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Electron Counted STEM-EELS Spectroscopy Optimized for low kV (< 80 kV) via Hybrid Pixel Detection

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Single electron counting direct detection cameras dramatically improve signal collection efficiency by rejecting signal read nose. As a result, these detectors have considerably higher detective quantum efficiency (DQE) than conventional indirect detection cameras which gives significant benefits to any (scanning) transmission electron microscope ((S)TEM) experiment that benefits from; low specimen dose, high speed acquisition, or targeting weak scattering signals such as high energy ionization edges in the electron energy-loss spectrum. Integrating a direct electron detector into a post column energy filter is particularly advantageous as this configuration enables the acquisition of: (energy filtered) counted images, selected area diffraction (SAD) patterns, convergent beam electron diffraction (CBED) patterns, and counted electron energy-loss spectroscopy (EELS) data, from the same specimen area as part of a single experiment.

Monolithic direct detection electron counting sensors have offered significant improvements for analytical TEM [1] but are optimized for intermediate voltage (> 80 kV) operation. Conversely, hybrid pixel sensors are optimized for lower voltage operation. This low voltage operation is highly compelling for (S)TEM analysis of many materials systems. Knock on damage is reduced or eliminated at low kV, which is ideal for materials that suffer little radiolytic damage yet are highly susceptible to knock on damage such as: transition metal dichalcogenides (TMDCs) or carbon-based materials (graphene, carbon nanotubes, etc.) [2, 3]. In addition to reduced knock-on damage, increased scattering contrast is also typically observed at reduced beam energy which is further beneficial for STEM analysis of weakly scattering low atomic number ($Z < 6$) materials.

Here we use a hybrid pixel based direct detection EELS spectrometer (GIF Continuum K3 with Stela) to demonstrate the advantages of low kV EF-4DSTEM and STEM EELS analysis [4]. DualEELS™ spectrum imaging has been performed on TMDC encapsulated silicon nanoparticles (MoS_2 / Si) at low beam energy (60 kV) and low probe current (20 pA) to avoid electron beam damage to the TMDC layer. An RGB composite of the silicon L, molybdenum M and sulfur L elemental maps with plural scattering correction applied are shown in figure (1). Summed dualEELS spectra extracted from the points shown in figure (1) are shown in figure (2). The silicon L, molybdenum M and sulfur L edges are clearly visible in core loss spectra extracted from the particle center and edge respectively. Advantages of plural scattering correction are clearly observed by comparing specimen relative thickness from the two positions and further comparing single scattering distribution and raw extracted core-loss spectra.

In addition to TMDCs, a wide variety of materials have been analyzed to investigate the benefits of low dose and low dose rate acquisition conditions, in addition to low kV analysis. These include materials such as: oxyfluorides, mixed halides, hetero-anionic materials, hybrid soft/hard interfaces, biomaterials and polymers. The advantages of these acquisition approaches will be presented [5].

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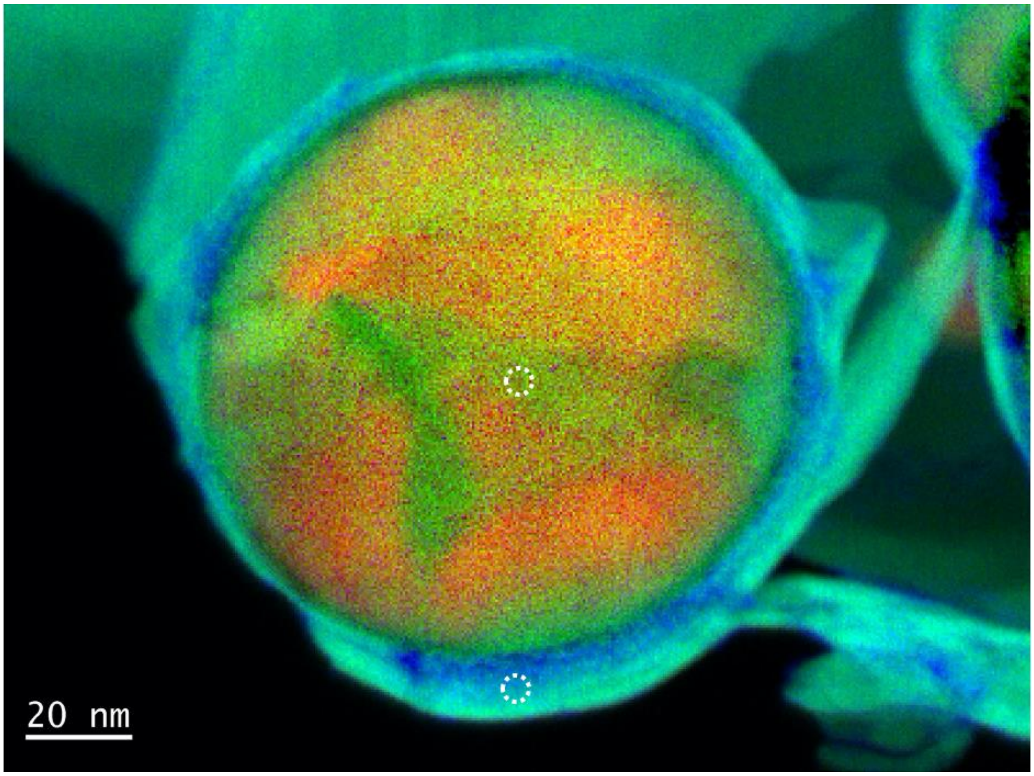

