

SEM Diaries - 44

Excitements in the Lab

A New EDS System, and Shelves

Jeremy Poole



Fig. 1: The sensor for the new EDS system, projecting from the side of the SEM chamber

I have been boring readers over a number of issues of SEM Diaries now with my long descriptions, graphs and numbers relating to Energy Dispersive Spectroscopy (EDS). My results were all obtained from a loan system that was some years “out of date” in its computing power and algorithms.

As reported in the previous two editions of SEM Diaries, the results with the loan system were not always reliable. This proved particularly problematic for one project (the Goldstone project) where the

ratio of iron to sulphur in a material that was supposed to be iron pyrites (FeS_2) provided an indicated composition of Fe_2S_3 . Analysis carried out by a contact at the Plymouth Electron Microscopy Centre confirmed that the material was, indeed, iron pyrites. This was the trigger for me to order a brand new system from Oxford Instruments.

The new EDS system was installed by TESCAN on the 6th January 2026, and a half-day of training was also provided.



Fig. 2: A screen from the Oxford Instruments EDS software (AztecOne) showing the analysis of the iron pyrites layer of the Goldstone specimen.

During the same visit the engineer also replaced the sensor of my backscattered electron detector (BSED). I had an accident with this a while back, when I drove a specimen into the detector by overshooting the intended vertical position. Although the detector still worked up to a point, one of the four quadrants had failed, and this led to performance degradation.

As of the end of January 2026, when I am typing this piece, I am far from fluent in the use of the new EDS, but I have made sufficient progress to re-examine samples from the Goldstone Project that had previously provided suspicious measurements from the loan system.

An example of this analysis is shown in Figure 2. The top half of the screen displays an electron micrograph of the scanned area, false-coloured to indicate the composition of the material across the specimen. The lower half of the screen shows the spectrum of the X-ray energy emitted by the specimen at a particular location - spectrum 1, represented by a

small square (arrowed). Also in the bottom half of the screen is a box providing the results of the analysis, and this is reproduced as Figure 3.

This tabulates the “Atomic” percentage of the elements that were detected over the small area indicated as spectrum 1 on Figure 3. It shows that the ratio of iron

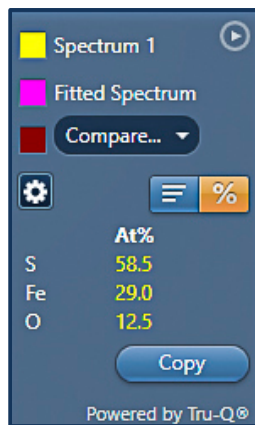


Fig. 3: The quantitative results of the analysis of the Goldstone sample showing the ratio of iron to sulphur of 29:58.5, which is within system error of 1:2, as expected.

atoms to sulphur atoms in the sampling area is a very good approximation to 1:2. This is the result that was determined by my contact at PEMC and confirmed that my new system provides results I could trust.

Regular readers will remember that I do not hire out my services on a commercial basis. In fact all my collaborator are good friends with whom I enjoy doing joint projects, and this leads to us learning from each other, which is reward enough.

As it happens, several projects with collaborators have led to work being published, twice in peer reviewed papers and once in an update to a standard work on the mineralogy of the British Isles. Two of these (the book and the Goldstone project) involved EDS analysis. I just hope that the conclusions in the book do not need to change if the author asks me

to repeat some of the analysis! For the Goldstone paper we were sufficiently sceptical of the results not to report them in any detail.

So, am I happy with the decision I made to buy a new EDS system? The answer is a (slightly qualified) “Yes”. The only real concern I have is that the sensor (Figure 1) is located on the front of the SEM and points into the lab. This could be vulnerable should anyone feel they could use it as a hand hold or knock it with a shoulder. Replacing it would cost “a lot of money”.

Changes to the Lab

Partly as a result of purchasing the new EDS system I decided that I needed to tidy up the lab somewhat, to reduce the number of boxes and sample tubes cluttering up every work surface, and to some extent the floor as well. This



Fig 4: The shelves above the (currently tidy) optical microscopy desk. Note the relocated speaker and monitor. The sloping discs (centre left) are for holding abrasives for the polisher used to prepare rock samples for analysis.

involved fitting four shelves above the desk I use for my light microscopy (Figure 4). I carried out this work over a weekend, without any help. This was a slightly dangerous undertaking. On at least two occasions descending from a stepladder took place in a less than controlled manner and within close proximity to the expensive sensor previously mentioned!

