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Enhanced Theoretical Model for Avoiding Mistakes in SEM-EDS Analysis

P. J. Statham

Oxford Instruments NanoAnalysis, High Wycombe, Bucks HP12 3SE, U.K.

Under the right conditions, SEM-EDS can be fast and accurate but how can we tell if results are unrealistic? Most software provides a display to show if fitted peaks and background concur with the measured spectrum but goodness of fit of fitted peaks is insensitive to a number of factors that have a serious effect on the analytical result, for example specimen charging, surface layers, variation in composition within the excited volume and incorrect or missing elements. The oft-used normalisation simply transfers any inaccuracy in results for one element to all others.

An un-normalised analytical total that is far from 100% reveals potential problems. When results are normalised for speed and convenience, an alternative "Check Total" can still be used [1] but neither measure points to the cause of a bad total. Many years ago, Duncumb suggested display of a synthesised spectrum might be helpful [2]. The potential to realise this principle came with subsequent development of a theoretical model optimised to match measured ratios of peak intensity to total background (P/Btot) [3]. In any attempt to develop parameterised expressions, a critical factor is the calculation of detector efficiency as a function of energy. Instead of a calculation, an efficiency characteristic has now been measured for a large solid angle SDD detector [4] and data acquired from an extensive set of standard materials at both 5kV and 20kV. The original theoretical model [3] has been enhanced to incorporate differential absorption and excitation of emission lines, including the effect of Coster-Kronig transitions. The parameterisation has been adjusted to fit measured characteristic intensities and both background shape and intensity at the same beam current. The theoretical spectrum for any analysis result can now be overlaid on the original spectrum and is sufficiently accurate to provide useful diagnostics.

Fig.1 shows a spectrum of Nickel-based superalloy where the overlaid results of peak and background fitting are good. The detail in Fig.1(b) shows a small Zn K peak that has been chosen in preference to Re L. The analysis total in Table 1 is only slightly high, but the theoretical spectrum in Fig.2 shows that if Zn were present at 0.92%, there should be a Zn L peak at 1keV. When Zn and Sr are removed and replaced with Re, Fig.3 shows much better agreement with the theoretical spectrum and the analytical total in Table 2 is also improved. At low kV, some lines may not even be excited so that analysis results after normalisation can conceal gross errors. In Fig.4, a 4kV spectrum of Quartz (SiO_2) gives a result where both the fitted spectrum and theoretical spectrum line up with the measurement. The spectrum of Fig.5 has similar well-fitted element peaks to Fig.4 and gives result O: 56% Si: 44% but when these values are used to calculate the theoretical spectrum, the inconsistent peak heights warn of a serious problem. The analysis is wrong because at 4kV the Ca K peak from Wollastonite($CaSiO_3$) is not even excited so Ca cannot be measured. In such cases, immediate access to the theoretical spectrum provides additional intelligence to judge if the analysis is valid and thus avoid mistakes and bad conclusions.

References

[1] P.J.Statham (2004) Microchim. Acta 145, 229-235
[2] P.Duncumb (1994) Mikrochim. Acta 114/115, 3-20
[3] P.Duncumb, I.R.Barkshire, P.J.Statham (2001) Microsc. Microanal 7, 341-355
[4] P.J.Statham (2010) Microscopy and Microanalysis 16 (Suppl 2), 1304-1305

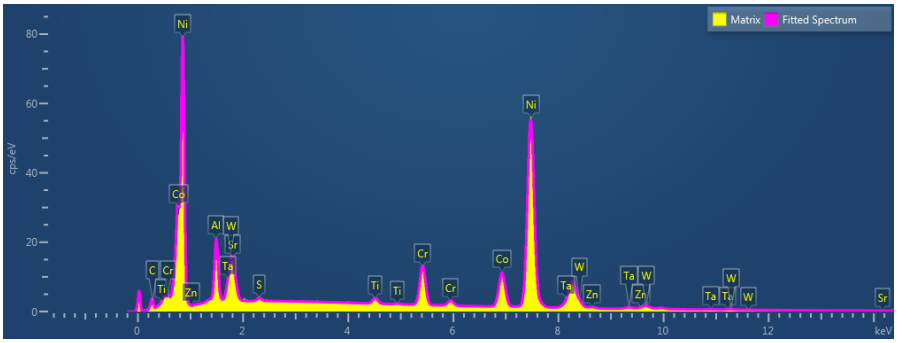

FIG. 1(a) Spectrum from Nickel superalloy (yellow bars) overlaid with result of spectrum fitting (pink).

