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Low-Loss Studies on Metallic and Insulating Nanostructures Using a Monochromatic Electron Beam

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In the last two decades, transmission electron microscopes have undergone a large number of improvements allowing ~ 100 meV energy resolutions for a close to one angstrom electron beam. These performances offer new possibilities in different TEM fields, in particular these ones concerning the low-energy-loss measurements: studies of the optical, dielectric and electronic properties of materials with unprecedented spatial information. In this contribution, we will present the opto-electronic properties studies carried out via low-loss EELS measurements of two different kind of nanostructures: gold nanoparticles (NP) of different sizes and shapes, as well as boron nitride nanotubes (BNNTs). These works illustrate the excellent capabilities offered by the use of a monochromator combined with deconvolution procedures, in a Cs probe corrected STEM, to study these properties within a very good spatial resolution.

These studies have been carried in a FEI Titan – XFEG - Cs probe corrected microscope equipped with a monochromator, working at 60, 80 and 300 KV and with an energy resolution, before deconvolution procedure, below 200 meV (even 150 meV for some of the cases). The spectra were recorded using spectrum-line(-image) mode [1,2]: 40-60 spectra (of 30-40 ms/each) were acquired for each probe position following a line (a 2D region) across the nano-object. We studied two different kind of nano-objects: gold nano-prisms of different morphologies (spheres, rods, stars, bi-pyramids…) and sizes (from few to several tens of nanometers), as well as multiwalled-BNNTs of few tens of nanometers in diameter (going from few to several tens of layers) [6].

The low-loss region of EEL spectra is strongly affected by the broad tails of the zero-loss peak (ZLP). We have used a monochromator which highly mitigates this effect. Moreover, in order to enhance spectral resolution and to have access to spectroscopic information below 1 eV, we have employed a Richardson-Lucy deconvolution procedure [3,4]. In this contribution, we will discuss about the benefits of the combination of these hardware and software procedures. Figure 1 (a) and (b) show how it is possible to get an energy resolution of ~ 90 meV and extracting spectroscopic information from below 1 eV (Fig. 1 (c)).

It is well known that in the case of metallic nanoparticles (NPs), their size, morphology, as well as, their local dielectric environment have strong impact on their optical properties [5]. In order to study all these effects, we have chosen gold NPs with a wide range of sizes, shapes, and dielectric environments. Figure 1 (c) displays low-loss spectra recorded on a core-shell Au-Ag bi-pyramidal NP (four first spectra from the bottom), compared to those recorded on a similar pure Au NP (three dashed ones at the top). A shift of some of the modes, as well as, other modifications in the response of the surface plasmon modes due to the morphology and the size of the Au-NPs (for instance, nanorods (Fig. 1 (b))) can be observed. All these results will be deeply discussed in this presentation.

In the case of BNNTs, we are interested into study the distribution of different excitations in the low-loss region due to different effects: bulk (number of walls and/or diameter), dispersion (orientation/geometry) and defects (structural or presence of impurities). These results are very significant for their potential applications in nanophotonic devices.

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[6] We thank N.J. Zaluzec for preliminary studies on gold nano-spheres and nano-rods, as well as, O. Stephan and M. Kociak for stimulating discussions. S. Albalde, R. Alvarez and L. Liz-Marzan for providing some of the Au NPs used in these studies, as well as, M. Treguer, L. Roiban and O. Ersen for providing other kind of Au-NPs and D. Golberg for multi-walled BNNTs.

