Microsc. Microanal. 8 (Suppl. 2), 2002
DOI 10.1017.S1431927602107835

Energy Dispersive Spectrometry Calibration For The HD-2000 STEM

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Energy Dispersive Spectrometry (EDS) systems are generally calibrated for quantified analysis by using standards that are high purity specimens of the elements measured, or without standards using theoretical calculations. While the calibration factors for many elements have been determined for applications in TEM and SEM, the factors are dependent on the setup of the microscope and the detector. These factors have yet to be determined for the Hitachi HD 2000 STEM, which has improved sensitivity due to a large collection angle. In addition, EDS is often used to determine the concentration of a trace element in a matrix. The accuracy and limit of detection of these low concentration measurements has not been established. Earlier reports proved the concept that a cross section high dose BF_2 implanted specimen could provide a standard for EDS measurement of F, [1] and that Co and Fe implants into Si calibrated the EDS signal for these metals. [2] The detection limits for these elements were also determined. This study extends the quantification approach to other elements of importance to the semiconductor industry and related fields, and determines the calibration factors, their errors and detection limits for these elements in the HD 2000 STEM.

The standards were created by high dose ion implantation. For ions implanted into silicon, a dose of $1x10^{16}$ atoms/cm^2 results in a peak concentration of approximately $1x10^{21}$ atoms/cm^3 or 2% atomic. The exact concentration can be determined using methods such as Rutherford Backscattering Spectrometry (RBS) and Secondary Ion Mass Spectrometry (SIMS). For this study, RBS dose measurements were made using a General Ionex Tandetron and SIMS depth profiles were obtained from a CAMECA IMS-6f magnetic sector instrument. Cross sectional specimens from these implanted silicon samples were prepared in a FEI 200TEM Focused Ion Beam (FIB) system and removed using the lift-out method. [3] EDS data was taken on a Hitachi HD 2000 STEM operating at 200 kV using an EDAX Pheonix Pro SiLi detector with a resolution of 130 eV.

The calibration factors were calculated using the standard Cliff-Lorimer method, comparing the X-ray signal intensity of the element of interest and that of Si, to the atomic concentration values determined by RBS and SIMS. [4] Absorption and fluorescence were ignored as the samples were ≤ 0.2 μm thick. The data was taken in line scan mode, using Net Intensity acquisition, at various dwell times, with an average of 4000 counts per second to optimize the analysis at these low concentrations. A thirty point moving average was used to smooth the data. The detection limits were determined to be the atomic concentration at which the variation in the calculated k factor exceeded one standard deviation. This data is shown in Table 1. An example of the STEM EDS data, overlaid with the SIMS profile, is shown for Ni implanted in Si in Figure 1.

References:
[1] C. B. Vartuli, et al., Microscopy and Microanalysis 2000 Proceedings, Springer (2000) 536.

[2] C. B. Vartuli, et al., Microscopy and Microanalysis 2001 Proceedings, Springer (2001) 200.
[3] L. A. Giannuzzi, et al., Materials Research Society Symposium Proceedings, 480, 19 (1997).
[4] Williams and Carter, "Transmission Electron Microscopy" Vol 3. 1996, New York. P600.

Element	Dose (cm^{-2})	k factor (for Atomic %)	Error %	Detection limit (Atomic %)
Cd (L)	2E16	2.24	5.5	0.1
Co (K)	2E16	1.40	5.2	0.25
Co (L)	1E16	0.50	10	0.7
Cu (K)	1E16	1.11	3.0	0.5
Fe (K)	1E16	1.30	7.1	0.3
Fe (L)	1E16	0.42	7.7	0.6
In (L)	4.8E15	0.91	3.0	0.2
Ni (K)	1E16	1.05	2.4	0.2
Ni (L)	1E16	0.78	6.2	0.6
Ti (K)	4.8E15	0.75	2.6	0.2

Table 1: k factors, k factor errors and detection limits for various elements.

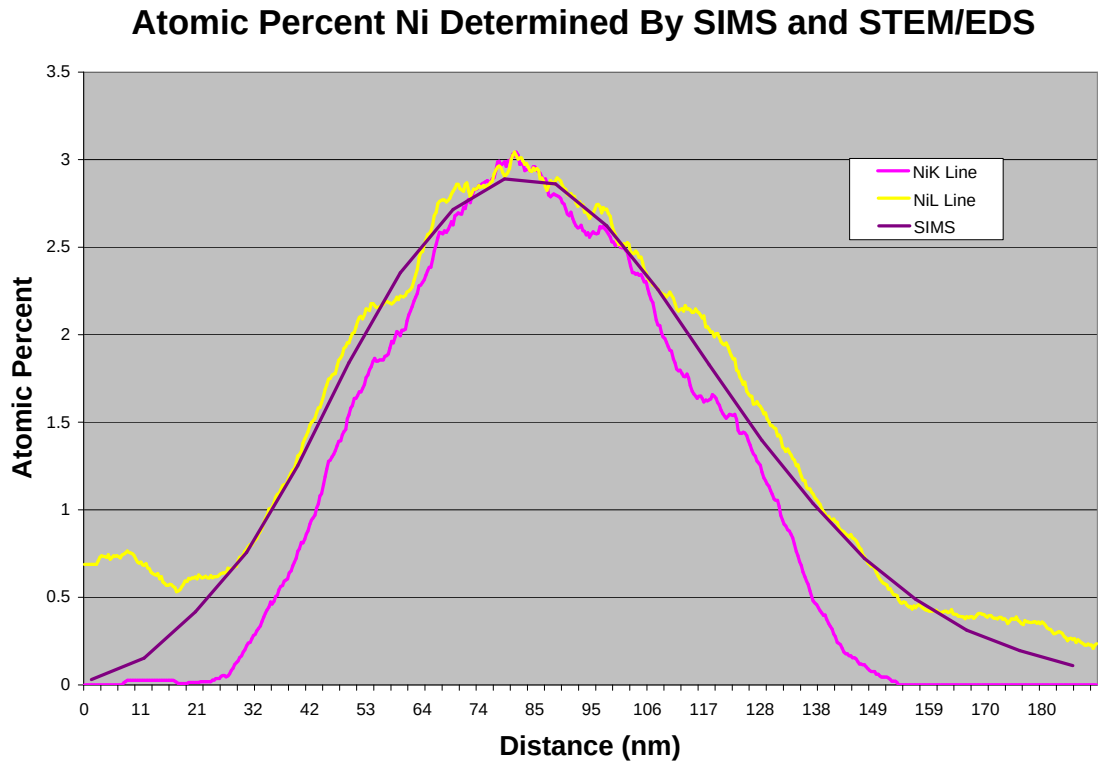

Figure 1. STEM EDS linescan and SIMS profile of 1E16 cm^{-2} Ni implanted into Si.

https://doi.org/10.1017/S1431927602107835 Published online by Cambridge University Press