The STEM multi-signal approach: learning the most from your nano-object

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In electron microscopy, the key information results from the interaction mechanisms between the primary beam of electrons and the material under study. Consequently, all recent efforts and successes for improving the instrumentation involved in acquiring most signals resulting from this interaction and for developing the required software for data processing and for theoretical simulation, have radically extended the domains of application for the newly designed instruments. The demonstrated breakthroughs in electron optics, such as the design, the practical realization and the use of correctors, filters and monochromators, together with the permanent progress in detector efficiency have pushed forward the performance limits, in terms of spatial resolution in imaging (sub-Å), as well as for energy resolution (sub 100 meV) in electron energy-loss spectroscopy (EELS). The STEM approach has proved to be well adapted to a thorough exploration, pixel after pixel, of the response of the specimen, because of the multiplicity of the detectors which can be implemented to capture simultaneously the different channels of information [1,2]. Spectrum-imaging modes deliver with a high level of accuracy structural maps through a set of angular detectors (HAADF, MAADF, ABF) in combination with elemental maps provided either by the core-loss EELS and/or the EDX signals. The improved energy resolution in EELS gives access to fine details in the electronic states which can be related to bonding, transfer of charge or environment variations at the unit-cell level. In the near-IR/visible/UV spectral domain, EELS can now be recorded simultaneously to the optical emission spectrum (CathodoLuminescence), thus combining the advantages of the ultimate resolution in space (electron spectroscopy) and in energy or wave-length (photon spectroscopy). As a consequence, the objects of the nanoworld, of natural or artificial origin, can now be fully characterized individually [3].

This spectacular broadening of the field of use for this new generation of electron microscopes will be demonstrated on two families of nanostructures: (i) *multi-valent oxide hetero-structures grown with atomic control of their layer thickness, together with their interfaces*. Beyond elemental mapping with atomic resolution now offered for nearly any element by the EELS and the EDX signals, monitoring the fine structures on the EELS core-loss edges (typically O-K and TM L_{23} edges) is essential for probing also local termination, strain, presence of vacancies and phase changes; (ii) *optical properties of metallic and semiconducting nanoparticles.* Surface plasmon modes have been mapped at typical energies down to 1eV and below, on a broad distribution of metallic particles with varying shape, size, substrate and interactions. It thus offers an alternative, with noticeably enhanced spatial resolution, to the more conventional optical techniques, for studying the distribution of the induced electro-magnetic fields in these photonics nanostructures. The combination of both spectroscopies – see figure – will extend its use to many cases involving optically emitting nanostructures, such as quantum dots or fluorescent nanoparticles.

Spectrum-imaging has generally connected up to now space and energy. No doubt however that in a near feature, other dimensions such as time, will be concerned giving access to a brand new domain of dynamical studies. This trend will grow together with the opportunity offered by Cs corrected microscopes to accommodate nano-laboratory devices (light injector, nano-indentation, variable temperature, environmental cells) in the specimen stages taking benefit of the enlarged space gap between the objective pole pieces [4].

References

[1] C. Colliex *et al,* Phil. Trans. R. Soc. A **367** (2009) 3845

[2] C. Colliex, J. Electron Microscopy **60** (2011) S161

[3] See MRS Bulletin, January 2012, on “Spectroscopic imaging in electron microscopy”

