SEM Diaries - 43

How to find out if the SEM is telling the truth

Jeremy Poole

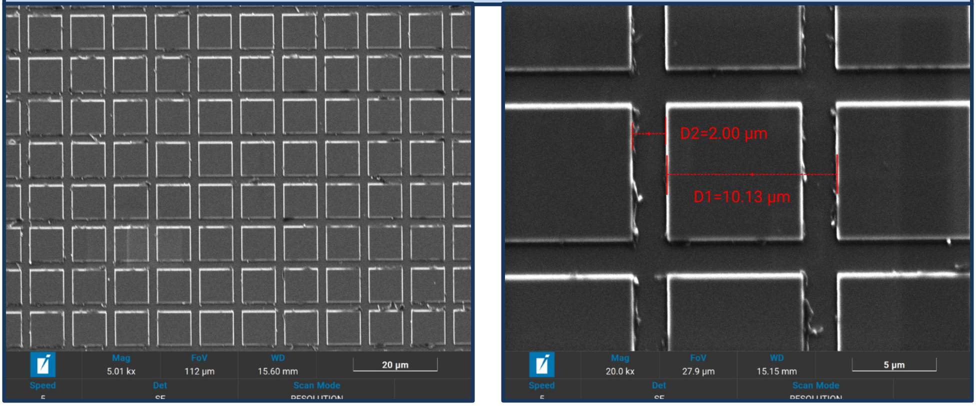

Fig. 1: Planotech Test Specimen at two different magnifications, both imaged using the in-chamber Secondary Electron Detector (SED). A) Left x2.5k, B) Right X10k

Long before I got immersed in electron microscopy I was curious about the scale of images made using optical microscopes. Some contributors to Balsam Post were careful to superimpose scale bars on their images. Some others gave a magnification number, which was normally simply the magnification of the objective x that of the photo eyepiece, and of course bore no relationship to the size at which it was reproduced on the printed page. Mostly, there was no indication of scale at all! In fact, even after I became Editor of Balsam Post I had no clear idea how to generate an accurate scale bar to go with an image. However, as Editor I was in a position to commission and article for my “Back to Basics” series from a contributor who did! I also jointly authored a more general article on how to “calibrate” an optical microscope using a stage micrometer.

Measurement of dimensions in the SEM

The SEM generates scale bars automatically, and displays these in the data area below the image. I seldom gave any thought as to whether the scale bar was actually accurate, but eventually became curious enough to purchase the SEM equivalent of a stage micrometer. This is a Silicon wafer etched into grids of squares with a spacing of 10 µm with a gap separating these squares of 1.9 µm (Figure 1).

For Figure 1B I have superimposed scale bars, as measured using my SEM, and these show the spacing of the squares measured as 10.13µm, which is accurate to around 1%. The width of the “gap” between the edges of the squares (specified as 1.9µm) is measured as 2.0 µm (around 5% high), but at this scale the precise position at which the measurement bars are located can make a significant difference to the reading. Of course, I am making the assumption that the accuracy of the fabrication of the test object is at least an order of magnitude better than what I am trying to measure. Given that the test object is engraved on a single wafer of silicon, and knowing the accuracy to which semiconductors can be fabricated on silicon wafers, I would be happy to take the accuracy of the dimensions on the test object to be highly accurate.

There are two other interesting observations that can be made about Figure 1B. The first is that the magnification value given in the data bar beneath the images indicates 20kx, but I know that the magnification value I selected on the user interface was 10kx. This is a strange feature of my TESCAN MIRA. Because I am saving images at twice the "standard" digital resolution of the image area on the SEM screen (1024x768), the SEM doubles the magnification value. The message here is to never try and scale an image directly off the screen based on the magnification, even if the screen zoom value is set to 100%. The Field of View (FoV) value remains correct though, so it is safe to scale using that value.

The second observation is that there are bright lines beneath the horizontal sides of the squares but less so above the horizontal sides, while the vertical sides have (thinner) bright lines down each side. I did wonder whether this is something to do with the way the test object is manufactured, so I rotated the specimen by 90 degrees in the SEM. I found that the relative brightnesses of the horizontal and vertical sides remained the same; that is, the relative brightness of the rulings was dependent on orientation in the chamber). From this I inferred that the effect is due to the use of the secondary electron detector, located "at the top of the screen". The operation of this detector is complicated. It can be envisaged as a directional light source illuminating the specimen, with the observer viewing the specimen along the axis of the beam. My interpretation of this is that the4 "bars" are, in fact channels engraved into the silicon substrate. The vertical side at the top of the channel is invisible to the SE Detector, while the vertical side at the lower edge of the channel is within view of the detector, and, what is more, being vertical a large number of secondary electrons are emitted. (The more vertical a feature on the specimen is, the more secondary electrons are emitted, and this gives the variation in brightness across a surface that constitutes the image.)

