

Windowless EDS Detection of N Lines and their Practical use in sub 2 kV X-ray mapping to Optimize Spatial Resolution

Simon Burgess¹, James Holland¹, Peter Statham¹ and Conor McCarthy¹

¹ Oxford Instruments NanoAnalysis, High Wycombe, UK

Reducing microscope accelerating voltage for elemental characterization by EDS improves the spatial resolution and surface sensitivity of results, with the potential to reduce emission volumes to less than 10 nm at 1 kV. In addition to instrumental challenges for SEM and EDS systems, the use of very low accelerating voltage reduces lines available for elemental analysis. Below 2 kV many common elements can no longer be analyzed without the use of low energy L lines (e.g. P-Cl), and M lines (e.g. Sr – Sn). This study considers whether N lines are sufficiently detectable and intense for useful nano-characterization, for otherwise undetectable elements at 2 kV Hf – Bi, and 1 kV Ba – Yb.

N-line emission line energies were tabulated by Bearden [1] and have been shown experimentally for W and Ta by EDS and WDS [2]. The introduction of new EDS detectors with improved electronics and windowless operation for maximizing low energy detection efficiency, has allowed the practical use of these lines to be tested. In Fig. 1 spectra from pure metals from Hf-Bi have been collected using an Oxford Instruments 100 mm² windowless X-Max Extreme SDD detector.

Usable intensities for the N₅-N_{6,7} / N₄-N₆ lines have been detected for Ta-Au. For bismuth an intense low energy N series starting at N₇O₅ is detected near 135 eV. As line energy increases the overlap with the C K α peak becomes an issue. For elements such as platinum and gold, there is a requirement for stringent sample preparation and cleaning to remove any carbon from the sample surface.

To test the practicality of using these lines a semi-conductor device containing tungsten structures has been studied. Normally 3 kV is used to excite W M lines, but the W N line can be excited at 1 kV, giving the opportunity for improved spatial resolution. X-ray maps collected at 3 kV show that W M maps (corrected for background and the SiK α overlap) and W N maps (energy-window (ROI)) show the same structures in the sample (Fig. 2). With the availability of W N, a lower accelerating voltage can be used to characterize element distribution in this sample. Reducing the accelerating voltage from 3 kV to 1 kV reduces the generation volume significantly and this can be seen in X-ray maps collected for Oxygen (Fig. 3). The O K α X-ray map at 3 kV shows a mix of poorly resolved surface and sub-surface structures, whereas the distribution of oxygen is clear in the 1 kV map. This improvement in spatial and depth resolution may offer engineers, investigating these structures, capability to do initial investigation in FIB-SEM, before transferring thinned lamellae to TEM for detailed analysis.

Reduction of accelerating voltage as low as 1 kV also limits the availability of low energy M lines, for example for lanthanide elements in ceramics and optical materials. Spectra collected from compounds of Ba (BaF₂), La (LaB₆), Ce (CeO₂), Pr (PrF₃) and Yb (YbF₃) – (Fig. 4) show intense peaks at 78 eV (Ba N), 82 eV (La N), 88 eV (Ce N), 93 eV (Pr N) and 187 eV (Yb N). The energies of these lines agree well with the tabulations of Bearden [1], but seem to have larger intensities than the higher energy N lines from Hf-Au.

Fig. 5 shows the potential for using Ce N and La N lines for X-ray mapping where La/Ce oxide nanostructures, some less than 10 nm in width, have been investigated in a ceramic sample. Due to the close overlap, the N line ROI map combines Ce and La information, but matches the La/Ce M data.

References

- [1] J. A. Bearden, Rev Mod Phys (1967) 39, p78
- [2] A. Scheffel *et al*, Microchim Acta (2008) 161, p471-473

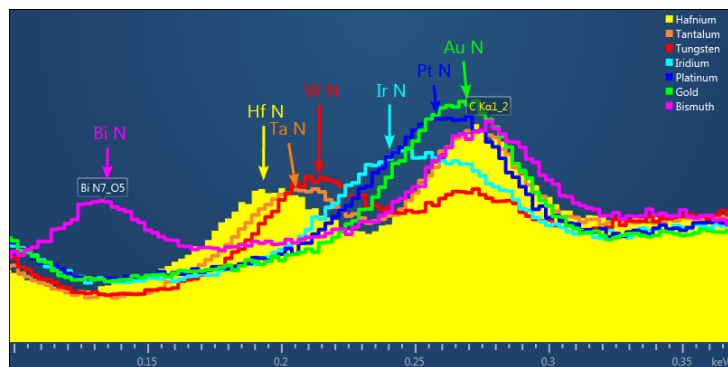


Fig. 1. EDS spectra collected with X-Max Extreme at 3kV for metals in the 6th row of the periodic table. N lines detected for Hf (195 eV), Ta (210 eV), W (215 eV), Ir (250 eV), Pt (260 eV), Au (265 eV), and Bi (135 eV). Samples were plasma cleaned for 30 minutes but show variable contribution of CK α . CK α has significant contribution to Ir N, Pt N, and Au N peaks.

