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Statistical analysis of low-voltage EDS spectrum images

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The benefits of using low (≤5 kV) operating voltages for energy-dispersive X-ray spectrometry (EDS) of bulk specimens have been explored only during the last few years.[1-4] This paper couples low-voltage EDS with two other emerging areas of characterization: spectrum imaging and multivariate statistical analysis (MSA). The specimen analyzed for this study was a cross section of a computer chip manufactured by a major semiconductor company. Data acquisition was performed with a Philips XL30-FEG SEM operated at 4 kV and equipped with an Oxford super-ATW detector and XP3 pulse processor. The specimen was normal to the electron beam and the take-off angle for acquisition was 35°. The microscope was operated with a 150 μm diameter final aperture at spot size 3, which yielded an X-ray count rate of ~2000 s^{-1}. EDS spectrum images were acquired as Adobe Photoshop files with the 4pi plug-in module. (The spectrum images could also be stored as NIH Image files, but the raw data are automatically rescaled as maximum-contrast (0-255) 8-bit TIFF images – even at 16-bit resolution – which poses an inconvenience for quantitative analysis.) The 4pi plug-in module is designed for EDS X-ray mapping and allows simultaneous acquisition of maps from 48 elements plus an SEM image. The spectrum image was acquired by re-defining the energy intervals of 48 elements to form a series of contiguous 20 eV windows from 1.25 kV to 2.19 kV. A spectrum image of 450 × 344 pixels was acquired from the specimen with a sampling density of 50 nm / pixel and a dwell time of 0.25 live seconds per pixel, for a total acquisition time of ~14 h. The binary data files were imported into Mathematica for analysis with software developed by the author at Oak Ridge National Laboratory.[5] A 400 × 300 pixel section of the original image was analyzed. MSA required ~185 Mbytes of memory and ~18 h of CPU time on a 300 MHz Power Macintosh 9600.

The results of this study are shown in Fig. 1. Image (a) is a secondary electron (SE) image of the analyzed area of the device. Images (b-d) are component images I0-I2 of the statistical analysis and spectra (e,f) are the MSA spectral components E1 and E2 corresponding to images I1 and I2. The plot in (g) gives the amplitude variations along the line trace shown in (d), averaging 10 pixels (0.5 μm) in the vertical direction. Image I0 gives the average X-ray intensity levels in the spectrum image; it is the image that would be acquired with a non-dispersive detector that collected all X-rays in the ~1 kV band of energies analyzed. Images I1 and I2 are chemically sensitive MSA components. The image contrast in I1 arises mainly from spectral differences between Al and Si, as indicated by E1, and the contrast in I2 is mainly due to the W plugs, as indicated by E2. In I1, the Al lines appear dark, the Si substrate (at bottom of image) appears brightest, and the SiO_2 dielectric is a medium grey. The W plugs assume a grey level that almost matches that of the SiO_2, and are almost invisible. However, this feature is dominant in I2. Note that excellent image contrast is obtained, even though the EDS detector cannot resolve the Si-K and W-Mα lines, which are only ~34 eV apart, as shown in E2. The next generation of EDS detectors will be able to resolve these X-ray lines,[6] which at the present ~100 eV resolution give rise to the "first derivative" feature in E2. A line trace (vertical average of 10 pixels) across the 600-nm-wide W plugs shown in (g) illustrates the excellent spatial resolution of the EDS spectrum image. The vertical averaging improves the noise at the expense of resolution. The resolution of individual line traces, defined as 90% of the total change in intensity, is ~4 pixels or ~200 nm.[7]

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