SEM Diaries - 33

Struggles, solutions - and elation

Jeremy Poole

Fig.1: Polished section of forest marble.

I am making fewer images this year than at the same time last year. In the first four months of 2022 I made 670, whilst this year the total has been only 382. The reasons for this are complex, but, for example there are only so many images one can make of a particular species of foram before the subject can be classed as "done". So, short of seeking out samples from new locations imaging forams can become repetitive.

Similarly, there is little point in making fresh images of the various identification features of spiders, if there are perfectly adequate images for a particular species already on my spider website. I need to collect in new areas and to this end, by the time you read this I shall have spent a few days with other members of the British Arachnological Society collecting in Northumbria.

Pollen, another favourite subject of mine, creates similar issues. The same wild flowers spring up each year on my regular walking routes, so once I have a few satisfactory images of the pollen of these species, in dehydrated and hydrated forms, imaging further examples can become tedious and routine, not to mention unnecessary. Perhaps Northumbria will provide fresh species of wild flower as well!

So, thank goodness for my recent interest in mineralogy. For various reasons this is taking a bit of time to "take off" but it does provide new challenges, and the novelty of the subject gives me an incentive to

buckle down to learn different preparation and imaging techniques and create pictures. Of course, the precise number of images made in a year should not be assumed to correlate with the amount of satisfaction received! In mineralogy, for example, not only do I like to image the subject matter, whether it is embedded and polished flat or still in its collected state, but I also use my EDS system to perform a chemical analysis of the material. While I tend to use an exposure of just over a minute for imaging, analysis of a material can take a few minutes for each of three or four individual "spots" on the subject matter, and around an hour for a compositional map of a wide area. Output, in terms of throughput, is significantly reduced.

I have mentioned now and then, both in this column and also in the Balsam Post Editorials, how beneficial it is to go to Society meetings, such as the one day ones, held now and then, or even better the residential weekends, such as are held at Flatford or the Cranedale Centre. I have had a number of useful chats at these with one member of QMC who is also into rocks and minerals, and been given samples. Another member, first met at Dale Fort, and now also a good friend is full of words of wisdom when it comes to dealing with materials such as minerals, gleaned from his years working in ceramics.

One such conversation helped me solve a literal square peg in a round hole dilemma. The problem is that the vice on my precision diamond saw has a V-slot in it and is designed for cutting round specimens, such as metal rod. The shape of rocks could be anything, but is very unlikely to be cylindrical. I have tried holding flat pieces of rock in the jaws but since the jaws are ground flat, rather than serrated, the specimen is very likely to move. In fact I recently broke my £250 diamond saw blade when the specimen did just that. Mike's solution is to bore into the rock with a diamond coated cutter designed for cutting holes in tiles and similar hard materials. This works very well, and it is possible to buy cutters that have an internal diameter just a little bit smaller than that of the mould into which the specimen will be placed for embedding. The problem of clamping the specimen while drilling into it still remains, but I have tried several techniques that seem to work. One is simply to put the sample on a piece of plywood on the bed of a milling machine or drill stand and push, say, three clamps up against the sides of the specimen to stop it rotating when it is "cored". An alternative way is to hold the specimen in a milling vice or the drilling equivalent, although this has some of the same issues as holding a rectangular specimen in the chuck on the saw (Figure 2).

The technique is simply to set the mill or drill to a slow speed and gently bring the bit down onto the specimen, while lubricating it with water (or rust inhibiting coolant) from a spray bottle. The bit should be raised periodically and the specimen sprayed around the circular groove that is being cut (Figures 3 and 4). Be gentle, with slow revs, light pressure, and plenty of lubrication. There is one minor issue with this technique, in that

Fig. 2: Diamond hole saw, together with forest marble rock, held in the jaws of a milling vice. Note the shell on the top surface of the specimen directly under the cutter.

Fig. 3: Coring in process.

the slurry of coolant and mineral dust could get onto the moving surfaces of the vice or even milling table. I use blue wipes to contain most of this slurry and take care to clean up afterwards.

