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X-ray Microanalysis of Nanoparticles in the Scanning Electron Microscope

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The use of nanotechnology is increasing rapidly in many sectors of society with the introduction of nanoparticles into a wide variety of consumer materials ranging from automotive paint to consumer products such as invisible sunscreens. As the use of nanomaterials increases, characterization methods are needed not only for the manufacturing sector to ensure product quality but also for the regulatory and health sectors to protect the environment and ensure worker safety and health. One of the techniques which is being considered for chemical characterization of nanoparticles (diameter < 200 nm) is the scanning electron microscope (SEM) equipped with an energy dispersive x-ray spectrometer (EDS). The x-ray microanalysis of nanoparticles in the SEM is complicated by the fact that under conventional operating conditions, the sizes of the electron beam interaction volume and subsequent x-ray generation volume are on the order of micrometers in most materials, which is considerably larger than the size of the nanoparticles. The result of this size discrepancy is that a significant portion of the electron beam is scattered out of the nanoparticle volume and can excite x-rays from adjacent particles or the mounting substrate. These spurious x-rays degrade the spatial resolution and analytical results by contributing to the x-ray spectrum obtained from the particle being directly interrogated by the beam.

Two analytical protocols that are being considered to improve the x-ray spatial resolution / analysis of nanoparticles in the SEM are. (1) The use of a low-voltage electron beam 5 keV or less to minimize the electron interaction and x-ray emission volumes. For example, the range of electrons in Al at 5 keV is only about 400 nanometers compared to about 4200 nanometers at 20 keV. Fig. 1 is a plot of the electron trajectories at 3 keV in a 100 nm diameter Al particle that shows, other than backscattering, most of the electron trajectories are contained within the particle volume. (2) The use of the highest electron beam voltage available on the SEM, usually 25 keV – 30 keV. The higher voltages coupled with mounting the particles on a thin-film substrate (e.g. 10 nm C) minimizes the x-ray emission volume since a large fraction of the electrons passes through the particle volume with little scattering. This means the beam broadening within the particle volume is minimal resulting in an x-ray emission volume that is in essence highly collimated as shown in Fig. 2, a plot of electron trajectories at 25 keV for a 100 nm diameter particle.

We are currently using NISTMonte to investigate the applicability of these two protocols for nanoparticles analysis [1]. Figs 3 and 4 show the synthesized x-ray spectra from the calculations of 2000 electron trajectories for two adjacent 100 nm particles one Al_2O_3 and the other Fe_2O_3. The calculations were run at beam voltages of 3 keV on a carbon substrate and 25 keV on an infinitely thin substrate. The beam position was 25 nm from the center of the Al_2O_3 particle in the direction of the Fe_2O_3 particle. The magnitude of the Fe L peaks relative to the Al K is significantly less for the 25 keV spectra compared to the 3 keV spectra. These results indicate that for 100 nm particles the high voltage protocol is superior to the low voltage protocol resulting in less beam scatter with much lower interference from the excitation of the adjacent Fe_2O_3 particle.

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References

[1] Nicholas Ritchie, submitted to Surface and Interface Analysis.

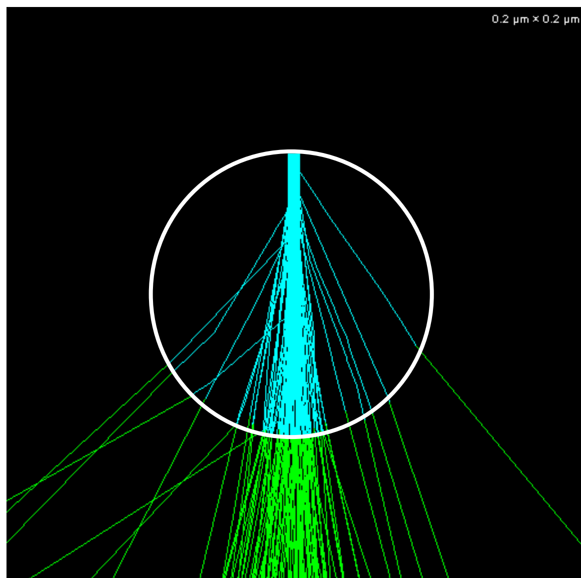

Fig. 1. Electron trajectory plot for a 100 nm diameter particle (outlined in white) at 25 keV.