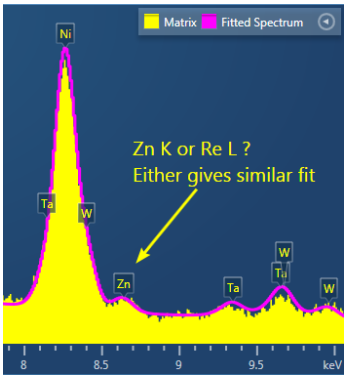

FIG. 1(b) Region showing small Zn peak

Elmt	Wt%
Al	5.08
S	0.25
Ti	0.70
Cr	6.22
Co	9.62
Ni	62.42
Zn	0.92
Sr	2.86
Ta	4.73
W	9.30
Total	102.10

Table 1: Analysis result

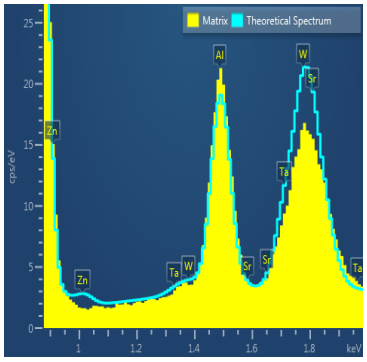

FIG. 2 Theoretical spectrum for Table 1 composition overlaid on original spectrum. Note differences near ZnL, TaM

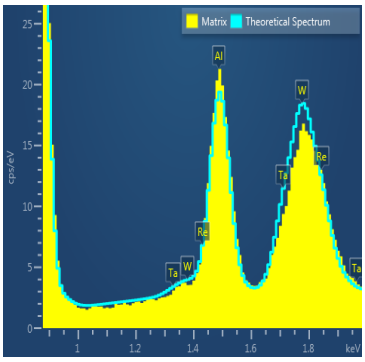

FIG. 3 Theoretical spectrum with Re instead of Zn and Sr

Elmt	Wt%
Al	5.12
S	0.23
Ti	0.7
Cr	6.22
Co	9.61
Ni	62.35
Ta	4.64
W	8.8
Re	2.37
Total	100.04

Table 2: Results used for fig.3

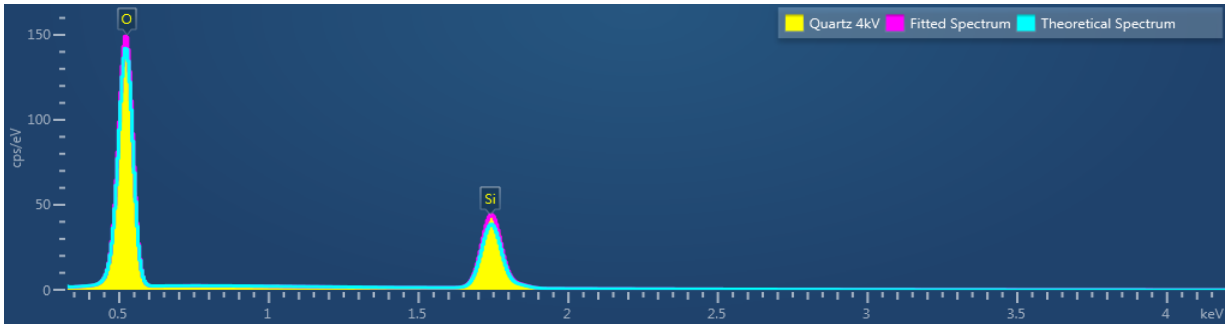

FIG. 4. SiO_2 4kV spectrum overlaid with fitted (pink) and theoretical spectrum (blue)

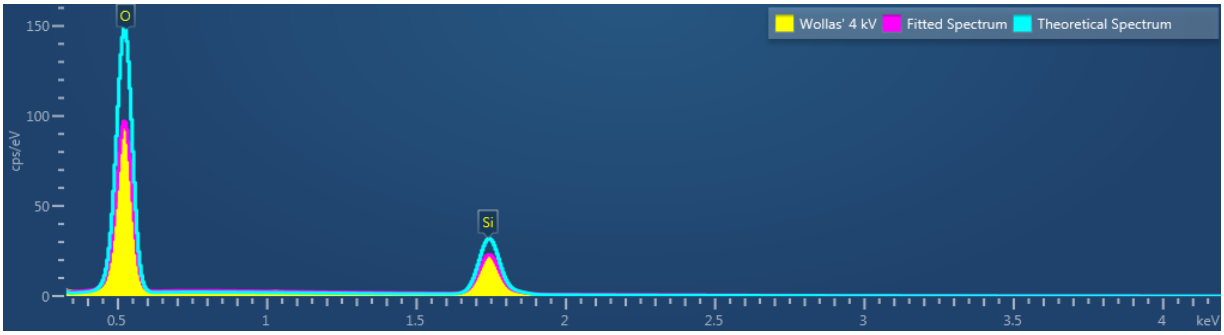

FIG. 5. $CaSiO_3$ 4kV spectrum overlaid with fitted (pink) and theoretical spectrum (blue)

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