Once the resulting core has been cut to an appropriate length (Figure 5) it can be placed in a flexible silicone mould and embedded in an acrylic or epoxy compound. Once the compound has set the face of the specimen can be polished as described in SEM Diaries - 31, January

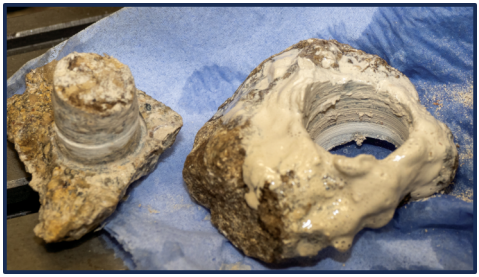

Fig. 4: Core (left) and remains of specimen

Fig. 5: Core held in the vice of the precision saw while being cut. The V-groove can just be seen behind the sample, on the right.

2023. Figure 1 shows a photograph of the polished surface of the specimen in the other illustrations.

Sadly the images from the SEM of the polished face, made using the backscattered electron detector (BSED) are not particularly exciting, compared to the interesting appearance of the polished face as photographed by a conventional camera. Shells tend to be composed of calcium carbonate, and the BSED is designed to create images illustrating differences in atomic number. The specimen seems to be composed almost entirely of shell, so there is little variation in atomic number across the face of the sample. In fact, the only significant contrast appears where flakes have peeled away from the shells during polishing providing a small amount of three-dimensionality (if that is a word!). (Figure 6).

To end on a positive note, at the end of March I travelled to Brno in the Czech Republic, at the invitation of TESCAN to give a lecture on my SEM imaging, and also to view an outdoor exhibition of 20 of my micrographs. The city of Brno is home to three major manufacturers of electron microscopes as well as to a larger number of companies that make ancillary equipment, and each year the mayor promotes a week of “days of electron microscopy” for the city.

Fig. 6: Low magnification BSED image of the polished forest marble specimen, illustrating topographic features rather than atomic number contrast.

TESCAN came up with the title “The art of SEM: from fantasy to reality over half a century” for my lecture, which was delivered at Brno Planetarium to an audience of 80. The talk was to be aimed at a “general audience” and described my “journey” from first seeing an SEM at the age of 19 to becoming the proud owner of a TESCAN MIRA half a century or more later. To justify the word “Art” in the title, I outlined the steps I go through to convert the images from the SEM to “Pictures” that some might like to hang on their walls. The talk went well, but it was amusing to hear the questions from the so-called “general” audience afterwards.

For example:

> *“Do you ever use low-vacuum mode in creating your pictures of spiders?”*
>
> *“Have you taken out a maintenance contract on your SEM?”*
>
> *“Have you considered upgrading to a focused ion beam (FIB) instrument?*

In fact, the only question that could genuinely be assumed to originate from a member of the general public asked if I keep spiders at home. Even that one came from my TESCAN host!

Before viewing the exhibition and giving the lecture I visited the TESCAN facility, where I was given a quick tour of their demonstration rooms and was introduced to several key personnel. Given my

The author posing against a display panel (Photo Barbora Novosadova)

current interest in EDS most of the allotted time was devoted to two activities. The first was a demonstration of the “fully integrated” TESCAN design for EDS. Rather than using a second PC to run proprietary software from a specialist supplier of spectroscopy solutions, TESCAN have developed their own software, which runs on the same PC as the rest of the SEM software. The user interface and results are all displayed on the main 32” display of the SEM. I have now asked TESCAN to quote me for such a system.

The other discussion was about the “TIMA”, or TESCAN Integrated Mineral Analyser. This uses multiple EDS detectors and a multi-specimen sample holder to analyse the composition of minerals in a rapid automated way. I made good use of my time with this team to discuss mineralogy in general, and they kindly provided me with a small but interesting sample to take home with me. I later sent them the results of my analysis of this and they provided me with some very useful guidance.

Outdoor exhibition of electron micrographs in Brno, Czech Republic