SEM Diaries - 31

The EDS has Landed

Jeremy Poole

Fig. 1: SEM Desk with EDAX screen on right and EDAX PC below.
Note also the abundance of keyboards and mice!

That is two extremely corny puns in successive issues of SEM Diaries. Well, it cannot be helped - I quite liked them.

In the last edition of SEM Diaries I described how I had been given the chance to explore the use of Energy Dispersive Spectroscopy (EDS) by taking up the offer of a system on a “try before you buy” sort of basis.In November I had the loan system installed so I was, in theory, all up and running! I say “in theory” simply because I was on a steep learning curve and the installer was unable to provide much in the way of training.

The new system comprises an X-ray sensor with a pulse processor and power supply (Figure 2), a Windows 7 PC and a monitor, together with input devices and various cables. The PC is loaded with the software that does the analysis and provides a user interface (entirely separate from that of the SEM itself.

So much for the kit, but what does it actually do? Well, as explained in SEM Diaries - 30, when electrons from the electron beam of the SEM penetrate a material they displace electrons orbiting around the nucleus of atoms from one “shell” to another “shell”, of higher energy. This electron will then return to its “normal” shell and in the process emit X-rays at a characteristic frequency unique to that element. These X-rays are captured by the detector and analysed by the software of the EDS system to provide a variety of displays.

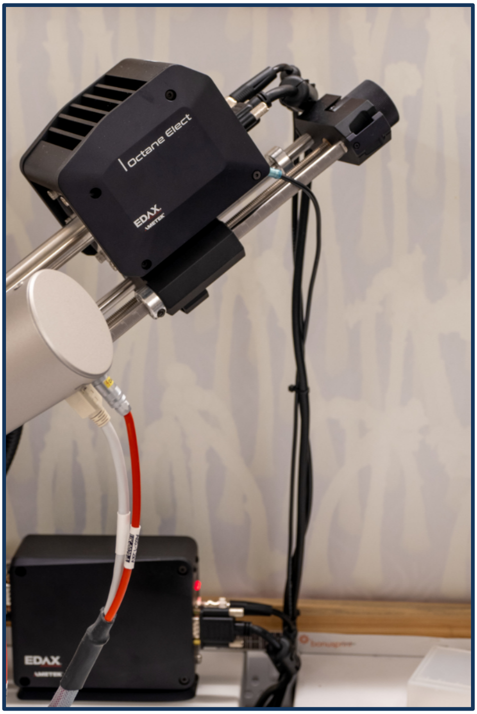

Fig. 2: The X-ray detector, with its pulse processor on the desk immediately below it.

In order to get reasonably accurate quantitative results of chemical composition it is necessary for the sample to be polished flat to better than 1 µm in accuracy. To achieve this, one needs to grind the surface on carbide abrasives before moving on to polishing with nylon material impregnated with diamond paste. When I visited Plymouth University in the summer, one of those present was doing his best to put me off the idea of EDS by stating that he had spent many hours grinding and polishing samples in his youth, and this included grinding down his fingernails. There must be an easier way, though?

Yes, there is! It is to procure a polishing machine. This would not only speed up the process considerably from the manual method, but also save one’s fingernails and fingertips from abrasion. Meanwhile, the microscopists at Plymouth University kindly volunteered to prepare a small number of samples for me to get me going.

The preparation process follows a number of steps, as follows:

1. Cut the sample so that it can fit in an embedding mould. This requires the use of a diamond impregnated blade. The moulds come in various sizes from 50 mm in diameter downwards. I have decided to standardise on 32 mm. Thus I would need a piece of rock no larger than about 25 mm across at its widest part and no more than 15 to 20 mm high.
2. The chosen face of the cut piece of rock is ground to an approximation of flat.
3. The sample is placed in a mould made of flexible silicone material and a resin based embedding agent is poured in to the required depth.
4. The embedded sample is then placed in a polishing machine. (The specimen is at the bottom of the mould, so it is not necessary to cut away large quantities of resin prior to further preparation.) By using first an abrasive disc and then reducing sizes of diamond pastes, down to 0.5 µm, the face is ground to the required flatness with no risk to the fingernails, but with a high impact on the bank balance.
5. Once the face of the embedded specimen has been polished to the required flatness it is placed, face up, in an aluminium holder of the required size and coated with a very fine layer of carbon to render it conducting.

