major issues to do with charging, and this in its turn led me to investigate the use of Low Vacuum mode.

The problem with moths (from the electron microscopists' point of view) is that they are covered in scales. This is not restricted to the wings, but the whole of their body seems to be affected. Now, butterfly and moth scales can make very attractive electron micrographs, but they are very prone to charging by the electron beam. Regular readers of this column will know that if a specimen is non-conducting (of electricity) it is normally required to be sputter coated with a very fine layer of gold or a gold alloy before imaging in the SEM. This is to conduct away any excess charge from the electron beam. If electrons strike the specimen but cannot be conducted away, a negative charge builds up on the specimen and this affects the image in a number of ways.

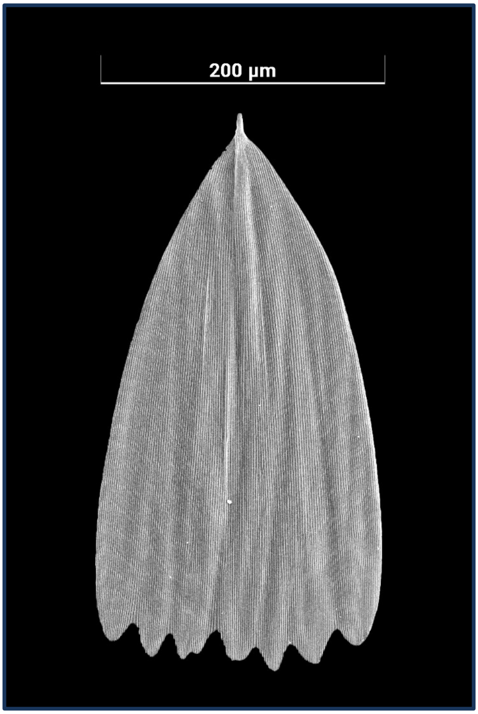

Fig. 2: Single wing scale imaged in the normal way, with the secondary electron detector.

Figure 2 shows an individual scale from the moth. The scale was simply brushed onto a conducting sticky tab on a stub, sputter coated and imaged in the normal way. The edges of the scale are in contact with the tab over most of the circumference and this, together with the sputter coating, provides a good conducting path for the incident electrons. Figure 3 (next page) is an image of a small part of this scale at much higher magnification, and shows much more detail of the structure of the scale.

However, when scales are imaged while still attached to a wing then problems can arise. There is no reliable path for charge to escape even if the whole wing is sputter coated. This is because individual scales lie on top of one another rather like roof tiles, and where a scale rests on top of another scale, the lower scale surface does not receive any coating on the part of the scale that is hidden beneath the upper scale. There may be some charge flow between adjacent scales but this cannot be guaranteed. Figure 4 shows a typical result if the scales are imaged in the conventional manner, using the secondary electron detector (SED) and sputter coated in gold/palladium alloy. There is a recognisable image, but it has a very streaky appearance. In even more serious cases of charging areas of the image may even "white out" caused by saturation of the amplifier in the SED by a large excess of electrons leaving the surface and being attracted to the detector.

There are a variety of techniques available to help minimise the charging or at least its effect on the image. Among

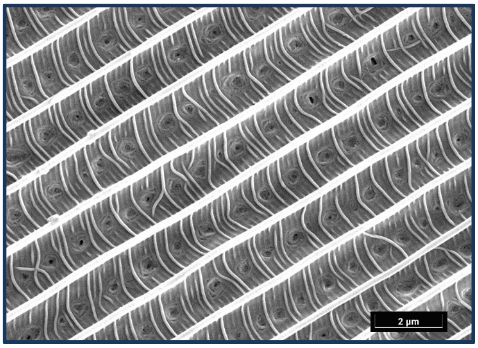

Fig: 3: Detailed view of part of the wing scale of Fig. 2 at a magnification setting of X20k.

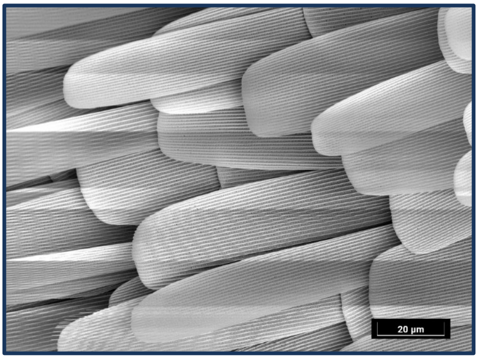

Fig. 4: Effect of moderate charging on a secondary electron detector image of overlapping wing scales.

