SEM Diaries - 14

What's in a Filament?

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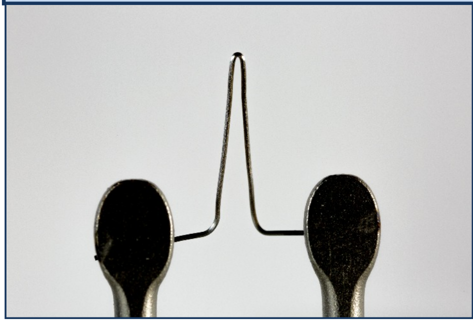

Fig. 1: New Tungsten Filament for my SEM

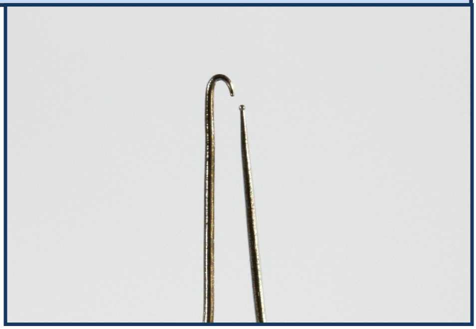

Fig. 2: Detail of Blown Tip of Filament

There are basically three ways to generate an electron stream in an electron microscope, whether it be an SEM or a TEM. The simplest, and that used on my SEM, is to pass a current through a piece of bent tungsten wire (Figure 1) to make it very hot, and then the electrons are able to escape from the material. The second is to use a crystal of lanthanum hexaboride (known as LAB6), or similar material, in place of the tungsten wire. This is a more efficient emitter of electrons and therefore provides a "brighter" beam. The third is to use a "Field Emission Gun" or FEG. Put simply, this strips the electrons off a (normally) hot tungsten crystal source with an extremely fine point by "pulling" them off with an electric field. The FEG provides a significantly brighter beam than even the LAB6 gun, and with this gun the electron beam can be made of significantly smaller diameter, providing higher resolution than either of the other two.

As in everything, you get what you pay for. A replacement tungsten filament costs of the order of £25, and lasts anything from 40 to 80 hours depending on how careful the operator is. LAB6 sources cost in the hundreds of pounds, and last longer, while FEG sources last between one and two years, and cost thousands. (All figures very approximate.) Furthermore, the vacuum requirements increase with the source brightness, compounding the cost of the electron microscope.

Replacing a tungsten filament when it reaches the end of its life - it blows in the same way as a conventional light bulb does - is not straightforward. The filament resides within a closed cylinder known as the Wehnelt cap (Figure 3). This has a fine hole in the end behind which the filament resides. The filament must be aligned accurately with the hole, not only axially, but also the distance of the filament tip behind the front of the hole (0.25 mm) must be set correctly.

When the filament blows, molten tungsten is sputtered around the inside of the end of the Wehnelt cap. This may sometimes obstruct the hole in the cap end or, worse,

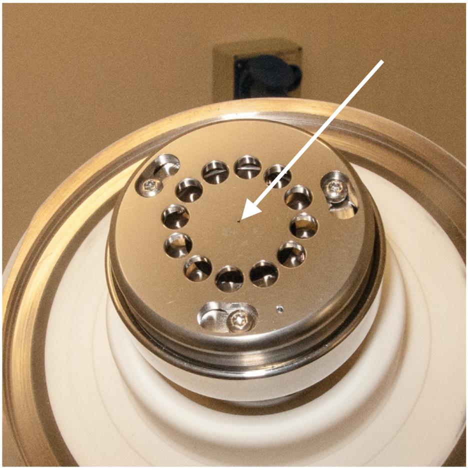

Fig. 3: View of the Wehnelt cap in situ on my SEM (with the column top hinged back). Note the very small central hole (arrowed), hiding the tip of the filament.

land on the anode's platinum aperture further down the column. The steps that need to be gone through to replace the filament are basically:

- Bring the SEM back to atmospheric pressure and allow the Wehnelt assembly to cool.
- Remove the Wehnelt assembly from the top of the column of the SEM and take out the blown filament.
- Clean the tungsten deposit from the inside of the cap and from the hole with metal polish, cotton wool buds and cocktail sticks.
- Remove the residue of the metal polish by dissolving it in acetone in an ultrasonic bath - changing the acetone at least once. Dry in an oven.
- Reassemble the Wehnelt assembly with a new filament and align the tungsten tip with the central hole in all three planes.
- Replace the Wehnelt assembly in the SEM, switch on, pump down and carry out a number of adjustments to set the correct filament voltage and align the beam once more.

