X-RAY MICROANALYSIS IN THE ENVIRONMENTAL SEM USING MAPPING AND FOURIER DECONVOLUTION TECHNIQUES

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X-ray microanalysis of any type of specimen in its natural state without the use of conventional SEM specimen preparation techniques has immense potential in a wide range of scientific and industrial applications. This capability would be particularly useful in microanalysis applications where it is highly desirable to preserve the integrity of the specimen, for example in semiconductor failure analysis and forensic investigations. In principle, this X-ray microanalysis goal can be achieved in an environmental or variable pressure scanning electron microscope (VPSEM) because specimen charging and vacuum stability problems are negated by the presence of a gas in the specimen chamber. However, the accuracy and spatial resolution of X-ray microanalysis in the VPSEM is significantly degraded by the chamber gas as it scatters primary beam electrons, generating spurious X-rays far from the analysis point.[1,2] To date, two different X-ray measurement strategies have been developed to facilitate X-ray microanalysis at high chamber pressure in the VPSEM. In the first approach, X-ray spectra measured at two different chamber pressures are subtracted to yield the X-ray peak intensities free from the spurious X-ray contribution.[3] The second method[4,5] utilizes a fine Beryllium wire beam stop to measure the X-ray spectrum excited by the scattered electrons. The high vacuum spectrum is then obtained by subtracting the beam stop X-ray spectrum from high pressure X-ray spectrum. The aim of the present work is to develop a simple yet rigorous method to conduct high spatial resolution quantitative X-ray microanalysis in the VPSEM under all VPSEM operating conditions.

An X-ray map collected in the VPSEM at high chamber pressure, P_C, is a convolution of the high vacuum X-ray map with the electron probe profile (EPP) at pressure, P_C, that is:

$$\mathrm{Map}(P_C) = \mathrm{EPP}(P_C) \otimes \mathrm{Map}(P=0)$$

Therefore, Fourier deconvolution techniques can be used to extract the high vacuum X-ray map from an X-ray map measured at pressure, P_C, as follows:

$$\mathrm{Map}(P=0) = \mathfrak{I}^{-1}\{\, \mathfrak{I}[\mathrm{Map}(P_C)] / \mathfrak{I}[\mathrm{EPP}(P_C)]\}$$

where $\mathfrak{I}[\mathrm{Map}(P_C)]$ is the Fourier transform of the X-ray map at pressure, P_C and $\mathfrak{I}[\mathrm{EPP}(P_C)]$ is the Fourier transform of the electron probe profile at pressure, P_C.

The availability of a reliable model for the electron probe profile as a function of VPSEM operating parameters is pivotal to the viability (and accuracy) of the presented technique. Currently, there is little experimental agreement on the shape of the scattered electron profile.[6] However, all probe profile measurements to date have overlooked gas phase - electron scattering above the pressure limiting aperture (PLA) assembly, which separates the high vacuum and high pressure regions of the VPSEM. Recent studies[7,8] have revealed that the scattered electron profile at the specimen plane can be significantly affected by electron - gas phase scattering above the PLA. There is little doubt that a universal model for the electron probe profile is imminent now that this additional contribution to the electron beam - gas phase scattering process has been recognized. Fourier analysis and X-ray mapping techniques can be used to verify the general shape of the theoretical scattered electron distribution by using:

$$EPP(P_C) = \mathfrak{I}^{-1}\{ \mathfrak{I}[Map(P_C)]/ \mathfrak{I}[Map(P=0)]\}$$

However, truncation errors in the Fourier transform eliminate this technique as a practical method to accurately measure the scattered electron profile.

A number of test specimens consisting of periodic micron-sized Aluminum tracks, dots and squares on a Silicon substrate have been prepared using standard semiconductor fabrication techniques. These specimens have been used to (i) evaluate the performance of the presented technique and (ii) determine the range of VPSEM operating parameters for reliable analysis. All X-ray maps were collected on a Philips XL30 ESEM equipped with an EDAX Phoenix EDS X-ray analyzer and a STW Si(Li) X-ray detector. The electron probe profiles used in the Fourier transform deconvolution calculations were measured using the PN junction in cross-sectioned solid state diode.[9]

References:

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