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STEM-IN-SEM FOR MEDIUM-RESOLUTION X-RAY MICROANALYSIS

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Efforts to improve the spatial resolution of microanalysis in the SEM have focused on two strategies: Using low primary beam energies to decrease the interaction volume for generation of x-rays [1]; and using a thin sample and higher primary beam energies (e.g., 30keV, so called STEM-in-SEM) [1]. Both methods have advantages and disadvantages. Low-voltage microanalysis while well suited to bulk, typically polished surfaces, has a distinct disadvantage in that the lower in primary beam energy we go, the fewer x-ray lines we have available and the more likely that those we do detect will be pathologically overlapped with lines from other elements. A good example of the low-voltage SEM approach is in the 3D x-ray microanalysis of materials via serial sectioning in the FIB-SEM [2]. In this case making separate thin sections is impractical and therefore low-voltage microanalysis coupled with multivariate statistical analysis to separate the peak overlaps was the best path forward [2,3]. STEM in SEM gives us the choice of numerous x-ray lines but requires that we have a thin specimen which therefore results in fewer x-rays from the smaller volume of material excited. There is a class of materials analysis problems however where we have a thin specimen but would like a simpler (albeit lower resolution) way of characterizing the elemental distributions than in a high-voltage STEM (e.g., 300kV). This is accomplished by using a SEM operating typically at 30kV and looking at conventionally prepared TEM specimens. In this work, an example of STEM in SEM microanalysis will be presented where we have used our annular 1 steradian solid-angle Si-drift detector [4] to improve the efficiency for x-ray collection from thin samples.

A STEM in SEM x-ray spectral image was acquired with a Zeiss Supra 55VP operating at 30kV and equipped with a Bruker AXS Model 4060 annular geometry Si-drift detector [4] from a polycrystalline Si micro-electromechanical device which had been coated with a thin layer of Al-oxide. The FIB specimen was approximately 100 nm thick. An x-ray spectral image consisting of 512 by 384 pixels each with 2048 channels was acquired at a microscope magnification of 30kX resulting in a pixel size of 7.4nm in just over two minutes with a per pixel total dwell time of 640 μsec. The average count-rate from the specimen was 270 kcps at a deadtime of 20% which results in approximately 130 counts per spectrum in the spectral image. Such a count rate acquired from a thin specimen is due for the most part to the large solid angle of the detector. Figure 1 shows the results of the multivariate statistical analysis (MSA) via the methods described in [2,3]. A ~25nm layer of Al-oxide is found coated on all original surfaces of the device. Figure 2 shows the respective spectral shapes for the component image overlay in Fig. 1. Measurement of the same specimen in the 300 kV STEM (not shown here) showed approximately the same thickness Al-oxide layer. In this case, for two relatively low-Z materials in contact, it seems that there is relatively little effect from beam spreading in the specimen and x-

ray generation away from the beam. In contrast, for combinations of low- and high-Z materials, and in particular high-Z-high-Z layers, the profiles broaden out due to significant interaction of the relatively low energy beam with the sample. Even in these extreme cases however, practical resolutions of 30-60nm are achievable with nominal thickness specimens of 50-100nm..

[1] J.I. Goldstein et al., Scanning Electron Microscopy and X-Ray Microanalysis, 3rd Ed. Klewer Academic/Plenum Publishers, New York 2003.
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[3] P.G. Kotula, M.R. Keenan and J.R. Michael. *Microsc. Microanal.* (2003) **9** 1-17.
[4] P.G. Kotula et al., *Microsc. Microanal.* (2008) **14** 116CD.
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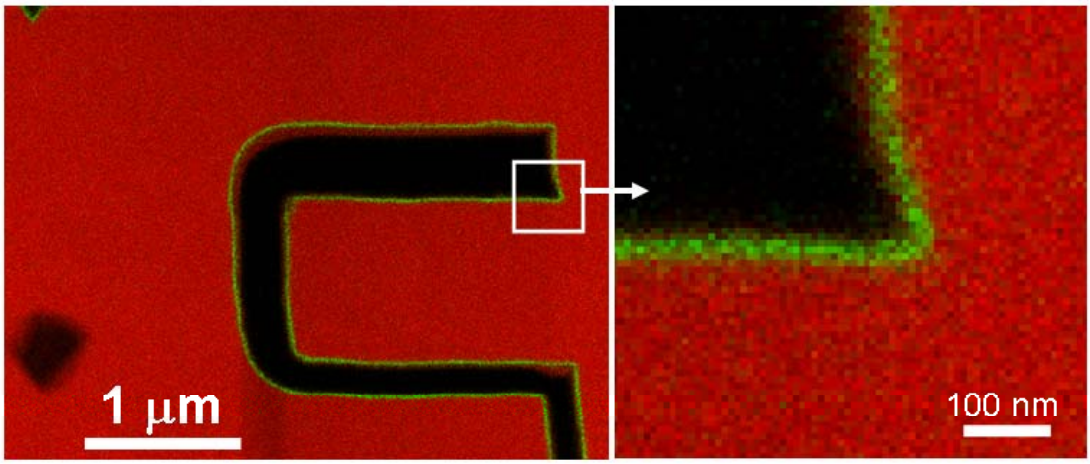

Figure 1. Partial MSA results from STEM in SEM with annular Si-drift detector EDS, spectral image. Red is Si and green is Al-oxide. The Al-oxide layer is about 3.5 pixels wide or ~25nm thick as seen in the magnified area at right.

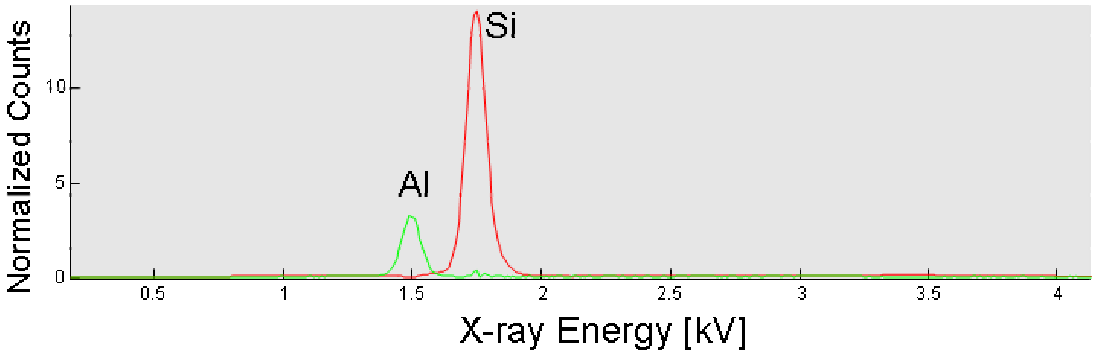

Figure 2. Spectral shapes for the components shown in Fig. 1. Note Oxygen is not visible with this detector due to the presence of an 11µm Be window.

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