On a good day I can do all this in about two hours. As you might imagine, I dread the thought of the filament blowing a few minutes into a demonstration to school children or anyone else!

Recently my "favourite" filament blew. Nothing unusual with that, you might think, especially as it had 52 hours of life already expended. But I called it my favourite because I was nurturing it to see just how long I could make it last.

Normally when a filament blows there is a definite breakage visible to the naked eye, near the tip. This is illustrated in Figure 2. When I removed my favourite filament I could see no break at all. I checked for continuity, which confirmed an open circuit, and I eventually determined that this arose because the tungsten wire had come detached from one of the legs to which it is secured. This was sufficiently unusual to me that I drew it to the attention of my filament supplier, who immediately supplied two free filaments on the condition I returned the damaged one for him to send back to the manufacturer. Bargain!

When I was fitting a new filament, I accidentally brushed the tungsten with a finger and bent it. In attempting to straighten it "it fell off in my hands". Given the mistreatment, this was not unexpected, but I wondered whether the wire had been broken or just become detached from the legs. What better way to determine this than to examine the legs of the filament in my SEM!

It is probably best, at this stage, to include a picture of a complete filament, as supplied by the manufacturer. This is shown in Figure 4. It shows how the legs are fed though and secured to a ceramic disc with an orientation slot. The legs are used as male contacts into a socket on the top of the column. This filament is actually

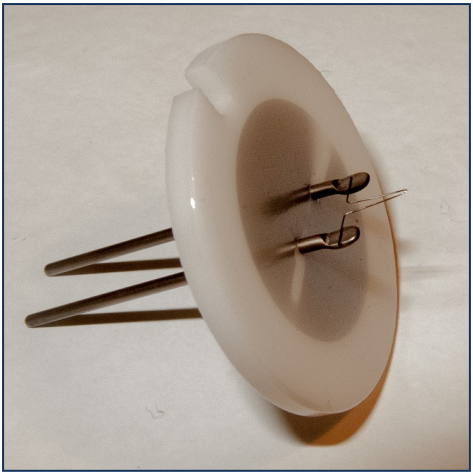

Fig. 4: A complete (but blown!) filament assembly, showing tungsten deposit on the insulator.

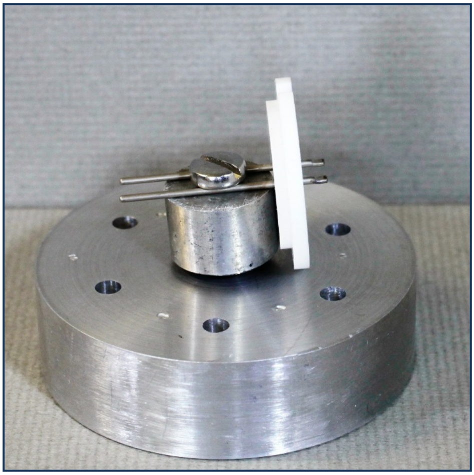

Fig. 5: Simple jig for supporting the filament in the SEM chamber. In this photo it is supported, outside of the SEM in a stub carrier.

blown, and the dark matter on the top of the base is evaporated tungsten.

I should also say that Figures 1, 2 and 4 are of a filament from a different manufacturer than the ones that I was having trouble with.

So, I have a filament to look at, but how do I mount it in the SEM? The solution I arrived at was very simple. I took a length of 1/2" diameter aluminium rod, about 1/2" long and stuck it on top of a standard stub using a conducting carbon pad, as used for mounting specimens. Before sticking these components together I drilled and tapped the Al cylinder with an M4 thread. An M4 cheese head stainless steel (non-magnetic) screw securely holds the filament legs, providing the required conducting path to the stage. This is shown in Figure 5.

