Direct observation of metal nanoparticles (NPs) electrodeposition on carbon nanotubes (CNTs) and glassy carbon supports by *situ* and *operando* TEM microscopy

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Metal Nanoparticles (NPs) are of great interest due to their exceptional catalytic, reactive and magnetic properties. Several applications, ranging from gas sensors, photodetectors, photovoltaic devices, and energy storage and conversion devices [1], are developed using as building block hybrid nanomaterials like decorated carbon nanotubes (CNT) or glassy carbon (GC). However, their device performances are strongly related with distribution, density, well-defined size, composition and morphology [2]. Particularly, electrodeposition process results a powerful method to form NP due to their key benefices, such as: low-cost of implementation, ambient operating conditions and the capability to control the properties of deposited materials such as size and density by adjusting the parameters involved in the deposition process. The real time survey of the evolution of nanosystems under certain electrochemical conditions (operando) enables to elucidate the NPs nucleation, formation and deposition depending on the electrodeposition parameters. The present work is mainly devoted to carry out in situ liquid TEM and STEM observations through a closed electrochemical cell, in order to distinguish the nucleation/formation NPs stages using different ionic salts. Firstly, cycle voltammetry (CV) technique was applied to identify the reduction pics for the formation of different metal NPs (Fe, Ni,Co and Cu). Subsequently, pulsed chronoamperometry (CA) was adopted to decorate CNT and GC supports, recording the evolution of the whole process: from the nucleation up to the NP growth. Figure 1 shows a decorated CNT with Ni NPs and their corresponding electrodeposition current and cumulative charge during the decoration process. The presented approach enables studying the influence of the main electrochemical parameters, such as, applied potential, flow rate, pulse duration on the NP formation, distribution, density and composition. In conclusion, the present study introduce the route to get a better understanding of the CNT and GC decoration process in order towards the precisely tailor of hybrid materials for the next generation of nanotechnological devices.

[1] S. Carnot, Réflexions sur la puissance motrice du feu et sur les machines propres à développer cette puissance (Bachelier, Paris, 1824)

[1] A. Gohier et al., High-rate capability silicon decorated vertically aligned carbon nanotubes for li-ion batteries, Adv. Mater. **24**, 2592 (2012).

[2] R.M. Penner et al. A Nose for Hydrogen Gas: Fast, Sensitive H 2 Sensors Using Electrodeposited Nanomaterials, Acc. Chem. Res. **50**, 1902 (2017).

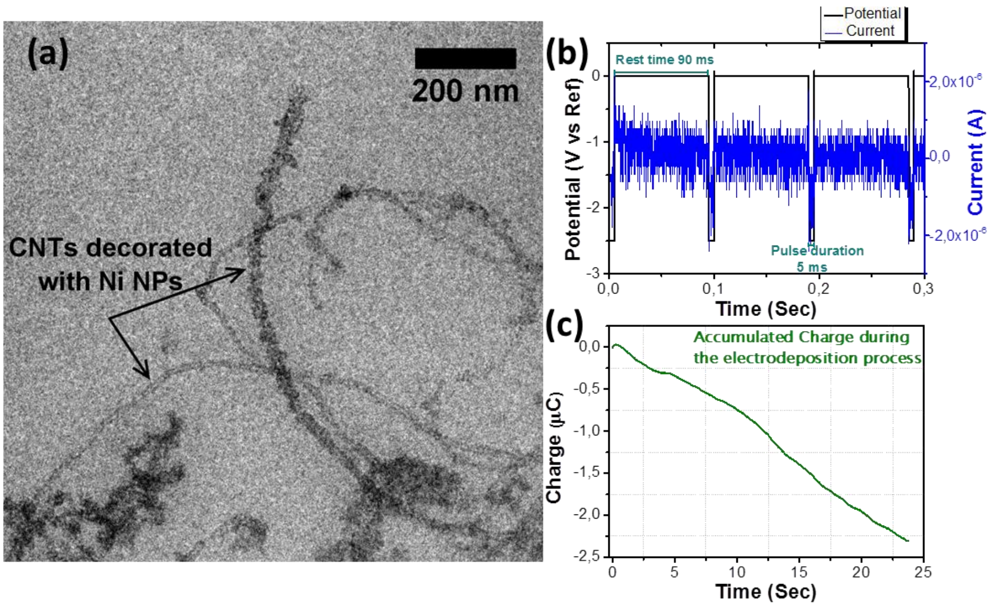

Figure 1: (a) Carbon nanotubes decorated with Ni NPS. (b) The associated applied potential during the electrodeposition process. (c) The corresponding accumulated charge.