Enquiries established that a new polisher can cost in excess of £10k (before adding on the cost of the consumables and VAT). A carbon coater would cost a similar if not greater amount. I did wonder if I could use my gold sputter coater, but was told that if I did I would “just see gold”.

To cut a long story short, I did manage to find a pre-owned sputter coater from Don, who supplied my first SEM, and a very well used polisher from a company called Spectrographic. One problem with polishers is that many of them seem to require a three phase electricity supply, even though they do not consume a great deal of power. Another issue is that they require a source of compressed air to actuate the plungers that exert a constant force on the specimen to hold it against the abrasive material. Eventually Spectrographic came up with a single phase, well-used, polisher and I negotiated a deal that went along these lines: Spectrographic would supply the coater and some consumables at a reasonable price, provided I would send to them a copy of my book. It transpired that I had had a long conversation with Paul, the manager of Spectrographic, at the Leeds Microscopical Society meeting in Pool in 2021, and had shown him a copy of the book while there. I had not made the connection to the company with whom I was negotiating!

In its simplest form a polisher is a revolving turntable on which is stuck an abrasive disc. The operator is thus relieved of the need to exercise too many hand movements, but is still at risk of grinding away his or her fingertips while pressing the sample onto the abrasive. A water supply drips water onto the disc for lubrication and to carry away the loose particles. One way of avoiding finger damage is to add a separate rotating head that contains a holder for several samples, and can apply a pre-determined pressure to the samples as it rotates about its own axis.

Once the face of the specimen has been abraded to the required degree the abrasive disc is replaced with a polishing cloth in which are embedded diamond particles as described earlier, and the water is replaced by an alcohol based lubricant.

The polisher that I actually bought is illustrated in Figure 3. This shows the turntable, fitted with an abrasive disc, along with the rotating head. Close inspection will show that there is an embedded specimen in the holder on the head.

I also bought a pre-owned saw from Spectrographic, but at the time of writing I have used neither of these tools in earnest.

Having waffled on so much about the apparatus I have left little space to talk about the results!

The output from the EDS presented here was generated using the sample shown in Figure 4. This is one of those from a mine in the North of England, prepared by Plymouth University, and as can be seen even from a cursory examination, it contains various different minerals.

Fig. 3: Struers Polisher, fitted with an abrasive disc. Various programmes are available for grinding and polishing, with or without water lubrication.

Fig. 4: Embedded mineral used for the results presented here.

The small area of this face viewed with the SEM is shown in Figure 5. This image was made using the secondary electron detector, rather than the back-scattered

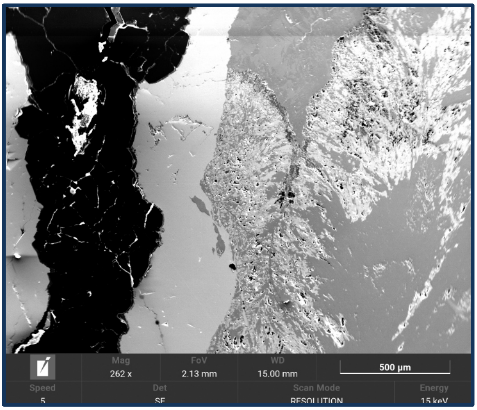

Fig. 5: Electron micrograph of a small part of the specimen of Figure 4.

electron detector used for most of the images in SEM Diaries-30.

Various methods of presentation of the results of a spectrographic measurement are available. Figure 6 shows a map of the scanned area, with the elements recorded at each location identified using a colour code. An alternative measurement method is to concentrate on a small spot within the area. Figure 7 (Page 16) shows the spectral peaks of two elements found at a spot in the black area. The left spot is labelled O (for oxygen) and the right hand one as Si (silicon).

The table of Figure 8 (bottom) records the composition of the various elements found at that location. It does not take a genius to work out that since there is just a bit more than twice as much oxygen as there is silicon, in Atomic %, the predominant compound under the spot is quartz ($Si\,O_2$).