Figure 1. RGB composite of EELS elemental maps acquired from MoS_2 encapsulated Si nanoparticle at 60 kV and probe current 20 pA. The silicon L, molybdenum M, and sulfur L maps are shown in red, blue and green respectively.

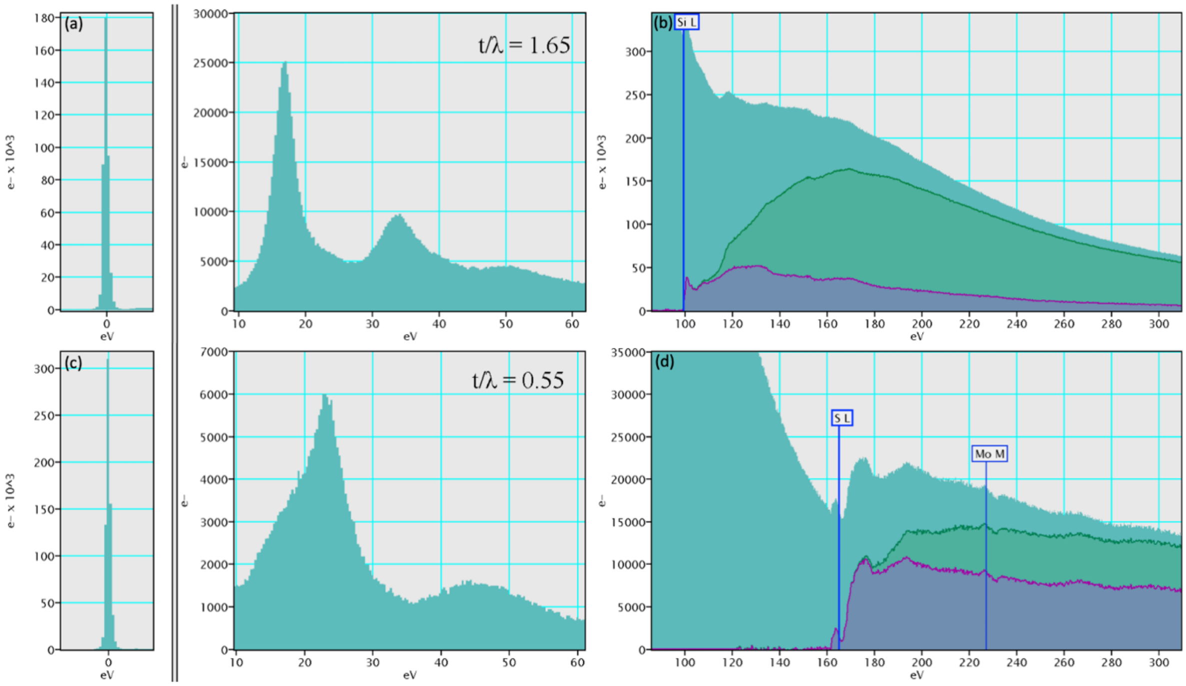

Figure 2. Dual-EELS spectra extracted from SI sub-regions shown in figure (1). The spectral pair taken from the nanoparticle center (a, b) comprises 7084 SI pixels summed. The spectral pair (c, d) taken from

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the nanoparticle edge comprises 2518 SI pixels summed. Raw spectral data is shown in teal. Core-loss spectra extracted using a power law background subtraction method are shown overlaid in green and single scatter distribution (SSD) spectra calculated using a Fourier ratio approach are shown overlaid in purple. Specimen relative thickness was calculated using a log ratio approach and is shown on the low loss spectrum in each case.

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