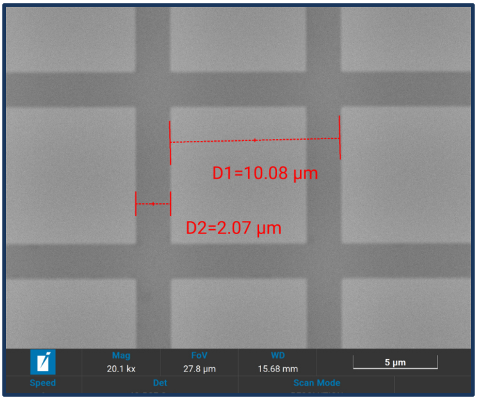

Fig. 2: Image of the same grid as used for Fig. 1, but made using the BSED

The backscattered electron detector (BSED) is a quadrant of photo-sensitive elements grouped around the beam aperture, and the image provided by this arrangement is much more uniform in appearance (Figure 2). The low contrast can be attributed to the fact that the sample is made of one single material (silica), so there is no elemental contrast. As is to be expected, the measured dimensions of the grid imaged using the BSED are much the same as with the SED.

I stated earlier that one possible reason for the unexpected appearance of the image made using the SED was the fact that the detector is mounted off to the side of the chamber, and creates “modelling” effects consisting of shadows and highlights. I do have a different detector, called the in-beam SE detector. This is mounted within the column. Secondary electrons spiral up the electron beam (don’t ask me about the physics of that, please) and are detected within the actual column. The advantage of this detector is that any secondary electrons that might be created by electrons bouncing off the sides of the chamber, or alternatively being emitted as a backscattered electron breaks the surface of the specimen, are not detected. This leads to higher resolution images. In fact the in-beam secondary electron detector gives the best resolution of any of the detectors of my SEM. This is specified as 1 nm.

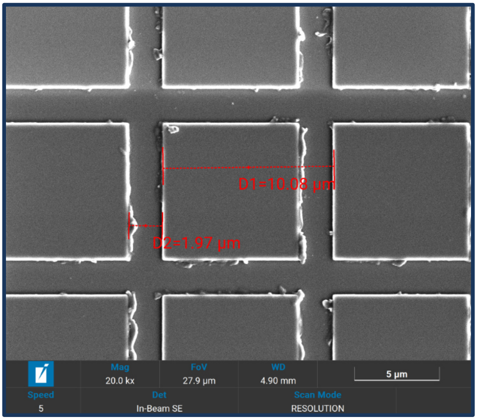

Fig. 3: Grid imaged using the in-beam SE Detector.

For completeness, an image of the grid taken with this detector is provided in Figure 3.

When I started writing this edition of SEM Diaries I simply intended to show that one could check measurements using a standard ruled specimen. I have to admit that my curiosity got the better of me, and I decided to illustrate the effect of using different detectors as well!

Checking the accuracy of EDS

EDS, short for Energy Dispersion Spectroscopy, is a technique for determining the chemical composition of a specimen at any location on its surface, as regular readers of SEM Diaries will know. You might also remember, that in SEM Diaries - 41 I commented on a mineral that resembled iron pyrites (FeS_2) optically, but the EDS system that I am currently using suggested it was a different chemical - ferric sulphide (Fe_2S_3). The outcome was that my collaborators on that particular project spent considerable time trying to think of a reason why that part of the specimen had that particular composition.

Assuming that the EDS detector and the test object are maintained at the correct angle relative to each other, and that the specimen is polished flat to a surface finish of better than 1 µm, a well-calibrated system is capable of measuring composition to an accuracy of around 1%. However, the software associated with the EDS system is expected to make a number of “assumptions” or estimates as to the elements present and their quantities, especially when some energy bands overlap. (I do not propose to explain how EDS works in this article. I have given more details about EDS in SEM Diaries 31, January 2023.)

