

SPECTRUM IMAGING: MICROANALYSIS FOR A NEW MILLENNIUM

I.M. Anderson

Metals & Ceramics Division, Oak Ridge National Laboratory, MS-6376, PO Box 2008, Oak Ridge, TN 37831-6376

Spectrum imaging, where a full spectrum is acquired at each pixel in a two-dimensional array,¹ provides a new paradigm for materials characterization that combines the advantages of traditional paradigms for imaging and analysis in electron optical characterization. Traditionally, electron optical imaging (e.g., secondary electron imaging of electron-opaque or bright-field imaging of electron-transparent specimens) emphasizes contrast production among distinct features of the microstructure. An excellent survey of the specimen microstructure is achieved – many pixels are acquired – but typically no quantitative analysis of image intensities is performed. In contrast, electron optical analysis techniques traditionally concentrate on quantitative elemental analysis (e.g., electron probe microanalysis), or at least comprehensive qualitative analysis through full spectrum acquisition (e.g., energy-dispersive X-ray or electron energy-loss spectrometry). However, these analyses are typically performed at only a few locations on the specimen, which are pre-selected on the basis of the image contrast generated as described above. Spectrum imaging combines the excellent survey of the microstructure traditionally associated with imaging with the full spectrum acquisition that is typical of microanalysis. With the pixel spacing suitably matched to the resolution of the microanalytical technique, spectrum imaging can provide a comprehensive characterization, so that all spectrally distinct features of the specimen microstructure are surveyed. Oversamplings of between 1 and 2 are common as these ensure sampling of all phases in the analyzed area while allowing the greatest possible sampling of the microstructure. Spectrum imaging can be performed by acquiring series of either spectra (e.g., typical of X-ray mapping techniques) or images (e.g., series of energy-filtered transmission electron microscopy images), and is applicable to any spectroscopic technique.

One factor that has limited the practicality of spectrum imaging is the large size of the raw data files that are generated with this method. Whereas a typical spectrum (2000 channels, 2 bytes per channel) comprises a few kbytes and a typical digital image (1000 × 1000 pixels, 1 byte per pixel) a Mbyte, the corresponding spectrum image (1000 × 1000 pixels × 2000 channels, 2 bytes per pixel) would comprise a few Gbytes of data. Although ongoing advances in computing power will soon render the manipulation of Gbyte-scale data files practical, the storage, retrieval and analysis of such data files is at present cumbersome, and currently spectrum images are limited to fewer pixels and/or channels. However, even for modest spectrum images (a few tens of Mbyte), “data mining” methods are required to extract the relevant specimen-related information, and a variety of strategies for spectrum image analysis are being pursued.² Useful distinctions among analytical methods can be made between: robust methods that analyze the data in a comprehensive fashion, but may be slow and memory intensive, and faster routines that may exhibit artifacts or overlook minor features of the microstructure; methods that interrogate the spectrum as a whole, as opposed to user-defined spectral features, such as regions-of-interest (ROIs); and methods requiring iterative operator intervention as opposed to automated analysis.

An illustration of issues surrounding spectrum image acquisition and analysis is shown in Fig. 1. 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, and an EMI-SPEC integrated acquisition system. The specimen was normal to the electron beam and the take-off angle was 35°. The microscope was operated with a 30 μm diameter final aperture at spot size 6, which yielded an X-ray count rate of ~1500 s⁻¹. A spectrum image of 200 × 150 pixels was acquired at 100 nm per pixel and 10 eV per channel, and a dwell time of 1 s per pixel, for a total (overnight) acquisition time of ~8.25 h. The resolution of the EDS spectrum imaging had been previously determined as ~160 nm for these acquisition parameters and this specimen.³ A backscattered electron (BSE) image of the analyzed area of the specimen is shown in Fig. 1a. The bright features in the BSE image are the tungsten interconnects, which have a much higher average atomic number than any other feature in the microstructure. Partial representations of the corresponding spectrum image using three different analysis and display methods are shown in Fig 1b-d. Some specimen drift is evident in these images, especially early in the acquisition, from an apparent rightward leaning of the tungsten interconnects in the X-ray images that is absent in the BSE image. Drift correction is an important consideration for spectrum imaging, and is available in commercial spectrum imaging packages.⁴ A conventional X-ray map, formed with a ROI centered on the Co-L X-ray lines, is shown in Fig. 1b. Whereas the cobalt silicide layers are highlighted in this image, considerable contrast is evident from other microstructural features because of the atomic number dependence of the X-ray continuum. The continuum-based contrast is similar to that of the BSE image in Fig. 1a, and the tungsten interconnects are especially prominent in Fig. 1b. This continuum contribution to the contrast is common to all ROI-based X-ray maps.⁵ Fig. 1c shows a component image arising from linear multivariate statistical analysis (MSA) of the spectrum image. MSA is a robust analysis technique, requiring no a priori input from the analyst, is suitable for automation, but is relatively time consuming and memory intensive.^{2-3,6} The image in Fig. 1c corresponds to the second significant component, which is weighted positively on the

