SEM Diaries - 22

More on rotatable stubs, and a new SEM!

Jeremy Poole

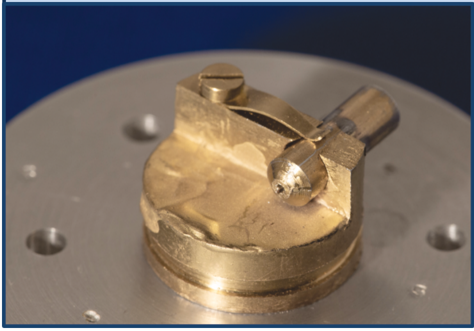


Fig. 1: The Mark II rotating stub holder fitted with a Mk I insert

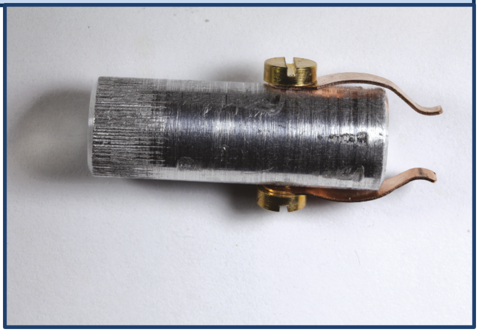


Fig. 3: Tool for handling the insert

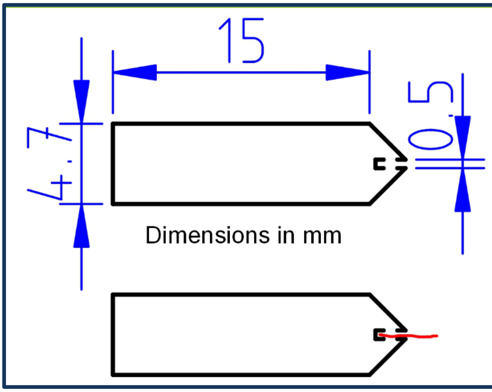


Fig. 2: Dimensioned drawing of the Mk I insert with specimen (in red)

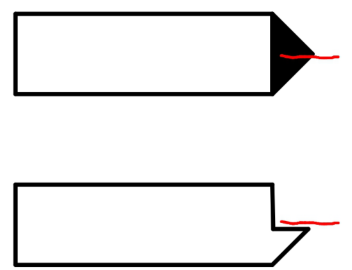


Fig. 4: Drawing of the Mk 2 insert, with specimen (in red)

Regular followers of this column will remember my interest in the legs of spiders and insects, and the need to align them under the electron beam at a precise orientation to show the desired features, which are often obscured by hairs from most viewing angles. To assist in this alignment I designed and built a special holder that permits the precise orientation of the leg to be set, prior to its being mounted on the stage (Figure 1). The leg is mounted in the end of a small aluminium rod (the “insert”), which in turn is held in place in a holder by means of a spring.

In use, the insert is rotated by hand while viewing the leg under a stereo microscope until the desired orientation is achieved. The holder, complete with insert, is then transferred to the chamber of the SEM.

As will be seen from the drawing (Figure 2) the leg was originally fixed to the insert by dropping one end of it into a 0.5 mm diameter hole in its end, also under a stereo microscope. Anyone who has tried to thread a needle will understand the challenge that inserting the leg into the hole in the insert can present, even if the needle itself is held steady in something such as a vice. Just imagine that the thread is very brittle and one wrong move could cause it to break and be wasted and you can understand the difficulty of achieving this successfully with a dried spider leg!

Even assuming that the leg is successfully inserted and glued in position it remains brittle, and anything that touches the leg itself is likely to snap it off the insert. You will understand that wherever possible I will fit several inserts with similar specimens in the hope that one will survive.

