Comparison of Secondary Electron Energy Filtering Techniques in Scanning Electron and Ion Beam Microscopy

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Imaging an energy filtered Secondary Electron (SE) signal can reveal more information than observing the entire spectrum of detectable SEs at once, as is the case for a standard SE image. It has been shown that energy filtering of the SE signal can highlight nanoscale resolution chemical contrast in photovoltaic polymers [1,2], go some way into quantifying dopant contrast in semiconductors [3], and has been used to bypass contamination layers, enhancing contrast [4]. In SEM, SE energy filtered imaging techniques have been demonstrated predominantly using FEI Sirion columns [1-4]. They are also seen in scanning He ion microscopes (HeIM) where contrast enhancement and reversal can be used to differentiate very thin surface layers from their substrates [5]. While this research is useful, the ability to apply this technique in just a select few systems has limited its use.

Energy filters used range from fixed system solutions utilising existing and standard in-column optics to a portable SE detector, used in both SEM and HeIM, capable of isolating SE1 and SE3 signals from higher energy SE2s. Fixed, system filters include: deflector electrodes in the FEI Sirion column, energy reflecting mirror grids in the JEOL 7800f and FEI El-star columns for SEM, and a retractable filter grid mounted on a probe within the chamber of a Ziess Orion HeIM.

In this research, SE energy filtered imaging has been applied across FEGSEM/FIB platforms and HeIM to assess its transferability between microscopes with different primary beam types, columns, detectors, and energy filter configurations. In each system, images series of three reference samples were taken; gold, HOPG (Highly Ordered Pyrolytic Graphite), and ruthenium. These samples were chosen as they have very different theoretical SE spectra with SE peak energies ranging from 2.5 (HOPG) - 11eV (ruthenium) [6]. Spectra were acquired by measuring the rate of change in the grey scale of images with respect to the change in energy filter voltage. Differences in these spectra were then used to compare the system's ability to resolve between materials' SE energies. The technique has subsequently then been applied to some metallic alloys for phase differentiation e.g. powerplant steel for the analysis of carbides within the structure.

Only a select few SEMs and the HeIM are capable of providing enough energy resolution between the SE peaks for the SE energy filtered imaging technique to be used. The JEOL 7800f can produce spectra, showing separate peaks of Au and Ru with 95% confidence, but not between Au and HOPG; elements with SE energy peak values closer together. However, other systems such as the Ziess Orion have shown much higher energy resolutions with significant differences between the peaks (Figure 2), suggesting these systems are capable of SE energy filtered imaging.

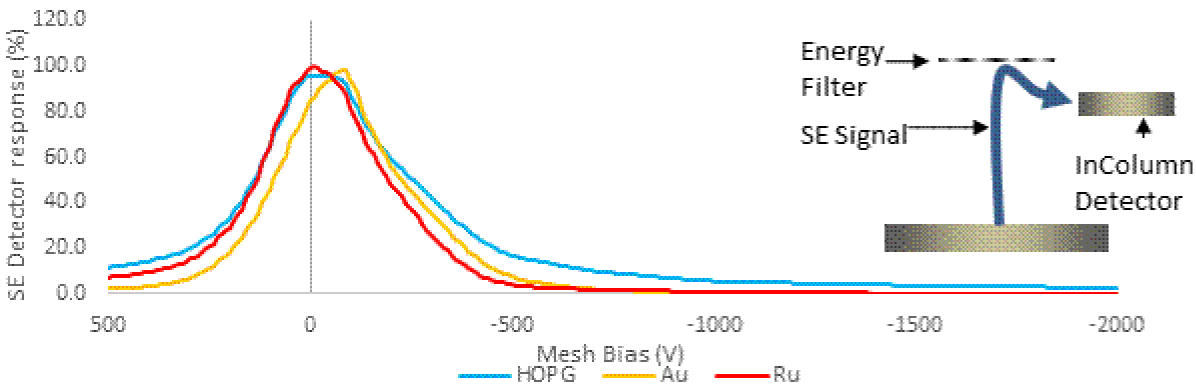

Figure 1. *SE spectra of the three reference samples collected in the JEOL 7800f and schematic of the filtration method. Raw data presented from original images.*

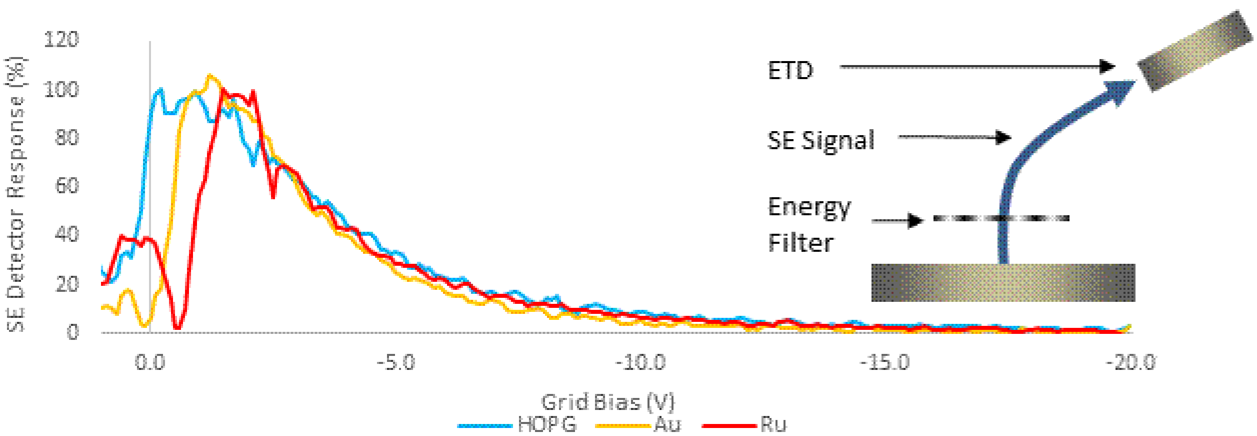

Figure 2. *SE Spectra of the three reference samples collected in the Ziess Orion Nanofab and schematic of the filtration method. Data presented has undergone a differential average smoothing with a window of 5 points.*

References

[1] Masters RC et al. Sub-nanometre resolution imaging of polymer - fullerene photovoltaic blends using energy-filtered scanning electron microscopy. Nature Communications. 2015;6:6928.

[2] Kumar V, et al. Nanoscale Mapping of Bromide Segregation on the Cross Sections of Complex Hybrid Perovskite Photovoltaic Films Using Secondary Electron Hyperspectral Imaging in a Scanning Electron Microscope. ACS Omega. 2017;2(5):2126 - 33.

[3] Kazemian P, et al. Quantitative secondary electron energy filtering in a scanning electron microscope and its applications. Ultramicroscopy. 2007;107:140 - 50.

[4] Rodenburg C, et al. Energy selective scanning electron microscopy to reduce the effect of contamination layers on scanning electron microscope dopant mapping. Ultramicroscopy. 2010;110(9):1185 - 91.

[5] Mikhailovskii VY, Petrov Y V, Vyvenko OF. Energy Filtration of Secondary and Backscattered Electrons by the Method of the Retarding Potential in Scanning Electron and Ion Microscopy. Journal of Surface Investigation. 2015;9(1):196 - 202.

[7] Joy DC, Prasad MS, Meyer HM. Experimental secondary electron spectra under SEM conditions. Journal of Microscopy. 2004;215(1):77 - 85.

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