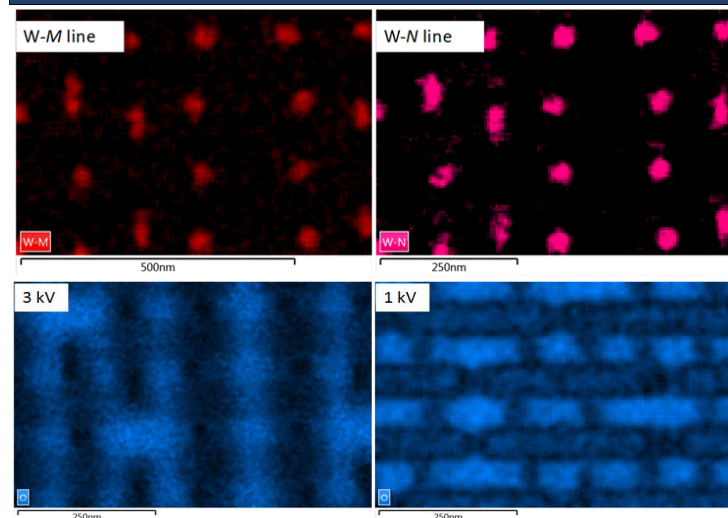


Fig. 2. Comparison of overlap-corrected (for SiK α) and background subtracted W M X-ray map with the ROI W N X-ray map for a semi-conductor processing device. Data calculated from a SmartMap spectrum image collected at 3 kV, shows both W lines reveal the same sample structures.

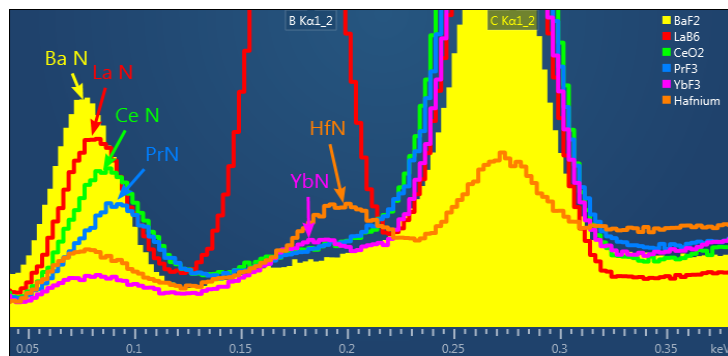


Fig. 3 Comparison of O K α ROI X-ray maps from the semi-conductor processing device collected at 3 kV and 1 kV. The 1 kV X-ray map shows improved lateral and depth resolution and resolves the structures. SmartMaps collected on FEI Magellan at Shanghai Ceramics Institute, China.

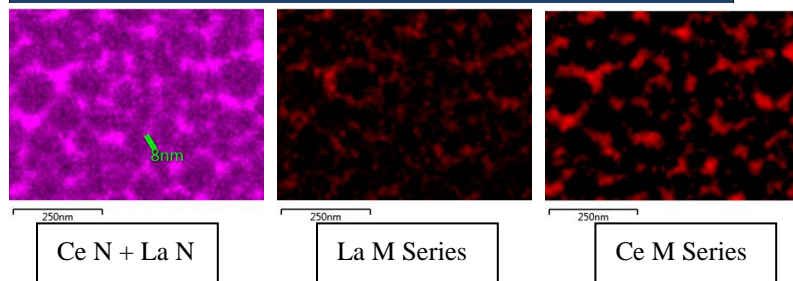


Fig. 4. EDS spectra collected with X-Max Extreme at 3 kV for barium and selected lanthanide elements. For comparison the spectrum for Hafnium shown in Fig. 1 is included. N lines detected for Ba (78 eV), La (82 eV), Ce (88 eV), Pr (93 eV), and Yb (185 eV). Intensities for Ba, La and Ce N lines are higher than for the 6th row metals. All samples were carbon coated.

Fig. 5. X-ray maps (2 kV) collected from a ceramic sample containing 5-50 nm Ce/La oxide structures. M series maps are deconvolved and background subtracted. Ce N and La N cannot be resolved so are shown as a combined ROI X-ray map. SmartMap collected on Zeiss Merlin, at Toyo Corp., Japan.