

Introduction

Historically energy dispersive X-ray spectroscopy has been focused towards high energies where X-ray lines are well separated and the X-ray background is low. Recently the demand for higher spatial resolution, whether that is when analysing semiconductor devices or measuring precipitates in metals, has increased dramatically. To ensure maximum sensitivity when working under these demanding conditions the **Ultim®** range of EDS detectors combine the largest area SDD with optimised geometries to consistently deliver a higher count rate.

High spatial resolution is necessary when analysing specimens with very small structures such as the NAND Flash memory device studied in this note. There are a range of features that vary in size from 500nm down to less than 10nm. The sample also contains a large number of elements that cover a wide X-ray energy range. In order to increase EDS mapping resolution it is necessary to minimise the volume from which you generate X-rays. Fig. 1 shows the X-ray generation volume for L-series X-rays in pure Fe and the decrease in this volume as incident electron energy is decreased from 20kV to 1kV.

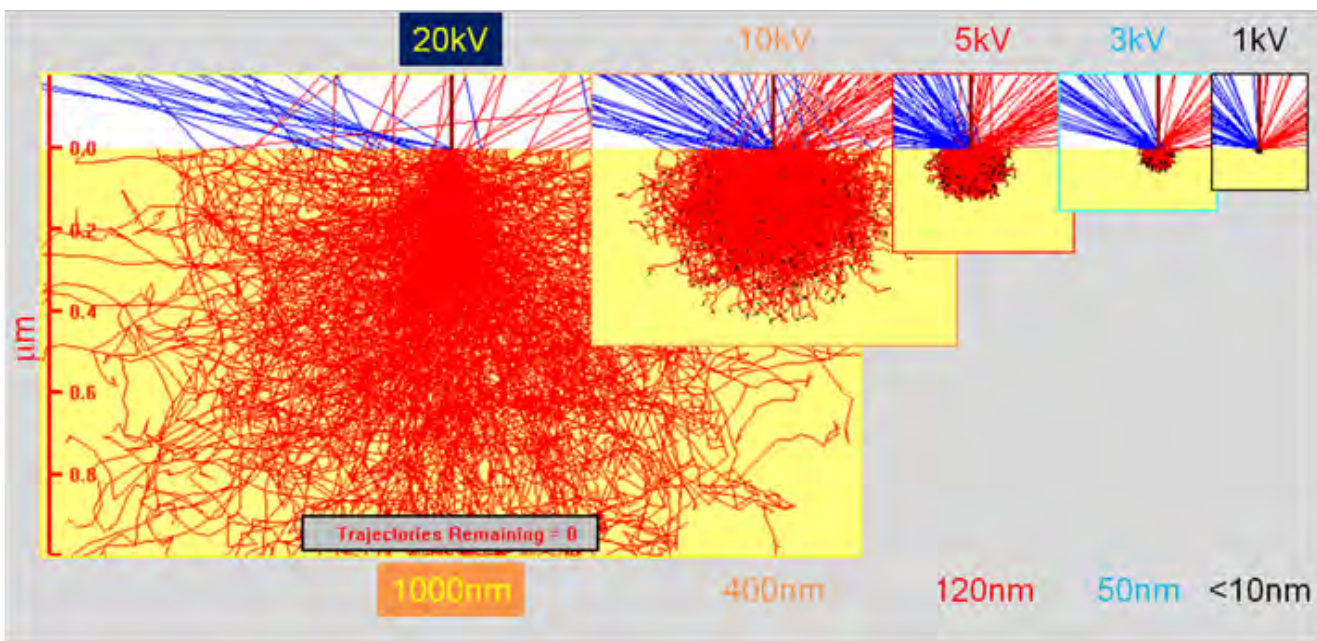


Fig. 1. Monte Carlo simulations of the generation volume of Fe L α X-rays in pure Fe at varying electron energy.

However, as the energy of incident electrons is reduced the number of X-rays as well as the number of available X-ray lines is also reduced. For example if we were to take a specimen of pure iron and then under exactly the same microscope conditions vary the incident electron energy a large change in the number of X-rays generated would be observed. Moving from 20kV to 10 kV decreases the number of counts in the K series X-ray lines by 90% whilst the L series remains unchanged. As electron energy is further reduced to 5kV and even 3kV the counts into K series disappear entirely and count rates into the L series are decreased by 50% and 80% respectively.

To overcome this loss in counts the sensitivity of an X-ray detector must be maximised. The sensitivity of a detector is defined by the solid angle that it subtends. Generated X-rays are emitted in a hemisphere from the surface of the specimen at the incident beam position. The solid angle of a detector is the amount of this hemisphere that it captures and is in its simplest terms defined by the approximation: area of the detector (A) divided by the square of the distance from specimen to detector (d).

$$\text{solid angle} \sim A / d^2 (1)$$

By maximising A and minimising d the solid angle can be increased and the number of counts collected increased. When dealing with very low count rates such as in the case of very low electron energies it is important to have the largest detectors as close to the specimen as possible and therefore achieve the highest sensitivity. This can be shown practically on the flash memory device, Fig. 2.