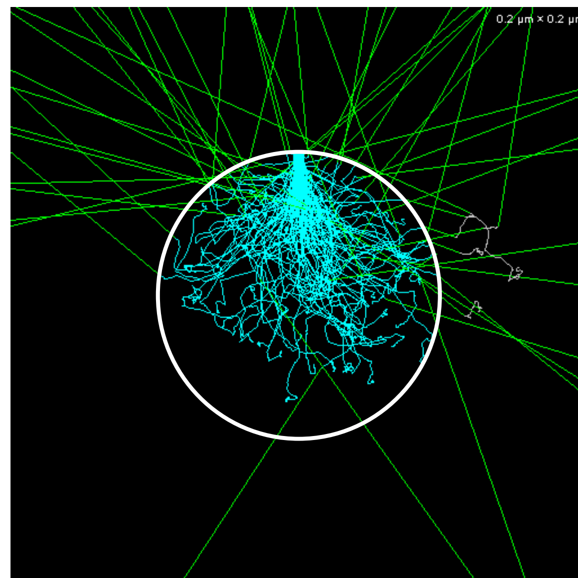

Fig. 2. Electron trajectory plot for a 100 nm diameter particle (outlined in white) at 3 keV.

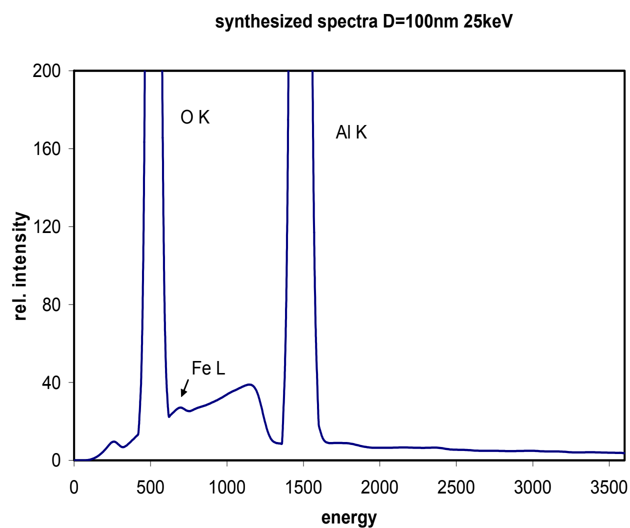

Fig. 3 Synthesized spectrum 25 keV.

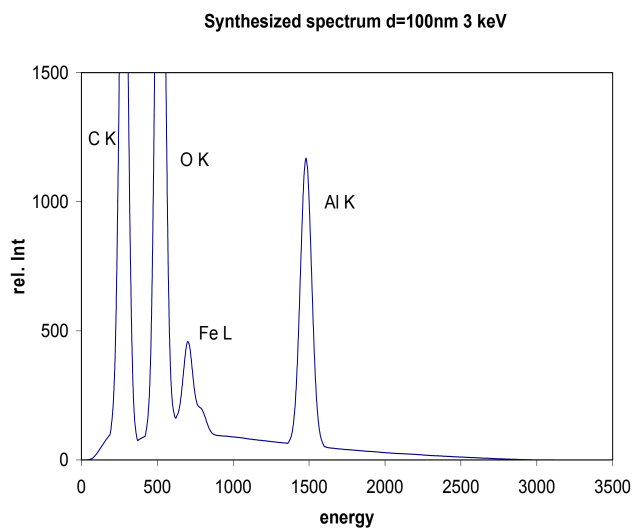

Fig. 4. Synthesized spectrum 3 keV.

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