Introduction

In Transmission Electron Microscopy (TEM) there are two 'go to' techniques for elemental analysis: Energy Dispersive X-ray Spectroscopy, (EDS), and Electron Energy Loss Spectroscopy, (EELS).

EDS is a mature technique that can be used for most specimens. The intensity of generated X-rays is proportional to the mass thickness of the sample. However, this can become a limitation for very thin specimens or those comprising light elements. On the other hand, EELS is more suitable for thin samples where the thickness is less than the inelastic mean free path of electrons in the material. EELS does, however, give high signal to noise for light elements. Simultaneous acquisition of both signals is a powerful tool for materials analysis.





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Simultaneous EDS & EELS

EELS

EDS



Fig. 1a-f. Maps of EELS and EDS signal for N, O, Si, Ni W and Ti acquired simultaneously

Semiconductor analysis by simultaneous EDS and EELS

A semiconductor device thin section was analysed to show the benefits of performing simultaneous acquisition of EDS and EELS data. Using an Oxford Instruments Ultim Max TLE windowless silicon drift detector for EDS and a Gatan Quantum GIF for EELS, it was possible to collect a wide range of chemical information with relatively short acquisition times. The GIF was used in Dual EELS mode to optimise acquisition for a wide energy range of elements. This allows spectra to be collected from two distinct energy ranges with differing energy resolutions. This is imperative for accurate analysis of light elements such as O and N as it allows high energy resolution spectra to be collected in the appropriate range while still collecting a low energy spectrum which includes a zero loss peak for later calibration. Low loss spectra were collected in the range 0-460 eV while core loss spectra were recorded in the range 400 eV-875 eV, both with an energy resolution of 0.25 eV. These energy ranges allow measurement of the majority of elements within the specimen. EDS spectra

were collected in the range 0-10 keV with a live time of 0.01 s per pixel, this energy range allowed the signal for every element in the sample to be collected in each pixel. These elemental signals were all collected simultaneously in a single acquisition.

As is shown in Fig. 1, both techniques give very clear maps of the elemental distribution throughout the specimen. The signal to noise in the EELS maps is however better for the light elements shown in Fig. 1a-c. For heavier elements the opposite is true, as is shown in the maps for Ni in Fig. 1d. Due to specimen thickness and acquisition conditions, the W region in this specimen is difficult to map with these EELS conditions. However, due to W's large elemental mass it is the perfect candidate for EDS. By combining EELS and EDS simultaneously, it is possible to acquire elemental maps of a wide energy range of elements quickly and accurately (Fig. 2).

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Because EELS is capable of measuring very small changes in energy which are typically not resolved by EDS, it is possible to map not just elemental information but also specimen chemistry. For example, this specimen and many similar semiconductor specimens contain a number of Si compounds. The absorption edges of Si (99 eV), SiO₂ (106 eV) and SiN (107 eV) are all slightly shifted in energy. The EELS signal for each of these compounds is shown in Fig. 3. This means that if an EELS map is collected at sufficient energy resolution then it should be possible to map not just the presence of Si, O and N but the actual distribution of Si, SiO, and SiN. Acquiring EELS spectra at sufficient resolution to generate these maps again requires acquisition conditions that are not optimised for heavier elements and fast acquisitions times. Once again, using TEM EDS as a complimentary simultaneous technique, it is possible to fill in the distribution of these elements and generate a map which contains a full picture of the elemental and compound distribution in the specimen.



Fig. 2.a) EDS and EELS combined spectral image. The colour key shows the elements and which technique was used to analyse them. b) Associated ADF image of the region.



Fig. 3. a) EELS spectra collected in the core loss region for Si, SiN and SiO with the associated spectral images. b) Combined spectral with Si compounds mapped using EELS and heavier elements mapped using EDS.



Application Note





SiC





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Conclusion

The use of simultaneous EDS-EELS acquisition to fully characterise a semiconductor device specimen has been demonstrated. It has been shown that, although each technique individually is able to characterise the whole specimen, there are limitations to consider when optimising analysis. By using the EELS optimised for light element analysis and the heavy element capabilities of EDS it is possible to generate full specimen maps with improved signal to noise in shorter acquisition times. It was also shown that by collecting EELS spectra at high energy resolution it is possible to identify not just single elements but also compounds. Using this technique the distributions of Si, SiN and SiO can be mapped in a specimen with high resolution while also using simultaneous EDS acquisition to map those elements not observable in the chosen EELS energy range.

Data collected in collaboration with the University of Warwick, UK

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The materials presented here are summary in nature, subject to change, and intended for general information only. Performances are configuration dependent. Additional details are available. Oxford Instruments NanoAnalysis is certified to ISO9001, ISO14001 and OHSAS 18001. AZtec is a Registered Trademarks of Oxford Instruments plc , all other trademarks acknowledged. © Oxford Instruments plc, 2018. All rights reserved. OINA/EDS/AN/EDSELS/1118.





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