Exasperated at the destruction of yet another spider leg, I recently designed an insertion tool (Figure 3). The idea is that this tool is placed over the top of the insert (leg up) which slides into the hollowed out tool and is held in place by the two springs. The insert is then slid into the hole in the flange of the base, bottom first, until it projects sufficiently from the other side of the flange to be able to be grabbed by finger and thumb. The holder can then be pulled away keeping it aligned with the axis of the insert, and the precise orientation in angle and forward-backward movement can be set as mentioned earlier. Once the specimen has been imaged and the stub removed from the SEM the tool is once more placed over the leg to protect it during removal from the base and insertion into a storage box (as described in SEM Diaries - 15). This tool reduces the attrition rate, but only by about 50%.

This still left the question of how to increase the chances of threading the leg into the hole in the insert in the first place. Then I remembered that there is a commercial equivalent to my rotating holder (which costs around £200 each, and explains why I have not bought half a dozen of these). This is not designed specifically for mounting legs or similar spindly objects, but can also be used for items of different shapes that nonetheless require accurate alignment. On that version the tip of the rotating insert, rather than being drilled, is actually milled away to half its diameter, creating a sort of platform on which the specimen can be glued. I decided to modify some of my inserts to copy this, and the result is shown in Figure 4. This proved very successful, in fact it was so successful that I decided to mass produce as many inserts as I could out of the remaining 3/16" (4. 7 mm) aluminium rod that I had in my workshop.

Mounting legs being made so much easier I soon ran out of the newly made blank inserts so turned my mind to ordering some more 3/16" aluminium. This was easier said than done however, as virtually every supplier has deleted that size from their inventories, replacing it with 5mm. I eventually found one vendor offering 3/16" but when I asked for a price even they quoted for 5mm. So, what's in 0.3 mm you may ask? Does it matter? Sadly, yes. It would be difficult to bore out the hole in the flange of the holders to 5mm without making insertion of the inserts even more fiddly. Perhaps I shall have to cut my losses and make some new bases, but they are quite difficult to make, sadly.

Out with the Old and in with the New

OK. I have teased you for long enough. As of the 24th July I have had a new SEM, and I mean brand new. In fact it is the first of this particular model in the country.

I mentioned last time that I had been impressed by the Thermo Fisher Prisma when I had a demonstration of it at MMC2019. I also mentioned that an enquiry I had placed with Thermo Fisher went unanswered. I had also contacted Zeiss and the Czech manufacturer TESCAN about their tungsten source SEMs, and they were keen to do business. TESCAN have the reputation of being very competitive, while Zeiss have the reputation of making very sound instruments and being the successor of Cambridge Scientific Instruments (who produced the first commercial SEM). With Zeiss, though, you could be paying for the name.

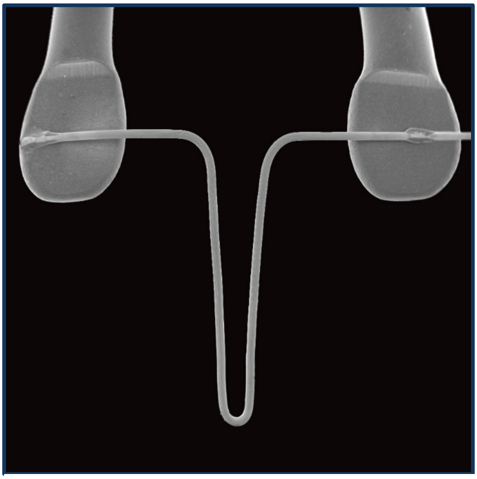
The process of viewing and negotiating on two very different SEMs was almost as much fun as owning my new SEM, but I shall spare you those details. Suffice it to say that I am now the proud owner of a TESCAN MIRA 4 SEM. This does not use a tungsten filament source, but a field emission electron gun (FEG). In automobile terms this is the equivalent of a Golf GTi as compared to a Renault Clio, or in light microscopy terms it represents an increase in numerical aperture of up to three times with my particular configuration (with a best resolution of 1 nm, compared to 3 nm for a tungsten gun). There are, of course, "Rolls Royce" SEMs as well, but a MIRA is (or rather was) far enough into the world of fantasy for me!

So, what is the difference between a tungsten source and a field emission one? Quite a lot, actually. In order for electrons to leave a metal they need to overcome something called the work function of that metal. For this to happen with tungsten it is necessary to heat the metal up to white heat and draw the electrons away using an electric field. The emission current of the electrons can reach between 100 and 200 µA, but sadly most of these electrons are lost by attraction to nearby objects at a positive potential.

