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IT-5-O-2552 Imaging and X-Ray Microanalysis at the Nanoscale with a Cold-Field Emission Scanning Electron Microscope

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The scanning electron microscope (SEM) was primary developed for imaging applications. With the introduction of the Si(Li) energy dispersive spectrometer (EDS), simultaneous imaging and x-ray microanalysis became possible. However, long working distance and high current were needed because the position and small solid angle of the EDS detector. SEM was initially and is still optimized for imaging applications, where the high spatial resolution is generally obtained at short working distance. This problem is still relevant today and unfortunately x-ray microanalysis is never performed in the best imaging conditions, i.e., not with the smallest probe size. With the introduction of an annular silicon drift detector (SDD) system, scanning electron microscopy is facing a revolution. This detector is inserted below the objective lens which gives a higher solid angle (up to 1.2 sr). In consequence, a lower working distance and probe current can be used. An improved spatial resolution becomes possible during x-ray microanalysis. At this point, the time required for x-ray imaging will be of the same order as for the atomic number contrast images achieved through backscattered electrons (BSE) imaging.

Carbon nanotubes (CNTs) decorated with platinum (Pt) nanoparticles are often used to evaluate the spatial resolution of cold-field emission scanning electron microscope (CFE-SEM). Figure 1 shows an example of high spatial resolution imaging and x-ray microanalysis of CNTs at low accelerating voltage (2.5 kV). A resolution of 19 nm and 24 nm were measured with SMART-J on the SE micrograph and the Pt x-ray map, respectively. Figure 2 shows another example of high spatial resolution imaging x-ray map obtained with an annular SDD of CNTs with low voltage scanning transmitted electron microscope (LVSTEM) mode at 20 kV. The dark-field micrograph had a spatial resolution of 6.5 nm and the Pt x-ray map had a spatial resolution of 8.9 nm. Currently, this system is limited to accelerating voltage below 20 kV and the shortest working distance is around 10 mm, which is shorter than the one used with a conventional SDD (15 mm on our system).

With this x-ray detector installed on a HITACHI SU-8230 cold-field emission scanning electron microscope, quantitative x-ray microanalysis with high spatial resolution at low beam energy and low current becomes possible with the possibility of using the various different type of imaging at the same time. Also, since the count rate can be as high as 1,500 kcps with our system, which lowers significantly the detection limit of elements as well as the minimum feature sizes of different phases that can be distinguished.

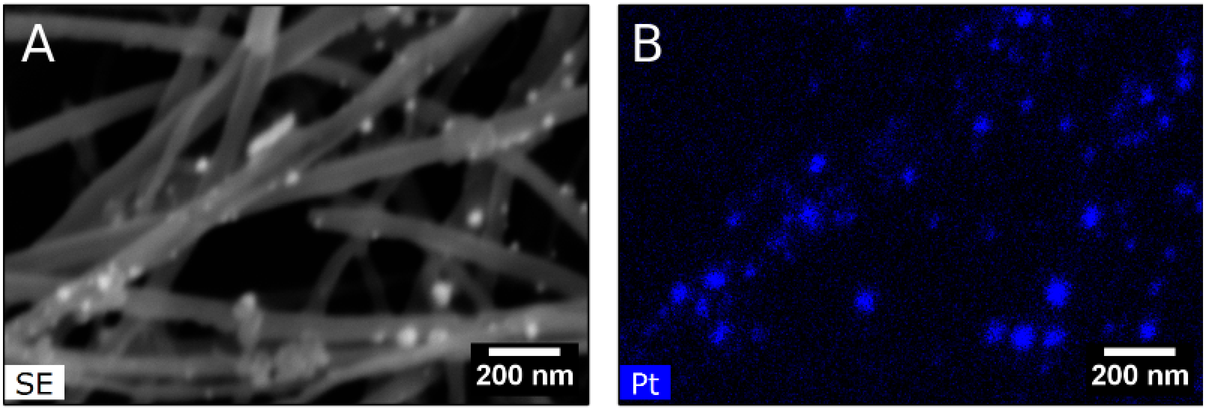

Fig. 1: Secondary electron micrograph of CNTs decorated with Pt nanoparticles was acquired at an accelerating voltage of 2.5 kV and a working distance of 9.4 mm. The Pt X-ray map was acquired with an annular silicon drift detector. The map acquisition time was 1433 s with a count rate of 81 kcps.

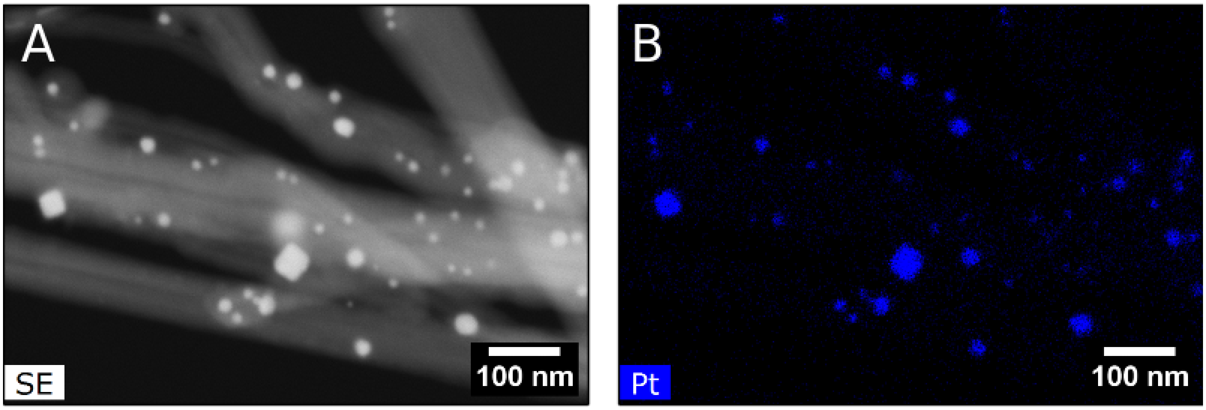

Fig. 2: Dark field micrograph of CNTs decorated with Pt nanoparticles was acquired in LV-STEM mode. The Pt X-ray map was acquired with an annular silicon drift detector. An accelerating voltage of 20 kV and a working distance of 10.5 mm were used. The map acquisition time was 412 s with a count rate of 7 kcps.