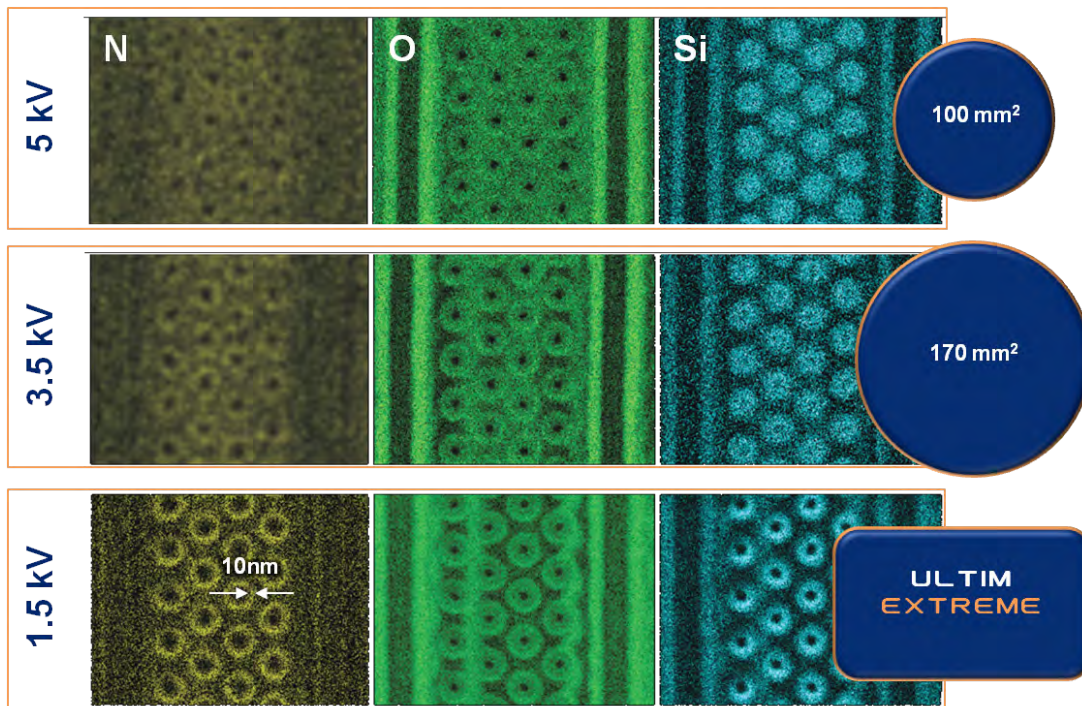


Fig. 2: EDS maps for N, O, and Si collected from a NAND flash memory device with **Ultim** Max detectors of 100 mm² and 170 mm² sensor areas and **Ultim** Extreme.

To maximise spatial resolution it is important to not only minimise electron energy but also the incident beam current due to the Boersch effect [1]. With this in mind the experimental conditions were fixed with an incident electron beam current of approximately 160 pA. At 5 kV with the **Ultim** Max 100 this resulted in an input count rate of 1600 cps when at the analytical working distance of 7 mm. Under these conditions EDS maps were collected for 1 hour. At 5 kV it is possible to easily distinguish the major structures in the specimen; these are the oxide and silicon regions around and in between the tungsten word lines, the maps of which are not shown. In the oxygen map some faint structure is visible around the “holes” in the map. Faint structures are also observable in the nitrogen map.

As the electron energy is reduced to 3.5 kV an **Ultim** Max 170 can be used to maintain the 1600 cps count rate. When the specimen is mapped for an hour significantly more structure is observable. In the oxygen map it is clear that there are separated rings around the previously observed “holes”. These correspond to the rings in the silicon map and also ring structures are now observable in the nitrogen map that suggest a complex layered structure within each of these rings.

This shows that when working at very low energies to improve spatial resolution in bulk samples sensitivity is the dominant factor in achieving accurate elemental maps. Sensitivity to very low energy X-rays can be further increased by removing the X-ray window, which typically absorbs up to 100% of sub 1 kV X-rays, depending on their energy. **Ultim** Extreme is a windowless detector which is also optimised to work at very short working distance to further improve spatial resolution. On the same microscope at the same beam current, the input count rate when using **Ultim** Extreme at a 4.5 mm working distance and incident electron energy of 1.5 kV was 5200 cps. The specimen can once again be mapped, however due to the high input count rate mapping time can now be reduced to 30 minutes. At 1.5 kV significantly more information is observed in the EDS maps with clear definition of the N ring structures in the charge trap layers within this device. Very surface specific oxygen structures are also observed such as the artefacts created by the variable milling rate of the broad beam ion mill used to prepare the specimen. When using very low energy such as 1.5 kV conventionally used X-ray lines for elements such as silicon and tungsten are no longer available for analysis. Instead low energy lines such as silicon L ℓ (0.092 keV) and tungsten N 5 N 6 , 7 (0.210 keV) must be used. Fortunately due to the windowless construction of **Ultim** Extreme, collection efficiency for these lines remains greater than 80% so they can be used to accurately map the distribution of those elements in a sample. As shown in Fig. 3.