The Schottky field emission source used on my new SEM also has a bent loop of wire, but attached to this is a single crystal of tungsten, pulled to a very fine point (typically 300 nm in radius) and coated with zirconium oxide (Figure 5). The source is heated by a current through the loop, but to a much lower temperature than a tungsten one. The work function that needs to be overcome is significantly lower than that for a tungsten filament, partly on account of its fine point and partly because of the zirconium oxide coating, which is replenished from a reservoir. An electric field helps draw the electrons out of the source metal (hence "field emission"), which is aided by the extremely fine point. The emission current is much the same as that of a tungsten emitter, but there are many fewer wasted electrons. Overall the "brightness" of this type of source is around 1,000 times that of a tungsten source.

So, given the superior performance of the FE gun compared to tungsten why bother with tungsten? Well, there are a number of trade-offs between the two technologies, some of which are summarised in Table 1 on the following page.

The biggest difference is cost, not only of a replacement source, but also of the additional capital cost of achieving the much higher vacuum around the gun, the provision of an uninterruptible power supply unit, and the cost of the electricity to keep the SEM powered up at all times. The biggest attractions are the improved resolution and the higher brightness. Whereas it would take me about 5 minutes to achieve a photo quality image of 2048 pixels width using my old tungsten system, a similar image can be



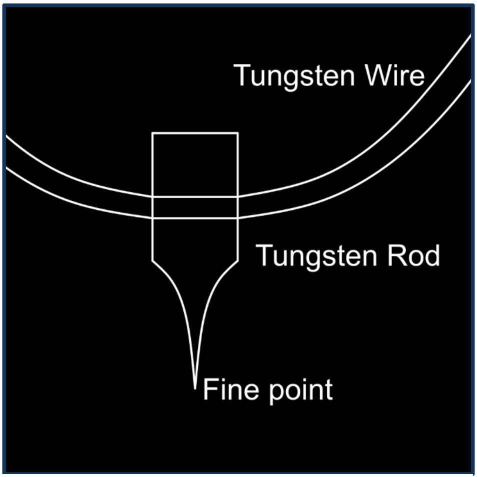


Fig.5: Left: Image of a conventional tungsten electron source.
Right: Representation of a Field Emission electron source.

Parameter	Tungsten	Field Emission	Units
Lifetime	~100 hours	3 years +	
Replacement cost	25	5,000	£ VAT extra!
User replaceable	Yes	No	
Required vacuum	$<10^{-5}$	$<10^{-8}$	Pa
Resilience	Stable. May be switched off.	Must be run continuously. A period of baking-in is needed following any accidental switching off.	
Brightness	10^4	10^7	Amps/ (cm^2.sr)
Minimum probe size (resolution)	3	1	nm

Table 1: Comparison of Tungsten and Schottky Field Emission Electron Sources

obtained in around 1 minute with my new SEM.

I mentioned cost a few times in the comparison of a tungsten and a FEG-based SEM. When I was considering buying a new instrument it never entered my head to purchase a FEG one. In fact, when I contacted TESCAN I was talking of one of their VEGA SEMs with a tungsten gun. I must admit our conversation did touch on one or two features that I had seen at MMC 2019 that were only available on a FEG. It was literally 30 minutes before I was due to have a telephone conference with Zeiss that TESCAN emailed me to offer the MIRA system at a price I could not afford … to decline, contingent only on my agreeing to support them in some non-onerous mutual collaboration ventures.

I did, however, follow through with Zeiss and look at their EVO, especially as my interest will always be aimed towards the lower resolution end of the spectrum, well within the capability of a tungsten instrument.

So, what are my first impressions (as I write this in the middle of August). Initially, I have to admit, it was a struggle to get used to the new user interface, despite a day of training at the time of commissioning. In fact, briefly I was asking myself why I had bought the instrument in the first place! However, I am now extremely

Fig. 6 (top): Don and Ken loading the old SEM onto their transport. Fig. 7 (below): The Mira is wheeled off its pallet.