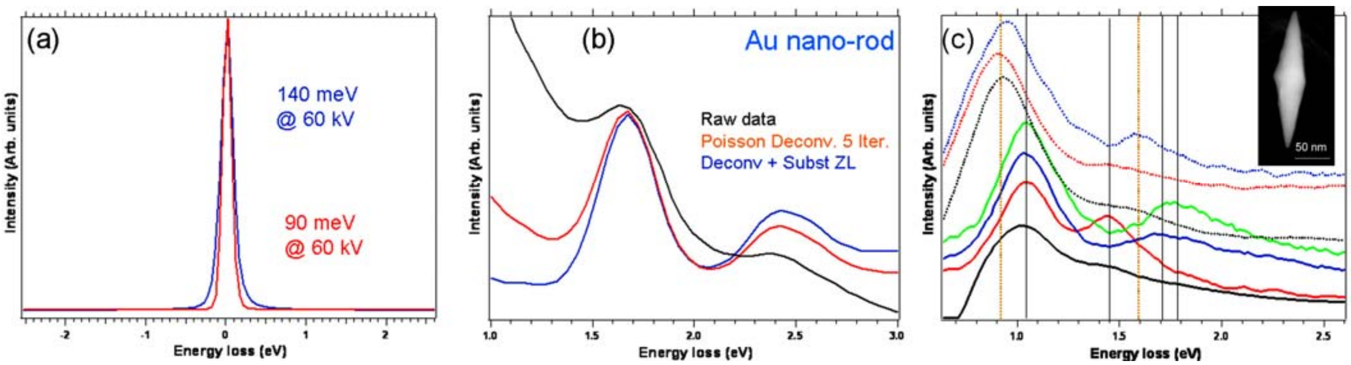

Figure 1. (a) Raw and deconvoluted (five iterations low-loss spectra, in red) low-loss spectra recorded on vacuum. **(b)** Low-loss EEL spectra acquired on an Au nanorod, showing the effect of the deconvolution and subtraction. **(c)** Low-loss spectra recorded on a core-shell Au-Ag bi-pyramidal NP (4 first spectra from the bottom), compared to those recorded on a similar pure Au NP (3 dashed ones at the top).

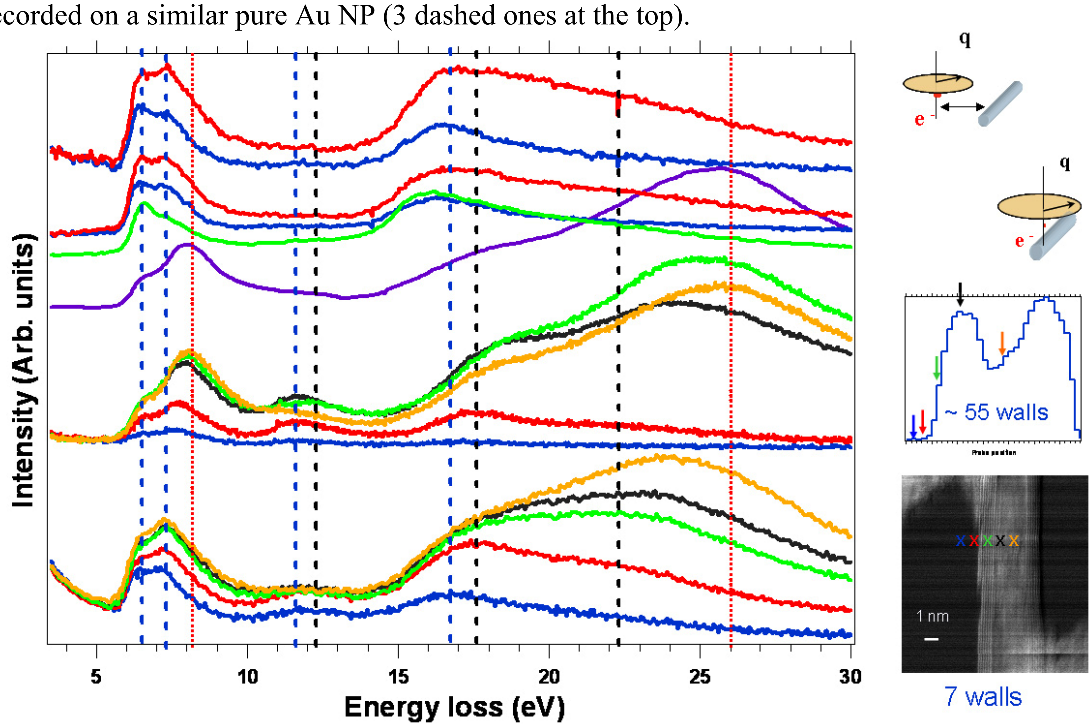

Figure 2. Low-loss spectra recorded on: (1) 7 walls BNNTs (first set of 5 spectra, positions marked on the HAADF image); (2) ~55 walls BNNTs (second set of 5 spectra, each spectrum recorded in the area marked in the intensity profile of the HAADF corresponding image); (3) Magenta: h-BN (electron beam parallel to the anisotropic axis); (4) Green: single-walled BNNT; (5) double-walled BNNT; and (6) triple-walled BNNT. From (3) to (6) see [2,4].

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