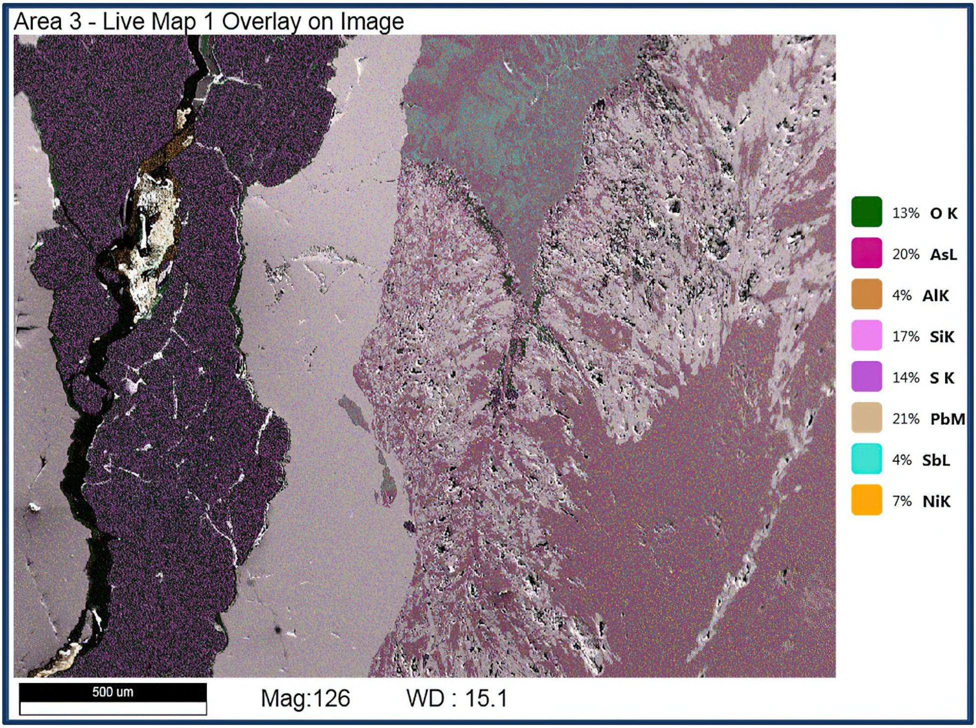

Fig. 6: Composition map of the surface of the scanned area

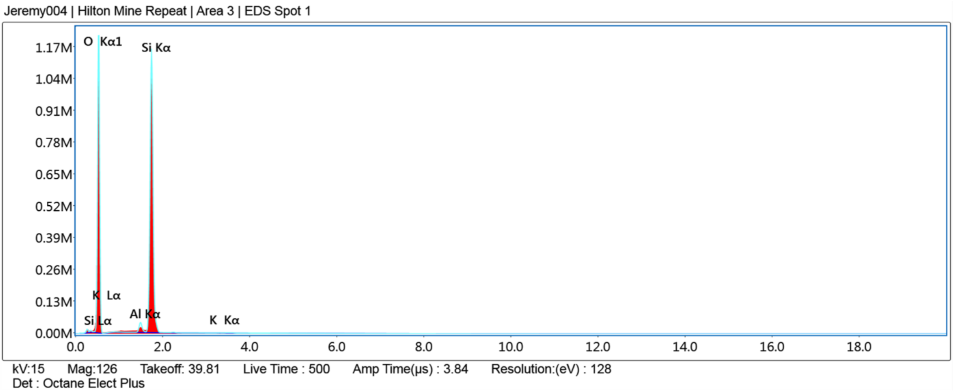

Fig. 7: Spectrum of area containing quartz (silica)

Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	A	F
O K	54.4	67.7	6.2	13976.8	0.2882	1.0463	0.5061	1
Al K	1.5	1.1	3.3	612.9	0.0116	0.9232	0.8244	1.0114
Si K	44	31.2	2.5	18648.3	0.3699	0.9422	0.8909	1.0007
K K	0.1	0	11.4	10.7	0.0004	0.8682	0.9365	1.0097

Fig. 8: Quantitative Results from spectrum of Figure 7