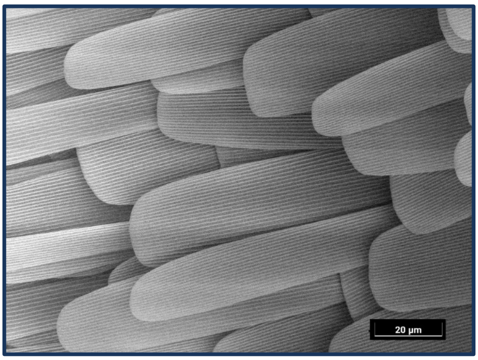

Fig. 5: The same specimen as in Fig. 4, but imaged using Low Vacuum Mode.

these are: to use the backscattered electron detector as opposed to the SED; to reduce the accelerating voltage to a low level; to increase the scan speed so that the electron beam does not spend so long at any particular location; or to provide a supply of positively charged ions to neutralise the charge on the specimen. This last technique, called low vacuum mode, was used to image the same sample as Figure 4, to produce the result shown in Figure 5. This demonstrates a significant improvement over the image created using the SED, although traces of charging can just be discerned.

The principle behind low vacuum (also known as variable pressure) mode is illustrated in Figure 6. A gas is introduced into the chamber (in my case nitrogen gas) and this reduces the vacuum in the chamber from around 10^{-3} Pa to, say, 30 Pa. (Atmospheric pressure is 10^5 Pa.) The vacuum is sufficiently high as to not break up the electron beam, so this will provide secondary electrons from the specimen in the same way is it would in conventional operation. Instead of the secondary electrons being attracted to the SED, they are instead attracted to an anode surrounding or adjacent to the pole piece. On its journey from the specimen to the

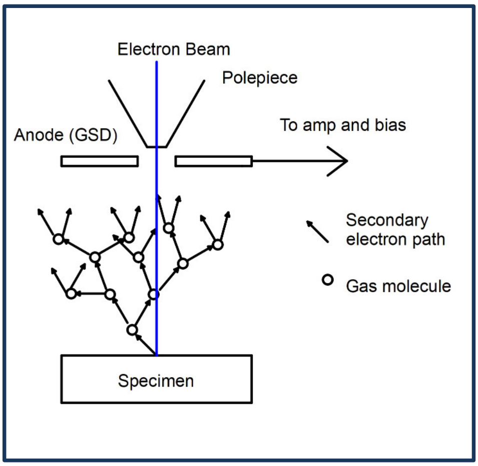

Fig. 6: Diagrammatic representation of the “chain reaction” created by electrons in a gaseous environment.

anode (which is held at a positive voltage) the electron may collide with gas molecules, and in the process the molecule is likely to be ionised, emitting one or more electrons and acquiring a positive charge. The emitted electrons will then progress and strike other gas molecules and something resembling a "chain reaction" will ensue so that for each secondary electron leaving the specimen a large number of electrons will be attracted to the anode, which also acts as a detector known (on TESCAN SEMs) as the Gaseous Secondary Detector, or GSD.

The clever bit, as far as my use of the low vacuum mode is concerned, is that the positively charged gas molecules are attracted to the excess negative charge on the specimen and reduce or eliminate this charging effect.

A number of parameters may be changed to fine-tune the system performance, including beam energy, scan time and chamber pressure. I mentioned that I used 30 Pa for my imaging, although my SEM can operate with chamber pressures upto 700 Pa.

If you have (roughly) followed me so far, you may have one or two questions in mind. In anticipation, here are some answers:

Q1: Given how LV mode cancels out charging, why not use it all the time, to avoid the need to sputter coat samples?

A1: The short answer is that the quality is not as good, either in terms of resolution or background noise, as it is with imaging coated samples with the SED. There may, however, be times when it is not permitted to sputter coat samples, particularly in the case of museum artefacts and rare materials such as the Winchcombe meteorite or similar. Also, when carrying out microanalysis (EDS) at high accuracy, the presence of coating materials such as gold or carbon would be included in the result.

Q2: How is the low vacuum of the chamber isolated from the necessary extremely high vacuum of 10^{-8} Pa around the electron gun?

A2: The SEM requires a clear (but narrow) path from the electron gun to the specimen. The vacuum differential is maintained by the various vacuum pumps having the capacity to maintain the required vacuum in each of the three main regions, despite ingress of some gas from the chamber. Apertures between the various regions (electron gun, column and chamber) restrict the flow of gas molecules. For especially low vacuums (values above 40 Pa) my SEM requires an additional aperture to be fitted, which is supplied with the SEM and screwed in when needed.

Q3: Why can't you use the conventional secondary electron detector rather than the GSD.

A3: The Everhart-Thornley SED works by attracting the secondary electrons towards a Faraday cage with a positive voltage of around 250 Volts (similar to the GSD). The electrons pass through the cage and encounter a field of around 10kV, which accelerates the electrons towards a scintillator on the end of a photo-multiplier. This 10kV field is sufficient to cause the gas to ionise and even spark between the 10kV source and earth. Obviously this is highly undesirable!