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APPLICATIONS OF MULTIVARIATE STATISTICAL ANALYSIS (MSA) IN MICROANALYSIS

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Recent improvements in computer hardware and software for the acquisition, storage and analysis of series of spectra and images allow for a change in strategy for quantitative microanalysis. For example, in the area of X-ray microanalysis, whereas compositional analysis and elemental distributions have been traditionally performed using point microanalyses and simple intensity mapping from a ROI, respectively, the two tasks are now routinely performed simultaneously through X-ray spectrum-imaging, where full spectra are acquired from pixels in a two-dimensional array of points on the specimen. Commercially available software now allows for the acquisition and storage of such spectrum-images, perhaps comprising as much as 100 MBytes of data or more.[1] A variety of post-acquisition processing tools are provided by the developer to allow the extraction of both X-ray intensity maps, with or without rudimentary background subtraction, or full spectra from pixels of interest. In order to maximize the extraction of information from these large data sets, a number of linear and nonlinear methods are currently being explored that identify statistically significant variations among the series of spectra without *a priori* assumptions about the content of the data set.[2] Among these methods, linear multivariate statistical analysis (MSA) has a number of significant advantages, including its comprehensiveness, since all spectral variations distinct from the Poisson noise level are identified, and its broad applicability to a variety of microanalytical techniques.[3,4] MSA also preserves the integrity of the raw data, since the results of the analysis are not contingent upon input from the analyst and there is a one-to-one correspondence between the raw data and the MSA results.

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An application of MSA to a low-voltage EDS spectrum image is shown in Figure 1. A preliminary analysis has been performed on this semiconductor chip specimen, where images were acquired using only a portion (1.2 – 2.2 keV) of the X-ray spectrum.[5] Data acquisition was performed with a Philips XL30/FEG SEM equipped with an Oxford super-ATW detector and XP3 pulse processor, and an EMiSPEC Vision integrated acquisition system. The XL30 was operated at 4 kV with a 30 μm final aperture. EDS spectra were acquired with a 35° takeoff angle at ~1500 input counts per second and ~20% dead time. The 200 × 150 pixel spectrum image was acquired with 20 nm per pixel, 10 eV per channel, and a 250 ms dwell. MSA was performed on a 200 channel selection (0.12 – 2.11 keV) of the spectrum image that includes all relevant characteristic X-ray peaks. A normalized eigenvalue plot of the first 50 MSA components is shown in Fig. 1a. The linear variation of the higher order (≥10) eigenvalues on the semilog plot is consistent with the variations of these spectral components arising solely from Poisson statistics. The first nine eigenvalues have an information value that is distinct from the Poission noise limit. Fig. 1 shows MSA component spectra (b-d) and images (e-g) corresponding to the first three eigenvalues. Bright (dark) features in the component images correspond to strongly positive (negative) characteristic peaks in the corresponding spectra. For example, the brightest areas in Fig. 1f come from the SiO_2 dielectric (corresponding to the strong O-K peak at ~0.5 keV), the darkest areas come from the Si substrate and the W plugs (with negative spectral features between ~1.6 and ~2.0 keV), and an intermediate grey level is displayed by the Al lines (weakly positive peak at ~1.5 keV). There is excellent discrimination between the Si substrate and the W plugs in Fig. 1g, despite the unresolved W-M and Si-K X-rays (separated by ~35 eV), which give rise to the first-derivitive-type feature in Fig. 1d. Also evident in Fig. 1d is the higher continuum X-ray intensity excited in the high-Z W plugs.[6]

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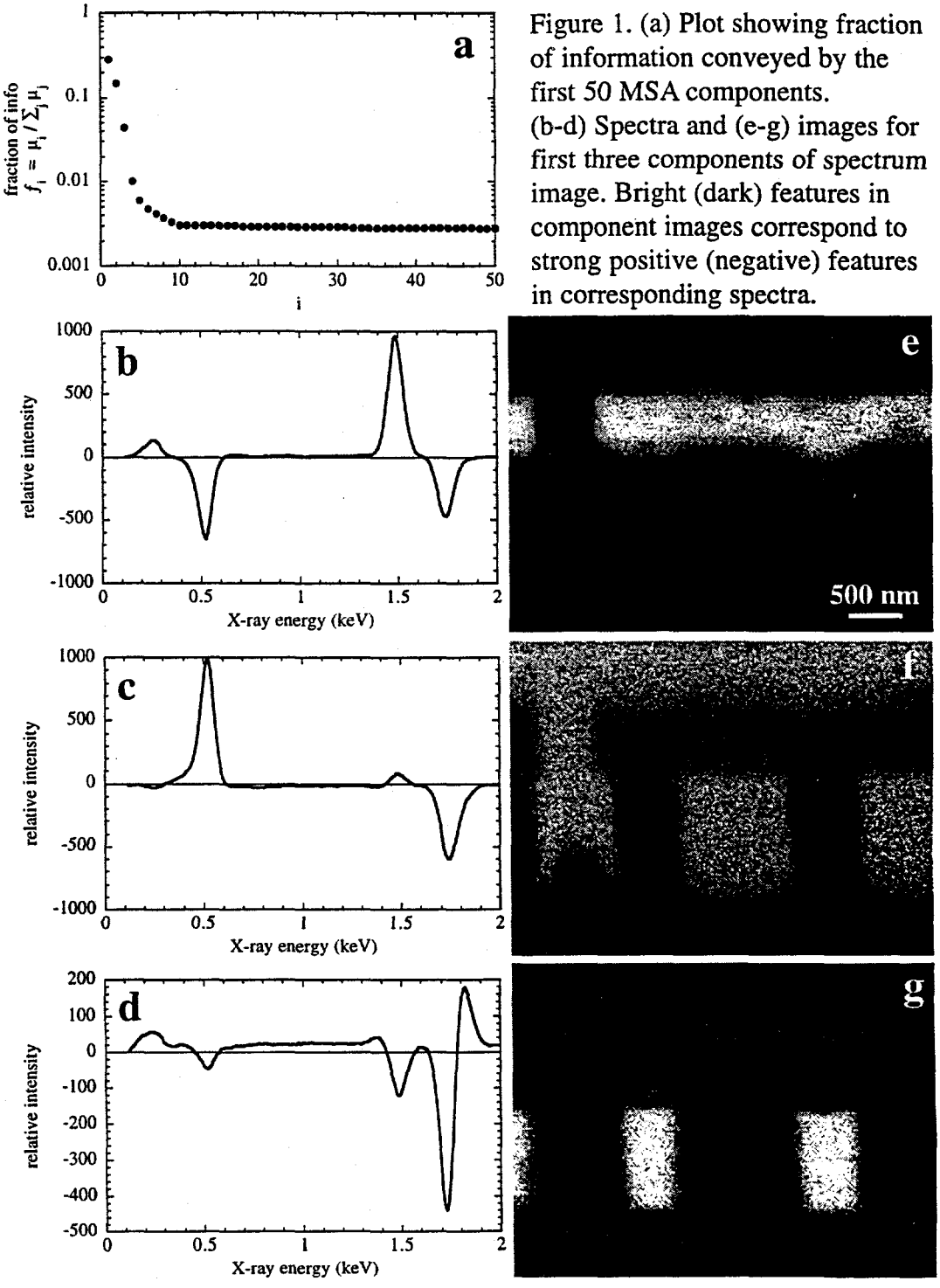

Figure 1. (a) Plot showing fraction of information conveyed by the first 50 MSA components. (b-d) Spectra and (e-g) images for first three components of spectrum image. Bright (dark) features in component images correspond to strong positive (negative) features in corresponding spectra.

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