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New detection methods for fast analysis of nano-particles

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In the last years, the sub nanometre spatial resolution at low beam landing voltage of the extreme high resolution scanning electron microscope (XHR SEM) [1] has enabled a routine characterization of particles no bigger than a few nanometres, without any specimen preparation [2]. To access even more precise information and work on a larger variety of materials, it is critical to efficiently collect the signal composed of secondary (SE) and back-scattered (BSE) electrons, and pick from the signal the fraction that delivers the most relevant contrast from the particles and their environment.

Development of the energy dispersive detection infrastructure for the XHR SEM

The newly developed infrastructure in the XHR SEM optimises the signal collection and the contrast thanks to specific optical elements and three detectors built inside the electron column. The immersion lens of the column not only focuses the electron beam on the sample, it also filters the BSE. The closer the BSE energy from the primary beam, the better these BSE are focused and the closer their trajectory is to the optical axis.

Near no-(energy)loss BSE are collected by a first detector called ICD, located higher up in the column (figure 1). Just below, a second detector called MD gathers the low-loss BSE. The remaining higher loss-BSE are directed toward the third detector (TLD), in the lower part of the final lens. To ensure clean BSE imaging, SE are blocked from entering the lens. Both the ICD and MD consist of a solid-state diode with boron layer technology that allows detecting electron energies as low as 200 eV. With this new detection setup, it is possible to work at short working distance to further optimize the signal collection. All three detectors can be used simultaneously: all BSE are detected at once, and their respective annular and energy filtering provides complementary materials information.

An additional optical element, Beam Deceleration (BD) [3], whereby a negative potential is applied on the surface of the sample, is added to the setup for the characterization of particles using very low landing energies (from a few 100 eV to 20 eV). In this case, all SE and BSE are reaccelerated towards the ICD and very high resolution, surface sensitive information is obtained.

Fast characterization of nano-particles and contrast optimization

Several types of nano-particles were characterized using this detection, using a Verios XHR SEM. In figure 2, high-loss, low-loss and no-loss BSE images of catalyst particles on top of nano-tubes are presented. The catalyst particles are coming out clearly when imaging with lower-loss BSE. Signal mixing can be used to further stress the part of the information that is of specific interest. Other particles, such as pyrite crystals in a shale sample or catalyst particles in a fuel cell electrode have been imaged clearly and at high resolution, using the same method. High resolution details of the carbon contamination on the surface of gold particles, using very low landing energies and the ICD, are shown in figure 3.

The collection of the complete SE/BSE signal and the possibility to filter it more accurately are key to better contrast tuning and understanding of its mechanisms. Using the energy dispersive detection in the XHR SEM, the fast characterization of various types of particles with very high resolution, high contrast and no beam damage has been demonstrated.

1. R. Young et al, Proc. SPIE7378, Scanning Microscopy 2009, 737803
2. T. Hachisu et al, Journal of Magnetism and Magnetic Materials 324 (2012) 303-308
3. D. Phifer et al, Microscopy Today (2009) 40-45

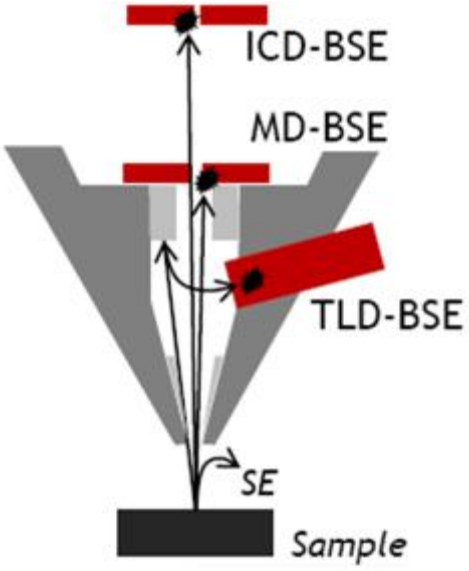

Figure 1. Energy dispersive detection setup in the XHR SEM, BSE mode.

