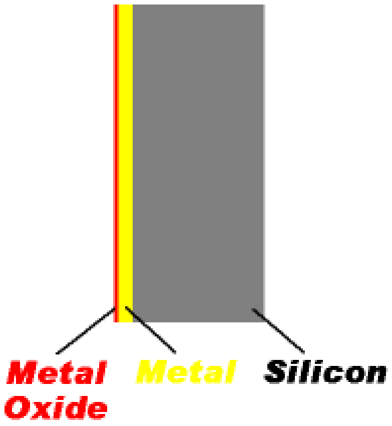
Electron Microscopy Report

This is an exercise where we were given the challenge of being handed a device – a micro-actuator – a brief of how it might be made, based on how such devices work and light microscopy images and asked how to investigate it further. The actual device didn't exist – it was a conceptual challenge.

The hypothesis of the component design describes the one dimensional structure through the component. Light microscopy has clearly determined the existence of two layers and the possibility of a third, but details and material composition are not clear.

First stage for the sample preparation would be to cut it through, hence exposing a surface that should demonstrate the cross section theorised. As the sample is believed to be largely conducting or semi conducting, metal coating is not necessary. However, polishing the cut edge should improve the image.



Hypothesised device cross-section

Initial imaging of the device (knowing the basic results from optical microscopy) will be under a Scanning Electron Microscope. In an SEM, a highly focused beam of high-energy (typically 50-200 keV) electrons are accelerated from a source (typically a heated sharp filament or sometimes a field emission source). These electrons are then focussed into a narrow beam by a series of electromagnetic lenses. Finally a set of coils at the bottom of the electron column scan the beam across the surface. Image resolution is effect by a number of factors, notably the size of the electron "spot" formed by the beam on the surface. The electrons then interact with the sample in a variety of ways.

Some are adsorbed my atoms close to the surface – these atoms then reemit what are known as secondary electrons. These are then collected by a detector. Intensity at the specific scan point can be almost directly correlated into topography of the sample.

Other electrons proceed deeper into the sample and are scattered off the various nuclei. These are eventually reflected back to the surface (or else simply adsorbed into the sample) and are known as back-scattered electrons. The intensity of the BSE image is related to the atomic number of the elements involved, however it typically merely provides contrast between different phases in a material rather than identifying elements.

Yet more electrons penetrate deeper into the sample. They excite the electron shells of atoms contained substantially, and when the atom returns to its ground state an x-ray is emitted. The wavelengths and intensities emitted are elementally unique. X-ray analysis can therefore be used to determine the composition and location of species in a sample.

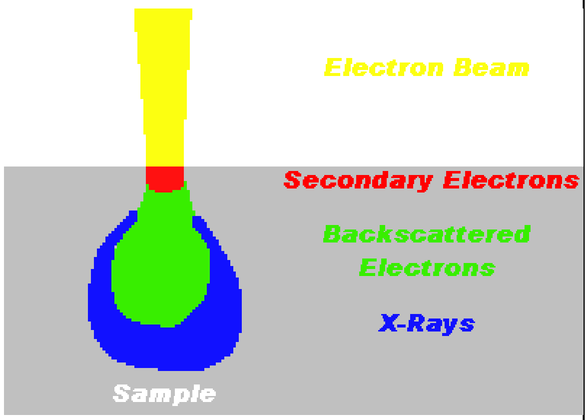


Diagram showing interaction volume and where each signal is produced

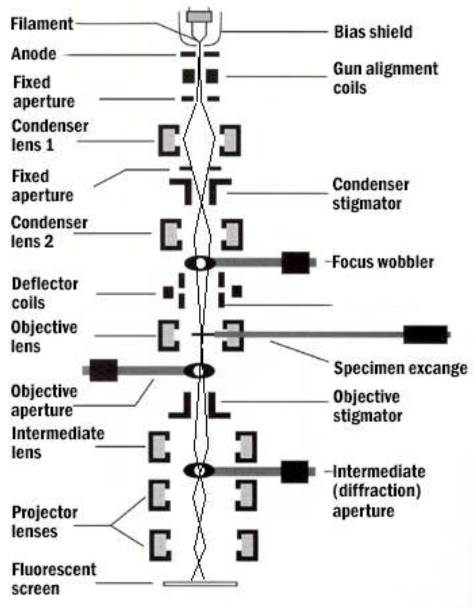
All these effects take place over what is known as the *interaction volume* – the space in the sample where the electrons interact and produce emissions. As the electrons travel deeper into the sample, they also spread more. The effect of this is that BSE and EDX are progressively lower in resolution possible than the topographic SE image due to the greater area over which the interactions happen.

