EDS Setup Calculations **Advanced Microscopy Facility**

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Prior to doing any EDS work on the Hitachi S-4800 SEM, it is recommended to study the following document, in order to get the most out of the instrument.

Before starting, read the following:

- "Let's familiarize ourselves with the SEM!" This book was provided to you during the SEM workshop. Specifically, pages 63-72 are relevant to EDS.
- Microscopy and Microanalysis, volume 12, pages 2-25 (2006), DOI: 10.1017/S1431927606060211

It is important to be familiar with the limitations of EDS in SEM. The key is that the resolution limit of EDS is not the same as the resolution of the SEM, and that it depends on both the specimen and the instrument settings. The biggest barrier to high resolution results is the large interaction volume of the bulk specimen. Thin film EDS analysis has a much higher resolution, and can be done in both TEM and SEM, but you need a specially prepared specimen.

With EDS, you need to know what you're looking for, to find it with any decent resolution. You may need to repeat the analysis with different acceleration voltages: first at high acceleration voltage to determine what elements are in the specimen, and then at a lower acceleration voltage that is tailored to analyze the elements that you are analyzing.

The following example will demonstrate the calculations necessary to select the instrument parameters. In this example, the specimen was a silicon substrate with alternating thin layers of Au and Co, which was to be mapped in cross section.

(1) Determine critical excitation energies for the elements in the specimen.

The critical excitation energies E_c for the two elements of interest are:

Co: 6.929 keV ($K\alpha$ line) or 0.776 keV ($L\alpha$ line)

Au: 9.713 keV ($L\alpha$ line) or 2.123 keV ($M\alpha$ line)

The accelerating voltage needs to accelerate electrons to at least this energy in order to excite electrons in these shells and cause characteristic x-rays to be emitted.

(2) Determine which electron shells you want to interact with, and determine the minimum electron energy E_o required.

E_o affects the x-ray count rate: higher E_o yields higher x-ray counts so you collect data more quickly. As well, higher E_o increases the interaction volume, which reduces the resolution.

E_o= 2.123 keV for Co ($L\alpha$) and Au ($M\alpha$)

E_o = 9.712 keV for Co ($K\alpha$) and Au ($L\alpha$)

(3) Determine the acceleration voltage to achieve energy E_o that maximizes separation of spectrum peaks from background signal, for the peaks you are studying.

Refer to Figure 1, page 70, of "Let's familiarize ourselves with the SEM!" This plot shows the relationship between I_{cha}/I_{con} and E_o/E_c.

I_{cha}: intensity of characteristic x-ray
I_{con}: intensity of the continuous background x-ray signal

This curve shows a maximum in I_{cha}/I_{con} (the separation of peaks from the background signal is optimal) when E_o/E_c is approximately 3. Using an E_o/E_c ratio of less than 3 will still result in characteristic x-rays, but the peaks in the spectrum will be less distinct.

Based on lower energy, E_o = 2.123 keV * 3 = 6.37 keV (round up to 7 keV)
Based on higher energy, E_o = 9.712 * 3 = 29.136 keV (round up to 30 keV)

(4) Determine the spatial resolution for bulk specimen x-ray mapping. From Microscopy and Microanalysis volume 12, pp2-25 (2006), the spatial resolution in μm is

$$R_x = \frac{0.064\left(E_o^{1.68} - E_c^{1.68}\right)}{\rho}$$

Where ρ is the density of the element:

Au: ρ = 19.30 g/cm^3
Co: ρ = 8.90 g/cm^3

You will need to calculate R_x for all the combinations of E_o, E_c, and ρ under consideration. For example, assuming you use $E_o = 7$ keV, the spatial resolution for Au and Co are:

$$R_x(Au) = \frac{0.064(7^{1.68} - 2.120^{1.68})}{19.30} = 0.075\ \mu m$$

$$R_x(Co) = \frac{0.064(7^{1.68} - 0.776^{1.68})}{8.90} = 0.184\ \mu m$$

Things get worse if you use a large E_o to increase x-ray count rate:

$$R_x(Au) = \frac{0.064(30^{1.68} - 2.120^{1.68})}{19.30} = 0.993\ \mu m$$

$$R_x(Co) = \frac{0.064(30^{1.68} - 0.776^{1.68})}{8.90} = 2.175\ \mu m$$

(5) Determine instrument magnification, such that the width in μm of the EDS map pixel is approximately the same as R_x.

It is recommended to oversample (pixels smaller than resolution) by 2:1 at most. The maximum size of the EDS map area in the Bruker software on our Hitachi S-4800 SEM is 640 px x 480 px. You can measure the area size in μm using the controller program on the SEM computer. The width of a pixel in the EDS map is:

$$W_p = \frac{W}{640}$$

Where W is the EDS map width in μm, as measured by the SEM controller computer. Adjust the magnification (measuring W at each magnification) until $W_p = R_x$.

(6) Determine total sampling time.

To achieve 2σ precision of +/- 1%, use $\sigma = \sqrt{n}/n$, therefore n = 40,000 counts per pixel

Adjust your total sampling time such that n counts are collected for each pixel in the map.