Coming back to the the so-called Fe_2S_3, I for one was convinced that the composition must be measured correctly, as the relative proportions of the two elements were in the ratio of 2:3 to a high accuracy! Anyhow, to cut a long story short, I decided to consult someone I know who works in the electron microscopy laboratory at Plymouth University. I met her at a conference and described our problem, and she offered to measure the sample in her lab. It "just so happened" that, in anticipation of her agreeing, I had an embedded specimen in my pocket, which I was able to hand over there and then.

The result, following EDS on the Plymouth SEM, identified the sample as iron pyrites. I was disappointed, though not particularly surprised, and was very glad that we had not made a big deal of the discrepancy in our joint paper.

Although I had no reason at all to dispute this result, I decided I would obtain some "guaranteed" iron pyrites and see if that was identified as Fe_2S_3 on my SEM.

So, how does one obtain "guaranteed" iron pyrites, or any other mineral for that matter? Well a couple of years ago another contact at Plymouth had put me in touch with a company called "Micro-Analysis Consultants" (MAC). They make standards specifically for metallurgists and mineralogists. They have a catalogue range, but are also willing to make a standard with any selection of minerals or metals chosen by the client.

I came up with a list of 12 minerals and two metals that would be useful in calibrating my EDS system, and of course included iron pyrites in the list. In due course the standard arrived . It consisted of a brass disc 32mm in diameter, with 14 cavities about 2mm in diameter, each with a different sample in it (Figure 4). The face had been polished to a sub-micron finish and carbon coated, and it was packed in its own box with a memory stick containing the calibration certificate.

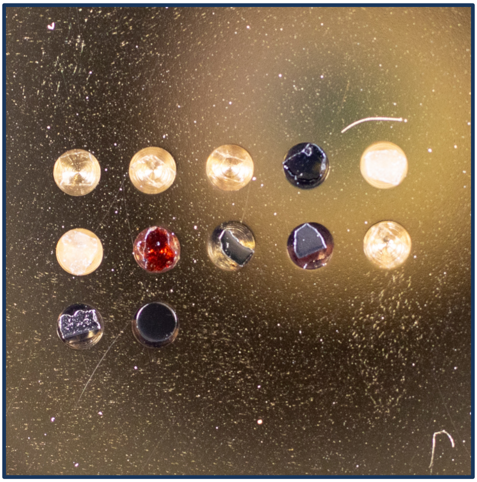

Fig. 4: Detail of the MAC standard, showing the cavities containing the mineral samples. The quality of the photo is much worse than the appearance of the standard, which is highly polished and hence very reflective.

Some of the minerals are hygroscopic, and some can be damaged by the electron beam. For this reason, MAC recommend storing the standard under vacuum or in a desiccator, along with refurbishment and re-calibration every two years. I guess this would consist mainly of re-polishing the surface and re-coating with carbon. This would be expensive, but hopefully significantly less than the four figure sum that was the cost of the brand new standard.

Needless to say, as soon as I received it mounted it in my SEM and did an analysis of the iron pyrites. The result, as I had expected, was the same as I had measured from my own sample!

So, given that "my" EDS system was so inaccurate in analysing that sample, what was the chance that it would be equally un-trustworthy in characterising other elements? My next step was to analyse all the other samples on the MAC standard and compare them with the calibration certificate. (This certificate provided the result of an analysis of each mineral on my particular standard using their highly accurate EDS system or some similar process.)

Fortunately, the analyses of most of the samples were found to be reasonably similar to that stated on the certificate. The iron pyrites result was by far the worst of those analysed.

So, what can I do with a dodgy EDS system? Can it be calibrated? Well, the loan system that I have does include a built in calibration routine but that did not make any difference to the result. The answer is to return the loan system and indulge myself by buying a brand new and up-to-date replacement, at great expense. This is made by Oxford Instruments (OI), who are the go-to suppliers of EDS in the UK, although there are other suppliers such as EDAX who are better known in the USA, and Brucker, who originated in Germany. In fact, TESCAN do have a version of EDS software that runs on my SEM, using detectors from EDAX. I viewed a version of this during my visit to their site in Brno in 2023, but their quote for their system was not dissimilar to that from OI. I decided that given how ubiquitous Oxford instruments are in the UK, it would be much easier to discuss use and results from an OI system with other users than to go down the TESCAN route.

The new system is due in mid-December. Watch this space!