The secondary electron image of the surface of the material may provide insight into the means used to lay down a layer (sputtering, evaporation and other deposition techniques all leave unique grain structures which may be visible topographically). However this will still be quite smooth and the SE image of the cross section produced by cutting should be almost flat.

However, the backscattered electron image will hopefully provide details about the structure. Contrast should be clearly visible between the different layers in the sample, as they are composed of different elements. EDX could then determine what elements are present within the sample, followed by an x-ray map to show where these respective elements are located.

If a sample of the device that had failed in use could be acquired, then examining this under the SEM would also be useful. SE imaging could potentially show any physical results of the failure, for example thin layers coming away from the surface. EDX could also show if there was any change in the elemental composition of the sample that may effect its operation.

Sample preparation for TEM is somewhat more difficult. A 3mm diameter section needs to be taken from the sample. The area we are interested in imaging should be highlighted by the SEM analysis, however our initial hypothesis suggests that a cross-sectional piece from near the metal-oxide surface would be the most useful to study. This sample then needs to be thinned to approximately 1 µm. Ion beam milling would seem a suitable way of carefully and controlled thinning of the sample disk.



Schematic of a TEM and ray diagram for electrons witin. Does not show EDX or EELs devices

A tunnelling electron microscope (TEM) accelerates electrons from a source, much like in SEM however typically to much higher energies/. These are then focussed to a beam, however unlike in SEM the ‘spot’ where the beam hits the sample is quite wide. A large number of electrons are then transmitted through the sample. These electrons are then focused beyond the sample and produce an image on a plane somewhat behind the sample.

The primary image produced by a TEM is one showing mass-thickness contrast – that is to say images where both higher mass and higher thickness produce darker images. In our sample, we would hope then to be able to see definition between the different layers of the device.

Diffraction contrast is another primary imaging mode of the TEM. When electrons are transmitted through the sample, many of them are diffracted by the crystal structure of the sample. This effectively produces several overlapping images. Through selecting one diffraction spot, both the contrast in a mass-thickness image can be improved (due to no interference from diffracted electrons) and also allows the discernment between individual crystals – their different alignments mean they all diffract at different angles. This diffraction contrast should assist in determining if the silicon wafer really is monocrystalline, however as we are only imaging a small area in the TEM, even if the image suggests the silicon is a single crystal it is possible that the crystals are just large and all we are imaging is one crystal, but the entire silicon substrate is polycrystalline.

EDX could once again be used to determine the precise nature and distribution of elements through the surface of the material. The x-rays are generated in much the same way as in an SEM an analysed by a similar detector. An x-ray map should once again show where the elements are located.

With the TEM there is also access to EELS – electron energy loss spectroscopy. This technique involving the study of how much energy is lost by transmitted electrons and can give details on not only what elements are present in a sample, but how they are bonded as well. This could give critical information on the bonding of the metal oxide layer (if this is present) and precisely what leads to the debonding failure. As with EDX, the EELS data can be localised to specific locations on the sample.

Once again, the ability to image the same area from a failed sample and to compare and contrast, noting any significant differences in structure or composition could provide very valuable insights into the cause and nature of the failure.

If the failure of the device is indeed caused by a debonding effect between the metal oxide and the metal, then a few approaches can be used. Perhaps a different metal oxide with greater affinity for the electrode could be used. It could be that the extremes of temperature are causing the problem – protecting the actuator from such high temperatures in the finished product may then be the best solution. Changing the electrode surface to mechanically as well as chemically affix the oxide layer could help prevent it from becoming unattached.