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SEM-EDS Mapping at the Nanoscale – the Low Voltage Approach

Ifat Kaplan-Ashiri[1]

[1.] Electron Microscopy Unit, Chemical Research Support Dept., Weizmann Institute of Science, Rehovot, Israel.
* Corresponding author: ifat.kaplan-ashiri@weizmann.ac.il

Scanning electron microscopy (SEM) is widely used as an imaging technique of bulk materials and surfaces in physics, chemistry and life science. The interaction of electrons, accelerated through the column, with the specimen results in the emission of different signals such as electrons, x-ray photons and occasionally light in the visible range. The different signals are the origin of the various contrasts and sample's information we can collect on every scan. While secondary electrons and backscattered electrons are deployed for topographical contrast imaging and material contrast imaging respectively, x-rays and visible light photons provide us with compositional information in the form of intensity mapping or spectra.

The various signals are emitted from different depths and volumes; secondary electrons are emitted relatively close to the specimen surface (nm range) while x-rays are emitted from larger depths, in the microns range. Simultaneous acquisition of secondary electrons (topographical imaging) and x-rays (elemental analysis and imaging) results in images with different spatial resolution. By reducing the accelerating voltage, the volume of interaction can also decreased and the spatial resolution can be improved.

Energy dispersive x-ray spectroscopy (EDS) is used to detect and analyze the emitted x-ray photons and provide us with elemental analysis of the specimen both qualitatively and quantitatively. Usually, the acceleration voltages are set to an over-voltage 1.5-3 times the characteristic x-rays energy of the K shells and L shells, to get a good signal to noise ratio. These conditions dictate the use of relatively high acceleration voltages as the atomic number increases and result in large depths (µm range) of x-rays photons emission. Therefore, EDS measurements are still a challenge when it comes to achieving better spatial resolution for the detection of nanoscale materials or resolving elemental information in the nanoscale range.

In the last decade the technology of EDS has tremendously improved as the SiLi detectors were replaced by silicon drift detectors (SDD), resulting in larger area detectors with higher counts rates. Nevertheless, one still needs to acquire spectra for long periods of time (many minutes) in order to get sufficient signal to noise, with spatial resolution in the µm range. To improve the spatial resolution by decreasing the accelerating voltage, we have been using new EDS technology.

A retractable annular four channel silicon drift detector (Bruker FlatQUAD) is installed on our SEM, it can be positioned below the pole piece and above the sample, it enables take off angle of 60-70° with no shadow effect. This technology improves tremendously the signal to noise ratio; hence, it makes elemental analysis and mapping of nanomaterials at low accelerating voltages feasible.

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EDS analysis and mapping of various nanomaterials at low voltages using the new technology will be presented together with the simulated emission depth and radii of the characteristic x-rays at different acceleration voltages.

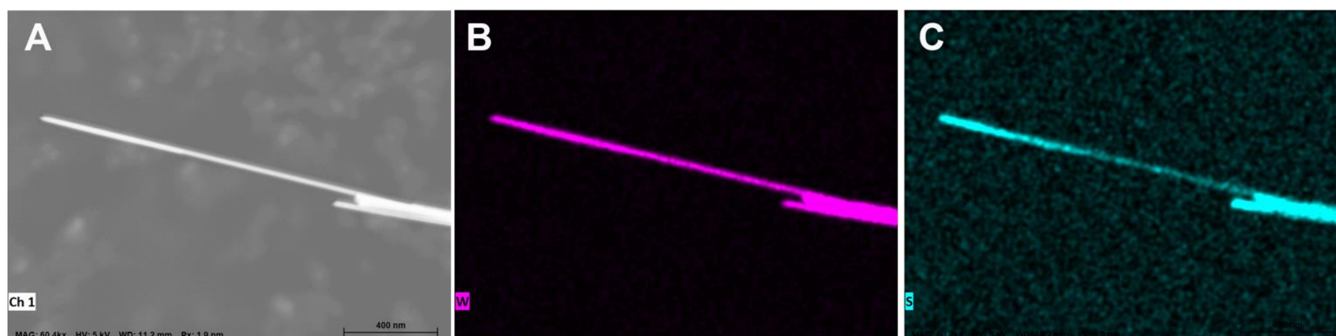

Figure 1. SEM (A) and elemental mapping (B and C) of WS_2 nanotube at accelerating voltage of 5kV

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