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Low Voltage Soft X-ray Emission Analysis from 100 V for Depth Chemical Information from a few nm to several hundred nm

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Electron microbeam analysis with Soft X-ray Emission Spectroscopy (SXES) has been demonstrated as a valuable technique for several kinds of applications such as lithium battery analysis, trace element analysis in steel, and chemical state analysis. The resulting high-resolution energy spectra are useful for observing soft X-rays produced from the electron transitions from valence bands to core shell holes which provide various K, L, M and N emission spectra for elements from lithium to uranium [1,2].

Recent advances in ultra-low voltage electron microscopy using secondary electrons have been shown to be very useful for imaging. Specifically, soft landing electrons produced by retarding the primary accelerating voltage with a stage bias has proven to be very useful for observing fine surface structures using only 10V of actual landing energy [3,4]. In order to generate the soft X-ray emission, an accelerating voltage of just over the critical excitation voltage of the X-ray emission line of interest is required. This optimum overvoltage is very low energy, often as low as 100 V. Moreover, the retarding mode is effective at producing probe currents of 100 nA or more, at these very low accelerating voltages. Using this strategy, several low voltage analyses were attempted to illustrate the application of combining an ultra-low voltage analysis with the SXES spectrometer.

First, oxidized beryllium was analyzed at various low voltages. These different voltages provide depth information due to differences in the penetration depth of the primary beam electrons. After a surface cleaning with Ar ions at 5kV for 30 min, a range of low voltages, 200, 400, 600 and 800 V, were applied to detect the Be-K emission spectra. The results were shown in figure 1. The main peak near 100 eV indicates that a chemical shift occurs between the 200V measurement and those at higher voltages. In addition, a sub peak by the molecular orbital bonding is observed at 90 eV when the lowest accelerating voltage is used. According to Monte Carlo simulation, the penetration depth of the electron beam is only 2nm when using a 200V accelerating voltage. Similar depth calculations were made for the various Be-K emission spectra collected at the various accelerating voltages as shown in Fig. 2. Moreover, doped boron in a silicon device was analyzed for its depth distribution. For quantification, the area offline correction method and area intensity ratio to pure standard was applied.

In this presentation, we will discuss possibilities of this WD-SXES for depth profiling, chemical state analyses and quantitative analyses.

References:

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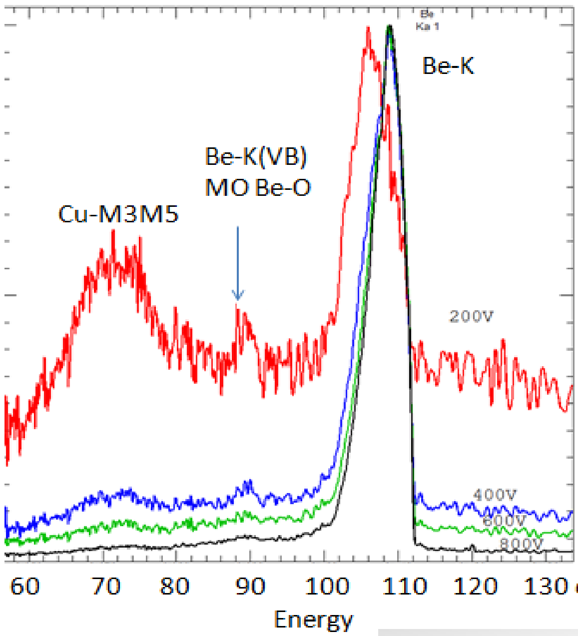
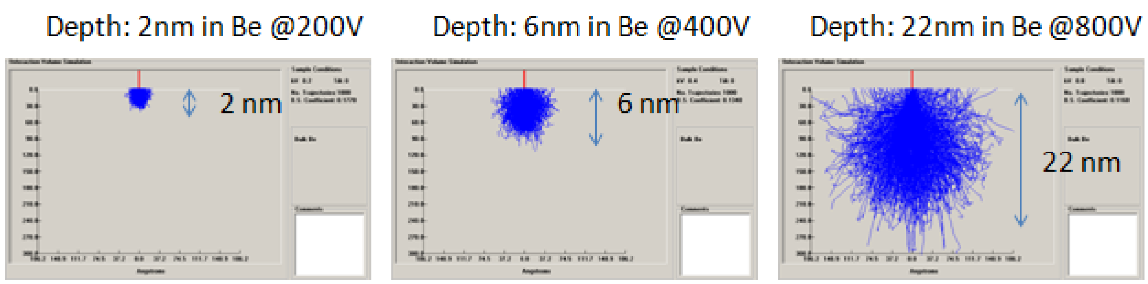


Figure 1. Be-K emission spectra obtained at 200V, 400V, 600V and 800V.



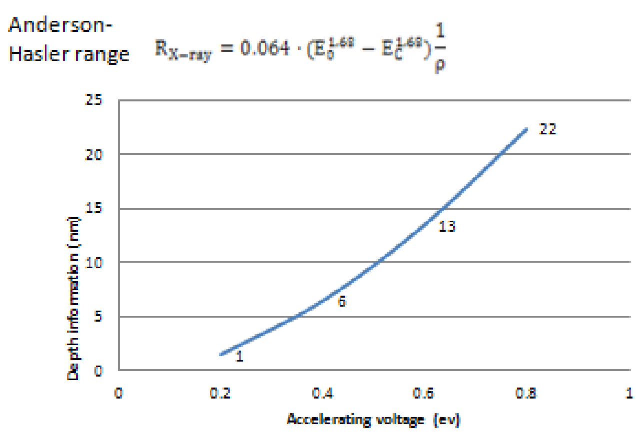


Figure 2. Monte Carlo simulation for penetration depth of electrons in beryllium at 200 V, 400 V and 800 V and calculated depth information at 200, 400, 600 and 800V.