EDS Quantification Using Fe L Peaks and Low Beam Energy

Jens Rafaelsen[1], Frank Eggert[1] and Masanobu Kawabata[2]

[1]Ametek / EDAX LLC, Mahwah, New Jersey, United States, [2]AMETEK / EDAX LLC, United States

While it has been shown that standardless quantification methods for Energy Dispersive X-Ray Spectrometry (EDS) can provide satisfactory results for a variety of samples under the right conditions [1], low beam energy quantification and quantification using L and M peaks below roughly 1.5 keV has proven to be exceptionally challenging [2]. Standards based analysis has frequently been used to compensate for shortcomings in the various quantification models, but significant deviations can occur even with standards-based quantification for low energy L-peaks [3].

In general, the number of counts N for element i in channel ch can be described as [4]:

$$N_i^{ch}=c_i\varepsilon_i It(\Omega/4\pi)\omega_i q_i S_i^{ch}(RAF)_i^{ch},$$

where c_i is the concentration of the element in question, ε is the detector efficiency at the relevant energy, I is the beam current, t is the collection time, Ω is the solid angle of detection, ω is the fluorescence yield, q is the weight of the line, and S is the primary ionizations generated, while RAF are the correction factors accounting for backscatter variations, sample self-absorption and fluorescence. For low energy peaks and low beam energy applications, the primary challenges come from correctly calculating the number of ionizations generated and applying the correct absorption correction. In most cases the solid angle and beam current are unknown and consequently most standardless results are normalized to 100%. However, by measuring a reference spectrum, it is possible to determine the $I\Omega/4\pi$ term and calculate non-normalized standardless results. This has the additional advantage that by measuring known composition sample, it is possible to calculate and apply a scaling factor to optimize the standardless model to a given set of conditions. In this case we have used a two-phase iron silicide sample (Figure 1) with pure Fe and Si as reference samples for the scaling factors or Standard Customized Coefficients (SCC) at 8, 5, and 3 kV of acceleration voltage. Example spectra can be seen in Figure 2.

Table 1 shows the quantification results using a regular normalized quantification approach as well as the non-normalized SCC approach. The normalized results show an overestimate of Si and an underestimate of Fe for both phases. When the SCC factors are applied, the Si results are seen to be much closer to the true value while Fe is significantly overestimated. Both the Si and Fe numbers are seen to decrease as the acceleration voltage is lowered, this could be caused by an increasing significance of the carbon coating as the voltage is reduced. Since both Si and Fe have been checked against pure samples where the quantification routine provides the correct composition of 100%, the ionization yield can be ruled out as the primary source of error. As the fluorescence correction is negligible in this sample, the most likely source of the deviation is the absorption correction and the associated Mass Absorption Coefficients (MACs), which is consistent with electron probe measurements on similar samples [5]. This highlights the need for more flexible models with the ability to customize and modify the MACs that are applied.

		Iron silicide 1		Iron silicide 2	
		Si	Fe	Si	Fe
Composition		**53.34**	**46.66**	**33.68**	**66.32**
Standardless analysis	8 kV	60.04	39.96	40.93	58.27
	5 kV	60.18	39.81	41.53	58.47
	3 kV	63.10	36.90	44.15	55.86
Standardless analysis with SCC	8 kV	52.75	58.07	33.33	78.03
	5 kV	50.42	57.20	31.92	77.08
	3 kV	44.53	55.84	27.93	75.66

Table 1: Quantification results using two different quantification approaches for the two distinct phases.
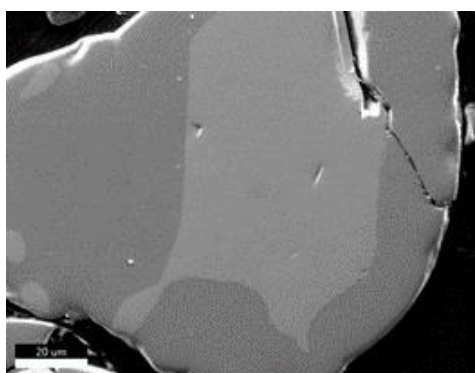

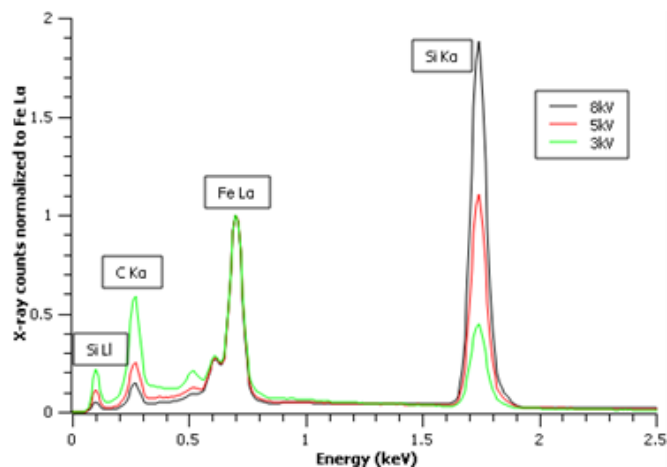**Figure 1.** Backscatter image of iron silicide sample showing two distinct phases.

Figure 2. EDS spectra showing the primary peaks at 8, 5, and 3 kV of acceleration voltage.

References

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