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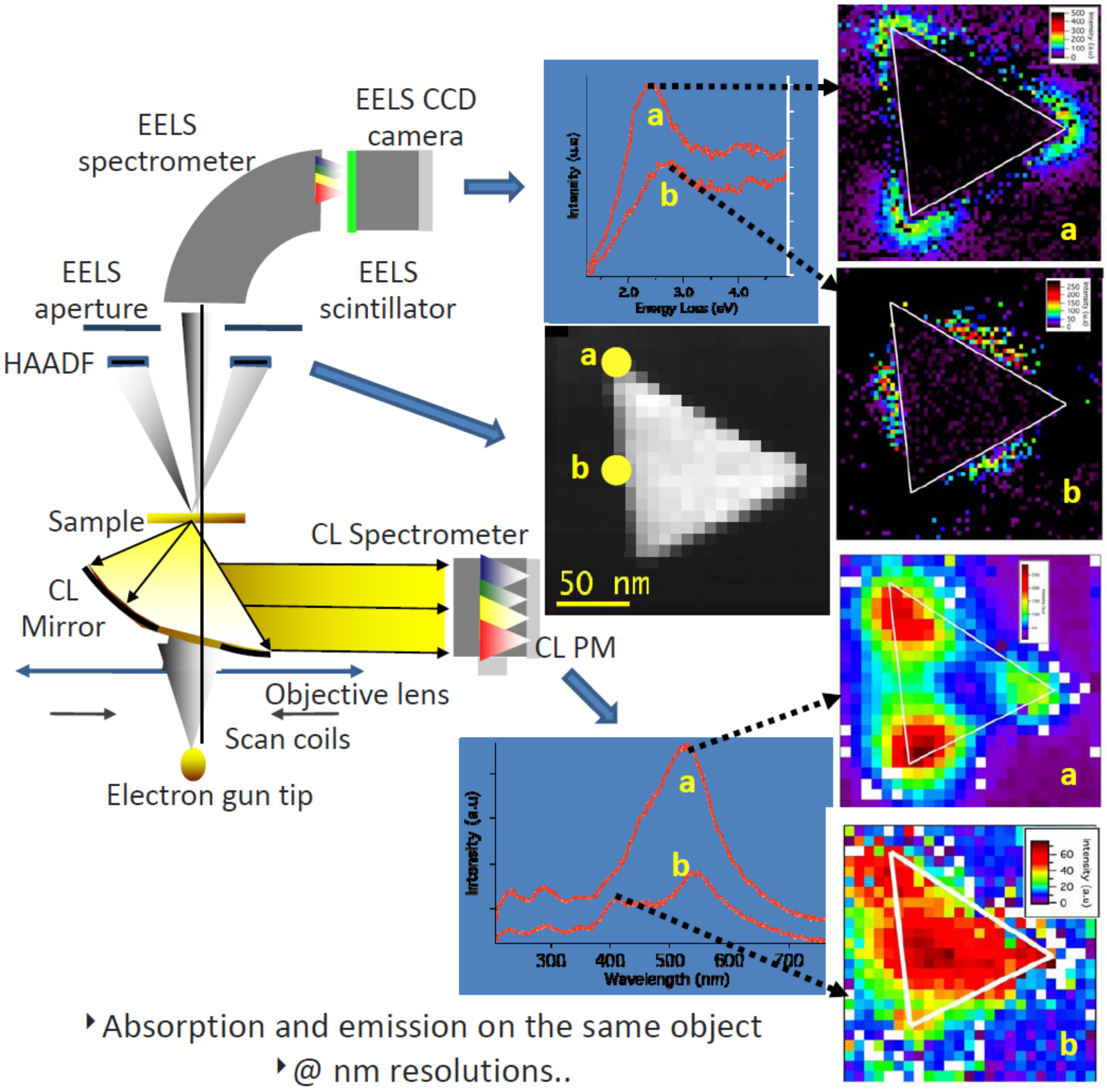

Figure. *Schematic diagram of a STEM microscope running in the multi-signal mapping mode. For each position of the incident probe on the specimen such as (a) or (b) indicated on the HAADF image of a gold nanoprism deposited on a thin amorphous carbon layer, one simultaneously records three signals (HAADF for topographical and structural mapping; EELS spectrum in the visible domain, from 2 to 4 eV, see top spectra;, optical emission – CL – spectrum between 300 and 700 nm in wavelength, see bottom spectra). The four maps on the right display the distribution in intensity of the major peaks identified in the spectra (diagram courtesy of M. Kociak and L. Zagonel))*