This proved to be a very useful exercise, and I can now make tea and coffee without having to search for a mug behind racks of specimens. I can also work at my optical microscope desk much more easily, having tidied up various cables and extension boards. The only question is, how long will it be before my flat surfaces are once more covered in “stuff”?

Another change to the lab is the installation of a replacement polisher for my rock samples. The original polisher that I bought (see SEM Diaries - 31) turned out to be less than ideal, and had issues with the water lubrication system. I saw a larger and more advanced polisher advertised by the same supplier (also secondhand) and took the plunge

and upgraded. The difference between the two units is that the new one has a larger platen and is more generally robust (Figure 5). It also has a system for dripping pre-specified doses of diamond suspension onto polishing cloths for the final stages of preparation. One disadvantage is that it weighs 70 kg, so when I got it home it remained in the luggage area of my car until I could persuade a friend to help me transport it from the car to the bench in the lab!

Impostor Syndrome

One of the “problems” with owning an SEM with EDS is that mineralogists or curious amateur geologists seem to think that I know what I am looking at! In the case of the pyrite, the colour of the layer in the specimen provided a very good clue as to what the mineral was, and so it was easy to check that the relevant elements were present in the required ratio (with the new EDS, that is)! Usually, the compositions are a lot more complex, and include “difficult” elements, such as carbon - which is also used to coat the sample, so the coating adds a bias to the result.

I was approached recently by a member of our local U3A Geology group with a specimen that a friend had recovered from the River Taw in Devon. It was an interesting shape, and given the somewhat “sharp” edges, was most unlikely to be very old. My first guess was that it might be molten iron from a foundry that had been quenched in water, but its specific gravity was a little over 3, so nothing like as dense as pure iron, whose S. G. is about 7.8.

My next guess, following up the foundry idea, was some sort of slag. Perhaps it was no coincidence that “Finch Foundry” was only a couple of miles up-river from the location where the specimen was found. The only trouble with that theory, is that although it is called a foundry,



Fig. 5: The replacement polisher. The unit on the left is the doser for providing measured volumes of diamond suspension.

Finch Foundry was in fact described as a forge. Perhaps they cast their blades (for agricultural tools) before beating them into shape?

The following figures illustrate the appearance of the specimen, and an analysis of the composition at a couple of locations. I ran this past a geologist friend and his first reaction was that it is likely to be iron slag. He did suggest that I see if it attracts a strong magnet, but when I tried this there was no trace of magnetic attraction. This makes the likelihood of its being iron slag less likely, although does not rule it out. He then commented on Spectrum 3, saying that perhaps the bright areas were most likely to be ilmenite (FeTiO_3). So, at this stage we do not have a definitive answer as to what it is, but there are some ideas, and I can analyse further spots to see if that sheds further light on the subject. One thing all parties are in agreement on is that the specimen was created as a result of human activity.

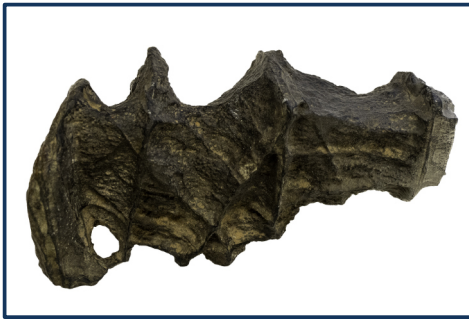


Fig. 6: The specimen as loaned to me for analysis. Overall length approx 7". I cut off a small sample at the right hand end, for embedding and polishing.

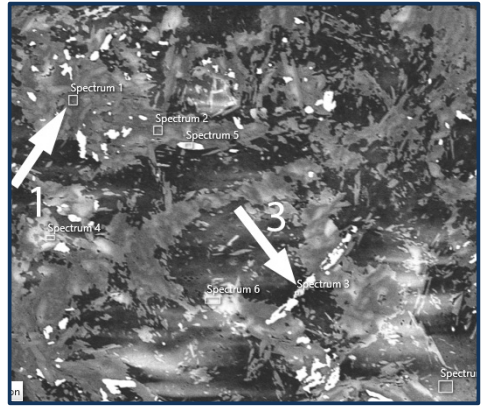


Fig. 7: An electron micrograph of the polished surface showing the areas where analysis was carried out - made using the repaired BSED.



Fig. 8: Showing analysis at two locations. Spectrum 1 is high in silicon and oxygen, suggesting a high silica content. This could be remnants of sand used for casting. Spectrum 3 has a similar amount of oxygen, but no silicon. It also contains metals, namely iron and, interestingly, some titanium. The current best guess for the composition of this area is ilmenite.