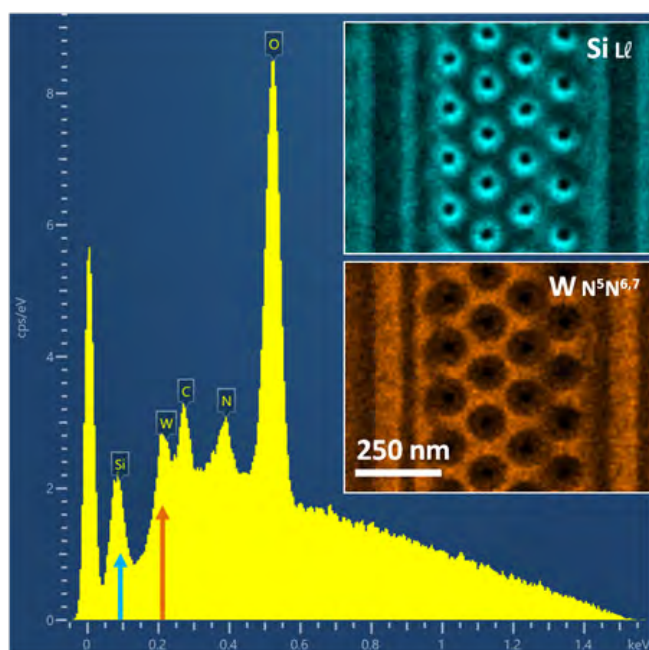


Fig. 3: 1.5 kV EDS spectrum showing low energy X-ray lines of Si L ℓ (0.092 keV) and W N 5 N 6 , 7 (0.210 keV). Inserts show elemental mapping using these two X-ray lines.

Just as important to acquiring accurate data is the ability to process the data. **AZtec** TruMap deconvolutes peak overlaps and removes the X-ray background in real time. This ensures that the structures in the elemental maps are real and are not the result of spectral artefacts. This is shown in Fig. 4. When using electron energies of between 2.5 kV and 5 kV silicon and tungsten are mapped using their K and M lines, 1.74 keV and 1.77 keV respectively. These X-ray lines overlap strongly. This means that when mapping Si K with a window integral (SmartMap) tungsten as well as silicon structures are observed. Using TruMap solves this overlap to display only the silicon counts and the real silicon distribution in the specimen.

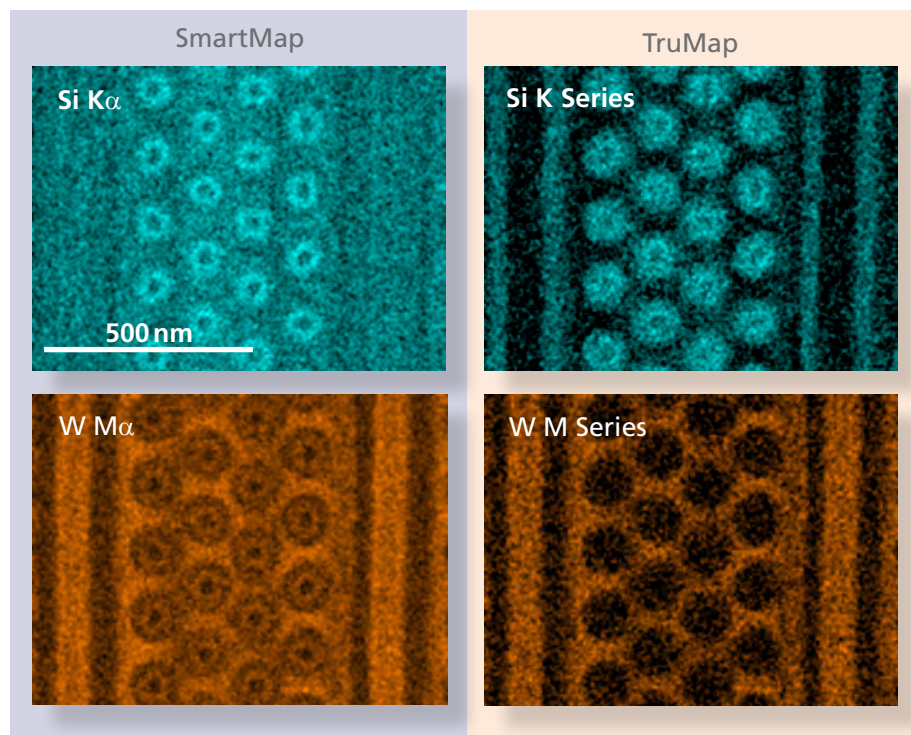


Fig. 4: Si and W maps collected at 3.5 kV processed using window integral mapping and **AZtec** TruMap.

Conclusion

By increasing detector sensitivity either by sensor size, sensor-sample distance, or window removal, it is possible to collect X-ray counts at high resolution imaging condition and achieve elemental maps with sub 20nm spatial resolution even on the lightest of elements. This combined with the data processing of Aztec TruMap allows the accurate mapping of elemental distributions in complex specimens with nano-structures such as a NAND flash memory device.

[1] Boersch, H., Z. Physik, 115 (1954)

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