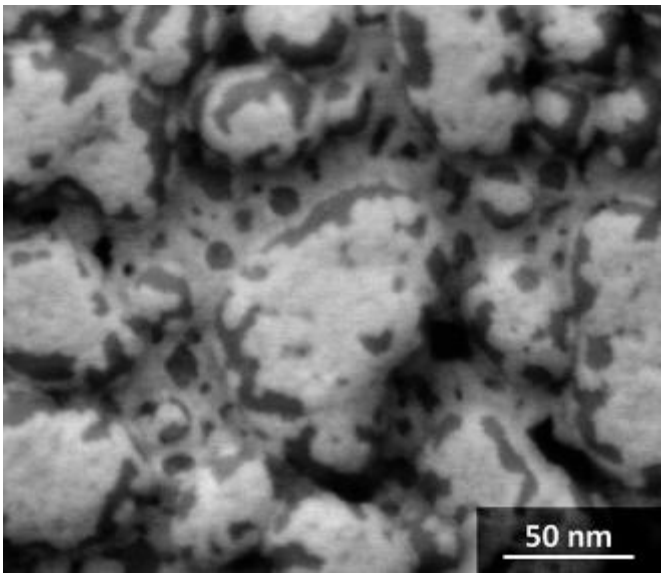

Figure 3. Gold particles on a Carbon substrate, imaged at a landing energy of 50 eV and with a beam current of 6 pA.

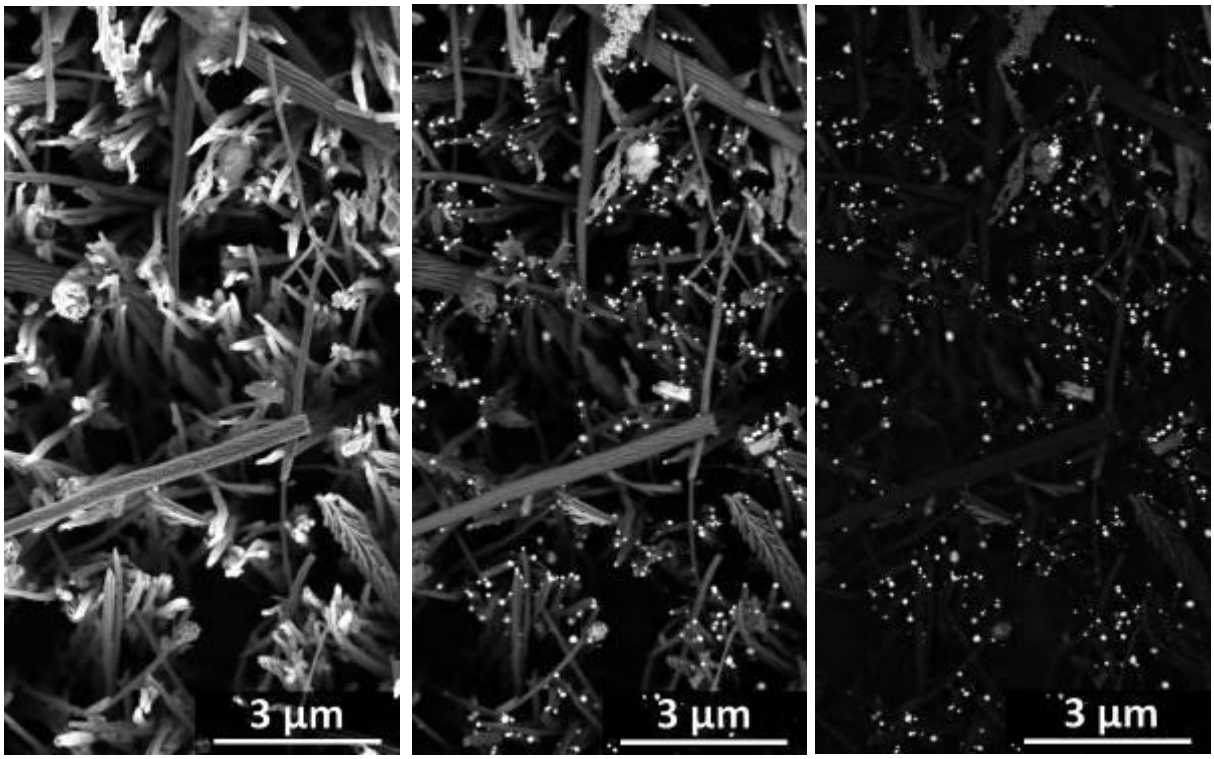

Figure 2. Nanotubes with catalyst particles on top, imaged at a landing energy of 2 keV simultaneously with the TLD (left), MD (middle) and ICD (right) detectors.