

SEM Diaries - 1

Gestation and Birth

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Series Introduction

At a recent Penkrige meeting, I let slip to some senior members of the Society that I am embarking on a project to procure a Scanning Electron Microscope (SEM) and use it to advance my interests in arachnology and photomicrography. After I had outlined the results of my researches into SEMs thus far they suggested that I write a series of articles in BP on my progress in procuring, installing and using an SEM. To be honest, I was initially somewhat unwilling to do this, on the basis that few readers of BP would be fortunate enough to be in the position of undertaking such a project themselves. However, I have now relented and hope that this series will

provide some interest, and also, eventually, some pictures to adorn the pages of BP.

First Encounter

As with all my major (and expensive!) projects, my decision to buy an SEM was not made on impulse, but has taken years of thought to come to fruition. In fact, I can trace my interest in SEMs back to October 1968, when as part of my induction as a new undergraduate in the Engineering Department of Cambridge University I was shown a research SEM by Professor Charles Oatley (“the father of the SEM”[1]). Oatley and his research students had been working on SEMs since

1948, and had finally persuaded Cambridge Scientific Instruments to produce the first commercial SEMs in 1965. I cannot now remember what was being displayed as a specimen, but I know it was chosen to demonstrate the “3-D” appearance of SEM images. I think it must have been part of an insect head. Whatever it was, this encounter left a lasting impression, although at the time I never envisaged owning one.

Periodic Refreshing of Interest

It was probably some years before I next set eyes on an SEM, or even on images produced using one. but since joining PMS and QMC I have fortunate enough to view instruments, for example at RMS microscopy congresses and at visits to the Electron Microscopy facility of the Natural History Museum. I also encountered some excellent images in a book on the anatomy of spiders, and purchased a number of “coffee table” books of SEM images, again mainly of entomological subjects. One notable volume, by David and Madeleine Spears, I purchased as a result of a review by Brian Bracegirdle in the *Quekett Journal* [2].

Specimen Preparation

It may seem strange to suddenly divert readers into the fine detail of specimen preparation, but this is crucial to the development of the history, if you are still with me.

Two major requirements of a specimen for observation in a conventional SEM are that it should be a) dry, and b) conducting. A moist specimen would evaporate water molecules in the vacuum of the column of the SEM which would interfere with the high vacuum and hence the electron beam, and a non-conducting specimen would build up charge on its surface which, again, would interfere with the electron beam and produce artefacts on the image.

I used to think, if I were going to own an SEM I would need to employ a technician

to prepare my specimens for me. (Where else would I obtain the expertise, but where would I find the money?)

Because it is the 3-D effect (actually a shadow effect) that makes an SEM image so striking (and useful) it is crucial that the specimen is not distorted during the drying process. Unfortunately, drying agents such as alcohol or acetone (or indeed any liquid) create surface tension on the surface of the specimen as they evaporate and this causes significant deformation of the specimen, rendering it next to useless. One way round this is to commence the evaporation at slightly above a combination of temperature and pressure, known as the “Critical Point” of a liquid, where the physical characteristics of liquid and gaseous phases are indistinguishable. This requires the use of a “Critical Point Dryer (CPD)”. The full process involves the dehydration of the specimen using ethanol or acetone, the replacement of the dehydrating agent by liquid CO₂, and then the conversion of the CO₂ to the gaseous phase at slightly above the critical point temperature. The critical point of CO₂ is at a temperature of 31 Celsius and a pressure of 73.8 bar (or 1,070 psi), so the system is maintained at a temperature above 31 as the pressure is reduced from, say, 1,200 psi to atmospheric pressure.

As can be imagined, with pressures as high as 1,200 psi, this process needs to be carried out using proprietary equipment and not using some home-made Heath-Robinson device.

In order to render the dried specimen conducting, it is necessary to coat it with a thin layer of a conducting material, such as gold or palladium. This requires the use of a “sputter-coater”. Again, this is not something one could cobble together at home.

The Last Two Years

A number of factors have led to the project taking off over the last two years. The first

of these was having the chance to discuss SEMs with a real user, David Spears, whom I encountered at a Dale Fort weekend, and to view more of his work. This, in turn, led to my visiting the Exhibition attached to the RMS Microscopy Congress in Manchester last June. I have previously written about that visit [3] so shall not repeat the details of those two days. Suffice it to say, that I came away from that event with the intention of proceeding, in slow time, with the purchase of either a new desktop SEM such as the Phenom Pure, or else a pre-owned instrument, preferably Variable Pressure, should a suitable one become available.

I initiated a dialogue with the importers of the Phenom, and was developing an interest, despite reservations about the minimum magnification, when I decided to also look into obtaining a pre-owned CPD and sputter chamber. A phone call to the main suppliers of new equipment (Quorum) led me to a company called “Tron Tech” and a few days later I received a phone call from Donald Ashcroft, one of their directors. It transpired that, in addition to their often sourcing pre-owned preparation equipment, they deal in pre-owned SEMs. Would I like to visit their premises to see what was on offer?

Never one to turn down an offer to view an SEM I made visits to two different premises to view a variety of instruments ranging from a high end Hitachi (way outside my budget and not that suitable) down to 30 year old analogue Cambridge Stereoscans. I guess I knew, within a few minutes of my first visit, which instrument I was going to end up buying. Two weeks later, and in the nick of time, I wrote a substantial cheque as a holding deposit for an FEI Inspect S50 (Frontispiece).

Now, where on earth can I put this half-ton of high technology? All will be revealed next time.

References

1. Everhart, T. E., Persistence Pays off: Sir Charles Oatley and the scanning electron microscope. *Journal of Vacuum Science Technology B* 14(6) 1996. (Available from the web)
2. David and Madeleine Spears, with Adrian Warren. *Unseen Companions. Big Views of Tiny Creatures.* Last Refuge, 2007
3. Jeremy Poole. *MMC Exhibition at Manchester.* *Balsam Post* 105, October 2014, Pages 34-37