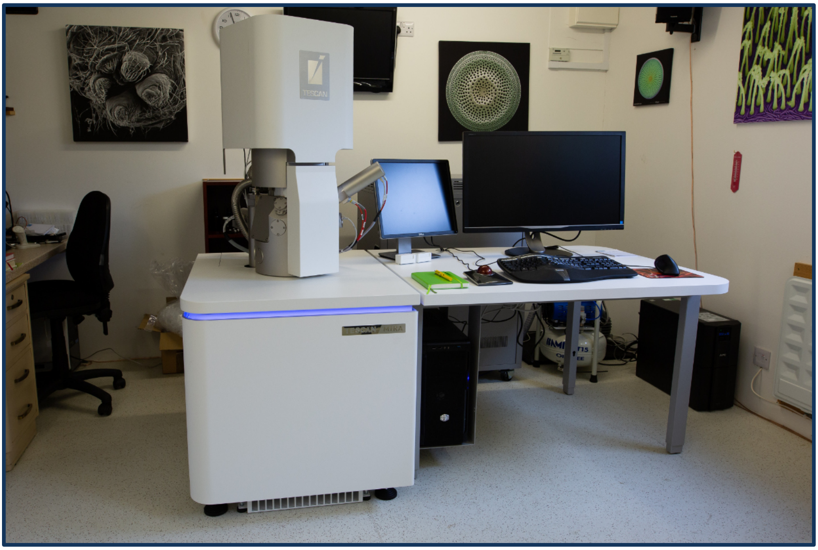


Fig. 8: The MIRA 4 installed in my Laboratory

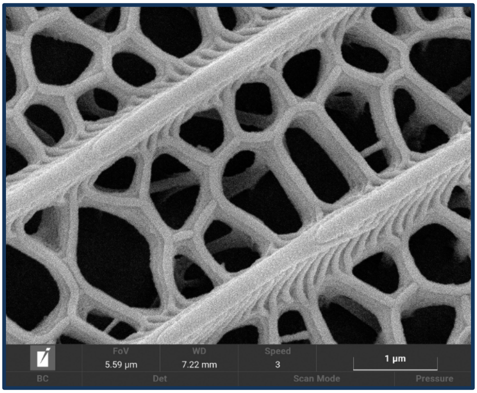


Fig. 9: Butterfly wing scale (detail)
FoV = 5.6µm

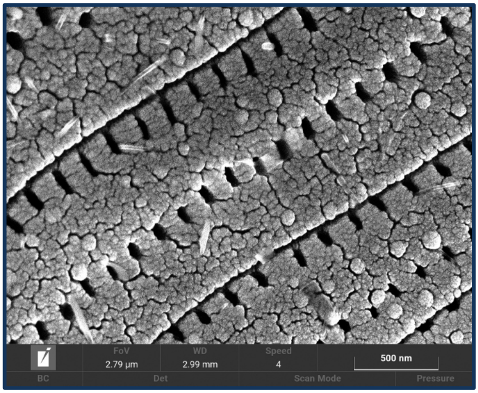


Fig. 10: Detail of a girdle band of the diatom *Diatoma mesodon* FoV = 2.8µm

positive about it all, even more so since having a further full day of training. Early results are shown in Figures 9 and 10. These were taken at the high magnification end of the performance spectrum, at a selected magnification of x50k and x100k respectively, and using the in-lens secondary electron detector for maximum resolution. Incidentally, the crazing on the surface of the diatom in Figure 10 is not the structure of the diatom, but the granularity of the gold/palladium coating, which the new capability of my FEG SEM now reveals.

It was sad saying goodbye to my old SEM. I shall also be seeing less of Don, since the MIRA 4 will be serviced and maintained by TESCAN rather than Tron-Tech. However, I am sure Don and I shall stay in touch, and he is actively working on finding a buyer for my Inspect. I shall certainly keep him on my Christmas card list!

Further details of my new SEM are at: www.jeremypoolesem.org.uk/mira4.html