I was fascinated by what I found when I finally pumped down the SEM and imaged the filament.

Figure 6 shows one of the feet of the filament I damaged. (This is inverted compared to the orientation of Figure 1.) What can be seen is tungsten wire resting in a groove on one of the feet . At the right hand end it would appear that a piece of tungsten wire has been broken off (a), and this is consistent with the condition of the tungsten loop seen under the light microscope. There is a gap between the wire and the groove in the middle (b), and nowhere is it clear how the wire is retained in the groove. The light toned particles are charged, so must be non-conducting, and could be from the polystyrene packing in which the filaments are supplied.

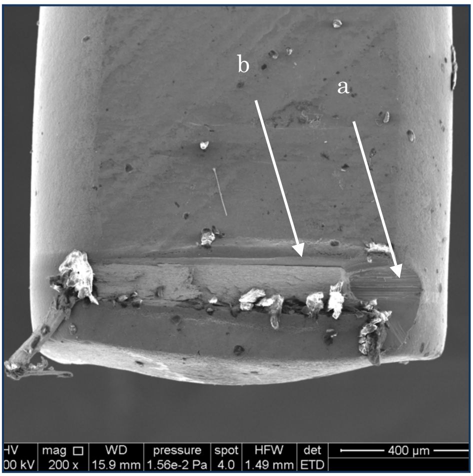

Fig. 6: One foot of damaged filament, with features mentioned in text indicated.

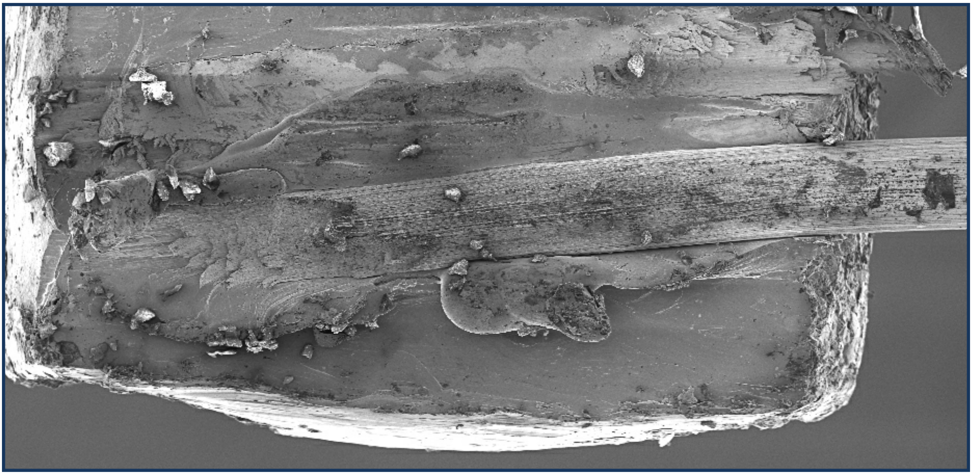

Fig. 7: Detail of the tungsten wire to foot bond on a new filament.

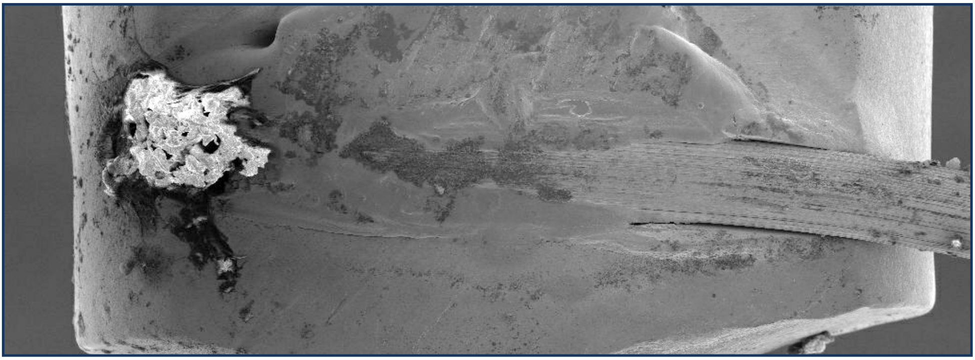

Fig. 8: Foot of a different new filament showing similar manufacturing “artefacts”.

