Setting-up of a novel approach to in-situ TEM study of structure-transport correlations on single nanostructures

A. Taurino[1], M. Catalano[1], R. Krahne[2], E. Cociancich[3] and E. Carlino[3]

1. Institute for Microelectronics and Microsystems, Italian National Research Council (CNR-IMM) – Lecce Section, via Monteroni, 73100 Lecce, Italy
2. TASC-INFM-CNR National Laboratory, Area Science Park Basovizza, 34012 Trieste, Italy
3. National Nanotechnology Laboratory of CNR-INFM, 73100 Lecce, Italy

antonietta.taurino@le.imm.cnr.it

Keywords: In-situ TEM, FIB, electron-transport correlations, nanostructures

Nanoscience and nanotechnology, top research fields of this century, depend on material and device tailoring, which in turn is strongly related to the properties of materials on atomic scale.

Transmission Electron Microscopy (TEM) is a powerful and versatile tool for studying nanomaterials, widely used in nanotechnology. This is due to a variety of direct techniques, which allow to obtaining information about the structure, chemistry, electronic and magnetic properties of materials at the highest spatial resolution.

A step further in the knowledge of nanomaterials and nanodevices is represented by the possibility of studying at atomic resolution their structural properties while an external force-field is applied, such as electric and/or magnetic fields, thermal annealing, stretching, etc.. For example, studying the structural properties of a single nanostructure while it is biased by an external electric field would allow to achieve basic knowledge on the transport properties on atomic scale and to correlate them with the structure and chemistry of the nanostructure. So far, there are no approaches for in-situ microscopy, representing a real scale down of, for example, an I-V measurement on single nanostructure. This is very challenging since the capability to handle single objects with typical sizes of few nanometers is required.

In-situ structure-transport correlations from individual nanostructures have been obtained by few groups in the world, basically on carbon nanotubes and in-situ fabricated molecules and nanowires [1,2].

In our work, we are interested in developing a general and original approach, which can be applied to nanostructures with typical sizes of few nanometers, which represents a real scale down of a macroscopic measurement, thus allowing to transform the electron microscope in a nanolaboratory where it is possible to handle, contact and measure individual nanostructures. The principle of our attempt is logically easy and straightforward but in reality requires skill and new methods and instruments for nano-manipulation, nano-lithography, high precision machinery, TEM/STEM, and electrical characterization.

In this contribution, we report on the set up and optimization of the processes required for the fulfillment of the in-situ TEM experiments on single nanostructures at atomic resolution; in particular, we focus on i) the fabrication of the substrate, for the immobilization and electrical connection of the nanostructure; ii) the development of the TEM holder, with the wiring connecting the nanostructures to the external power supply, capable of mechanical stability necessary for atomic resolution imaging.

One of the key point in the fabrication of the substrate is the realization of the nanocontacts, with a typical gap of few tens of nanometers. A further complication is represented by the requirement of electron transparency for HREM in the region of the nanocontacts. The ideal situation for HREM would be with the nanostructure suspended on the nanocontacts without any supporting material below.

W. Grogger, F. Hofer, P. Pölt (Eds.): MC2009, Vol. 3: Materials Science,
DOI: 10.3217/978-3-85125-062-6-636 , © Verlag der TU Graz 2009

Recently, dual-beam focused ion beam (FIB) technique has emerged as a powerful tool for nanotechnology, due to its capability of lithography with nanometric resolution. For this reason, FIB has been successfully used for the realization of the nanocontacts.

A pattern of Ti/Au electrodes (see Figure 1 b)) was fabricated by optical lithography and metal evaporation on a 400x400 µm^2 Si_3N_4 membrane, 100 nm thick, of a commercial Si/Si_3N_4 substrate. Through cuts were realized by FIB on the electrodes, thus allowing to obtaining the narrowest gaps, without leakage currents, and satisfying the requirement of electron transparency. Figure 1 c) shows a representative cut obtained by FIB. The nanorods were then immobilized in the gap by electrostatic trapping. Different strategies were explored in order to overcome the high contact resistance between the rod tips and the electrodes.

For the fabrication of the TEM specimen holder, high precision machinery was necessary. The role of the holder is to host the contacted nanostructure and to insert it between the pole pieces of the objective lens of a commercial high resolution electron microscope, with an accuracy of ±50 nm. Figure 1 a) shows the design of the holder with the wires connecting the nanostructures, in the vacuum of $1x10^{-6}$ Pa, to an external power supply. The mechanical stability of the holder, necessary to allow atomic resolution, was also a crucial point of the work. Figure 1 d) shows the TEM image of the electrode gap, 30 nm wide, with some nanorods in between. The presence of amorphous material resulting from the trapping is evident. Strategies for the removal of the amorphous layer leaving untouched the nanostructures have been studied and optimized in order to inhibit its eventual contribution to the electrical signal.

1. H. Ohinishi, Y. Kondo, K. Takayanagi, Nature **395**(6704) (1998) 780-783
2. Z.L. Wang, P. Poncharal, W.A. de Heer, Microsc. Microanal. **6**(3) (2000) 224-230

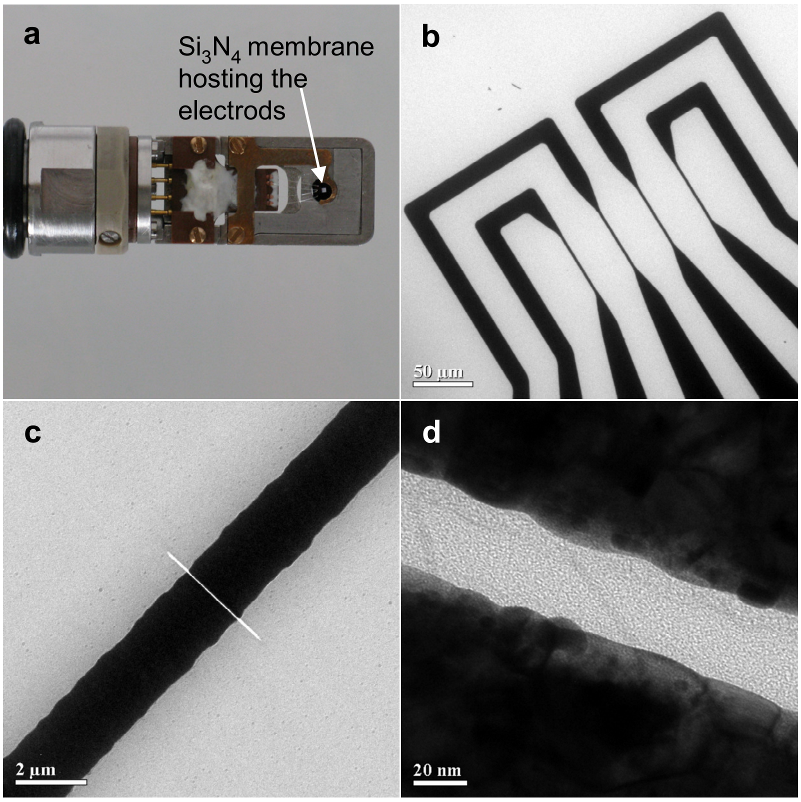

Figure 1. a) head of the TEM holder with the wires for the connection of the nanostructure to the external power supply; b) TEM image of the electrode pattern fabricated on the Si3N4 membrane; c) TEM image of one of the electrode with the FIB cut; d) high magnification TEM image of the electrode gap with some nanorods in between.

W. Grogger, F. Hofer, P. Pölt (Eds.): MC2009, Vol. 3: Materials Science,
DOI: 10.3217/978-3-85125-062-6-636 , © Verlag der TU Graz 2009