O-K peak and negatively on the Si-K/W-M peaks. Accordingly, the silicon dioxide dielectric is bright, the silicon substrate and tungsten interconnects dark, and the remaining phases (including the region at the top where the beam does not intercept the specimen) are an intermediate gray level. An alternative analysis method, based on multiple linear least squares (MLLSQ) fitting, is illustrated in Fig. 1d. Here, the spectrum in each pixel is subjected to a MLLSQ fitting of spectra acquired by the analyst from the specimen. The six basis spectra for the MLLSQ fit were acquired from the silicon, silicon dioxide, aluminum, tungsten, cobalt silicide and nitride phases; these were the distinct phases previously identified with MSA.³ The image in Fig. 1d corresponds to the silicon dioxide basis spectrum. The silicon dioxide phase is white, and all other phases black, to within the noise level of the data. MLLSQ fitting is one possible method for extracting the “pure” components from a spectrum image.⁷

1. C. Jeanguillaume and C. Colliex, *Ultramicrosc.* 28(1989)252.
2. N. Bonnet, *J. Microsc.* 190(1998)2.
3. I.M. Anderson, *Microsc. Microanal.* 5 [Suppl. 2: Proceedings] (1999)318.
4. <http://www.emispec.com>
5. D.E. Newbury and D.S. Bright, *Microsc. Microanal.* 4 [Suppl. 2: Proceedings] (1998)160.
6. P.G. Kotula et al., *Microsc. Microanal.* 5 [Suppl. 2: Proceedings] (1999)806; also these proceedings (2000).
7. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

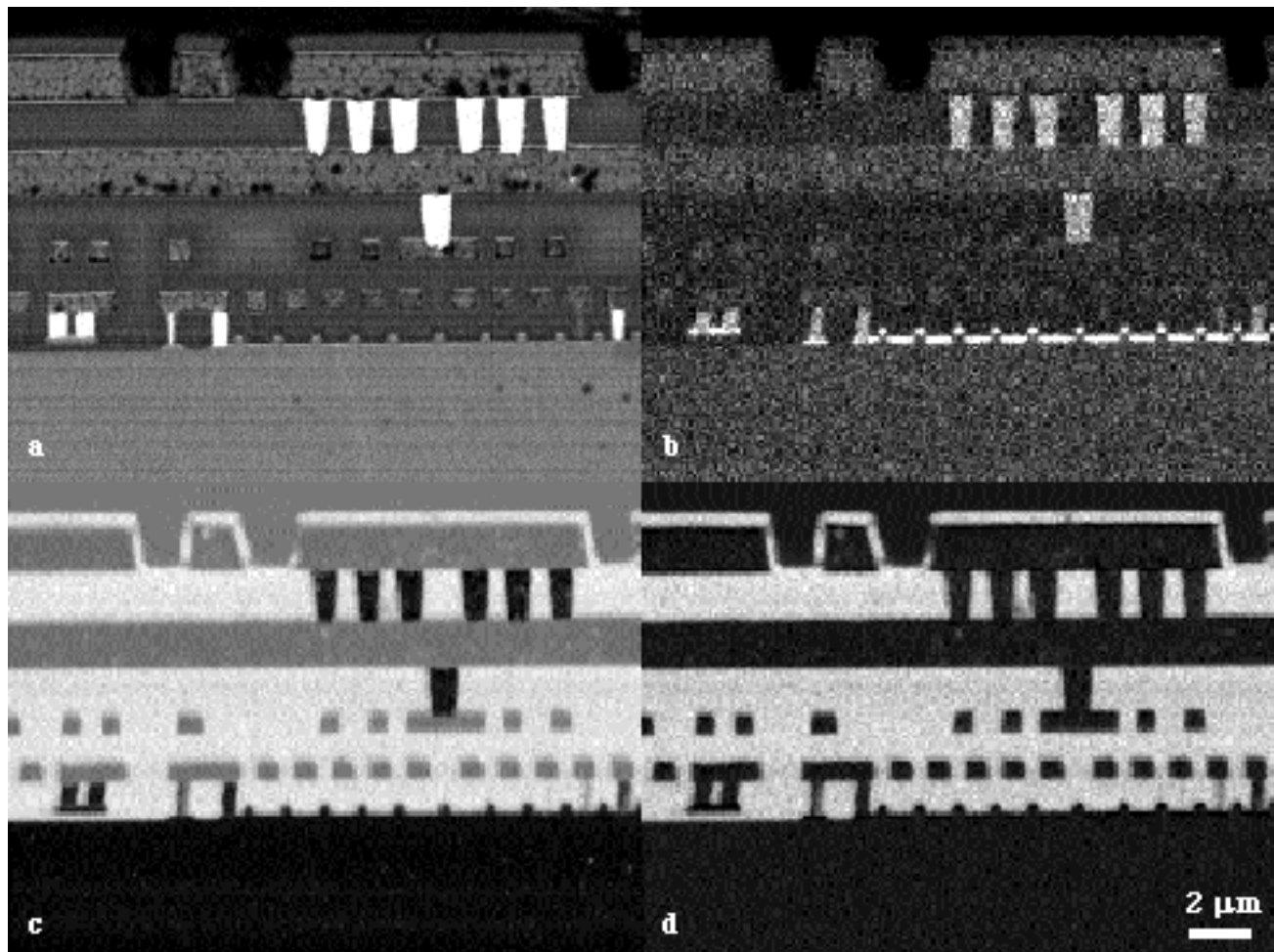


FIG. 1 – Images acquired at 4 kV of cross-sectioned computer chip: (a) BSE image; (b) X-ray map formed with ROI centered on Co-L; (c) MSA second component image (O-K); (d) MLLSQ image for silicon dioxide spectrum.