The filament of Figure 6 was brand new, but I had mishandled it, so I decided to look at a new filament straight from the packing. The result of this is shown, enlarged, in Figure 7. Key features to note are: the tungsten wire of the filament projects out to the right; there is a clean boundary between the tungsten and the groove in the foot along much of the right half of the foot; there appears to be tearing of the tungsten wire at the left hand end, as if it had been separated from the rest of the reel of metal using the properties of metal fatigue (bending a wire back and forwards until it breaks).

It is not clear exactly how the tungsten wire is secured in the slot. The area around the broken end of the tungsten does show some attempt at what might be welding, as does the blob underneath the tungsten to the right, although that does not actually seem to have bonded. If this were a wire on a printed circuit board I would say that the soldered joint was exceedingly “dry”!

I decided to look at a different unused filament, and the image is shown in Figure 8. This shows similar construction and similar issues, although the area of “successful” weld is greater. Of interest in this micrograph is the charged non-metallic material to the left of the image. Looking at it under a stereo microscope I determined that this is white material, in the cavity where the tungsten wire has been broken off during manufacture. I have not attempted to probe it with a tool, but it does not appear to resemble the white polystyrene packing material. Perhaps it is some sort of flux?

So, what is the conclusion from this exercise? I do not know enough about the manufacturing methods used to comment with any authority, but the micrographs do not "look good" to me. On the other hand, if you study manufactured items at degrees of magnification much higher than those normally used to inspect them then many other components might also not "look good". For example, the milled end of a metal bar may look and feel smooth to the naked eye and finger, but at high magnification under an SEM may look more like the Himalaya!

Having already been in touch with the suppliers of the filaments over the initial problem, I phoned them again to explain that I was surprised by what I had seen and that it might go some way to explaining the original failure. Shortly afterwards I was contacted by the Production Manager of the company that actually manufactures the filaments. (They manufacture filaments for a wide variety of electron microscopes and other electron beam equipment.) I sent him a report, illustrated by some of the micrographs in this article, and despite his being on holiday he has distributed this among his "team" and will be consulting with them on his return.

Updates on earlier issues

In SEM Diaries - 13 I described the steps I was taking to keep track of what is on any individual stub, by using pre-numbered stubs at great expense. Well, I have got into a routine with doing this, and it works quite well. Even so, I have come across instances when I have entered the wrong details in my stubs spreadsheet. With species and anatomical names going round in my head, I sometimes choose the wrong one from that melée! The situation is normally corrected as soon as I see the image appear on the SEM screen. Of course, if I use an angled stub, this will not be pre-numbered, so even more care is needed in traceability.

Talking of angled stubs, these featured in SEM Diaries - 12. What I did not mention was the difficulty of mounting a specimen on a 45° slope. This is attainable if the slope has a sticky pad on it, but if I was intending to use conducting glue, then - not a hope. I had resorted to pressing the stub in a bed of BluTack to make the sloped face horizontal, so that I could rest the specimen there as I gently applied the glue.

I finally got round to making a jig that would not only hold the stub at 45° (making the mounting face horizontal) but would also support it at precisely the same level as my mounting block for standard stubs, thus avoiding the need to re-focus the stereo microscope I use for detailed work. This jig is illustrated below (Figure 9). This may appear to be quite a simple construction. All the same, I managed to break two 3mm end-mills (at about £9 each) due to my own stupidity, while making it!

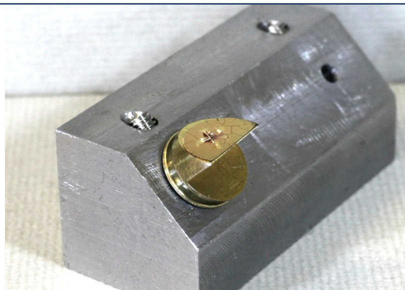

Fig. 9: Jig for supporting 45° stubs in a horizontal position