Comparative Study of High Resolution Electron Microscopy among Vanadium Oxide Nanotubes and NanoUrchins

V Lavayen[1]; C.O'Dwyer[2]; S. Newcomb[3]; M.A. Santa Ana[4]; E. Benavente[5]; G. Gonzalez[4]; C. M. Sotomayor Torres[6]

[1]Universidad Técnica Federico Santa María, Valparaíso 2390123, Chile, [2]Universidad de Chile, PO Box 653, Santiago, Chile, [3]Glebe Scientific Limited, Newport, Co. Tipperary, Ireland, [3]University of Limerick, Limerick, Ireland, [4]Universidad Tecnológica Metropolitana, Av. José Pedro Alessandri 1242, Santiago, Chile, [5]Catalan Institute of Nanotechnology, Universitat Autonoma de Barcelona, 08193, Spain
Email: vladimir.lavayen@usm.cl

Vanadium pentoxide nanostructures have attracted much attention due to the wealth of shapes and prospective applications in optical switching, energy storage as well as in functional nanomaterials. Inorganic nanostructures such as nanorods, nanotubes, and NanoCogs [1-7] have been studied to understand the correlation between structural and vibrational properties, as well the optical, electrical and synthetic methods among them. Here we present structural comparison characteristics among the vanadium oxide nanotubes (VO_x-NT) [1,4] and the nanotubular top section (Fig. 1) from a spherical, hollow-centered structure known as NanoUrchin [5], successfully synthesized by a chemical *douce* route, and VO_x-NT reported previously [1].
NTs in the NanoUrchin have an observed electron difraction interlaminar distance, 2.85 nm, that correspond a bi-layer of chains of the amine in *all-trans* conformation, however a geometrical model shows that this lenght is due to a double layer oriented amine either perpendicularly to the V_2O_5 planes with interpenetrated alkyl chains or forming an angle with them.
The tubes in the NanoUrchin are composed mainly of concentric, closed cylinders and such near-perfect tubes typically with an even number 3*n*, where *n* is a multiple parallel atomic layers of vanadate not observed in the VO_x-NTs. The longest NTs measured have several micrometers in length with diameters of ~80-100 nm and hollow centers around ~30-50 nm; Similar to the VO_x-NT [7,8].
Evidence for VOx layer branching can be seen at **S** and **T** in Fig. 1b. This image highlights the first observation of triplet layers, a feature never before observed in this hybrid material.
It was observed that VOx-NTs present structural defects due to experimental conditions (Fig 2,3), nonetheless most of NTs in the NanoUrchin are observed to be open-ended and appear to be rolled up as evidenced by the contrast variations at the tips, which confirm the enrolled mechanism described previosly to the VOx-NTs. A detailed observation of the ended part shows the presence of structural defects as well of the clear formation of some layer of carbonaceous material, the dark contrat field (Fig 1a) show higher crystallinity phases presence going down direction to the urchin core. The sidewalls of the tube are composed of regularly spaced lattice planes. Such defects are regularly observed in these tubes and in some instances; lattice-plane-termination dislocations are seen, Fig 2, 3.

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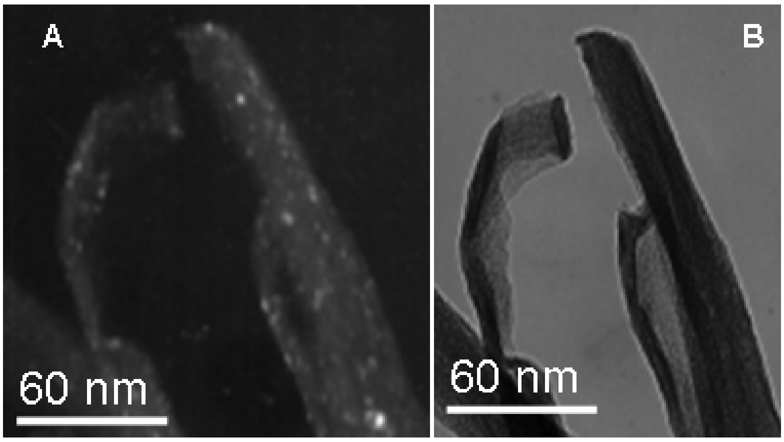

Figure 1. High-resolution TEM image along of the tip nanostructures of the NanoUrchin (a) dark field and (b) brigth field, it can be appreciate the presence of hybrid nanowings (white circle) structure and some V_2O_5 crytal phase.

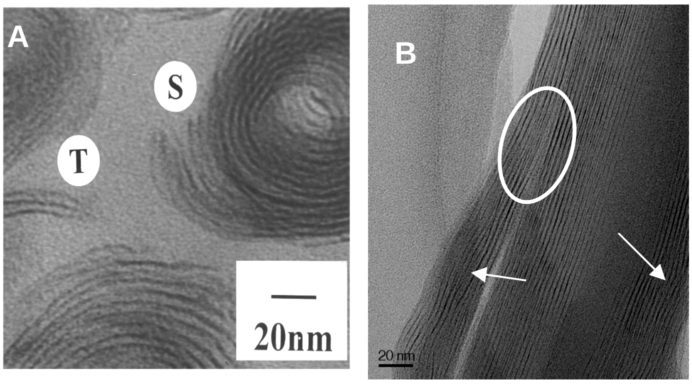

Figure 2. High-resolution bright field TEM image along (a) angled longitudinal directions showing the open-ended cross-sections. (b) high resolution image of adjacent NTs showing areas of atomic layer zipping, as well of structural defects (arrows).

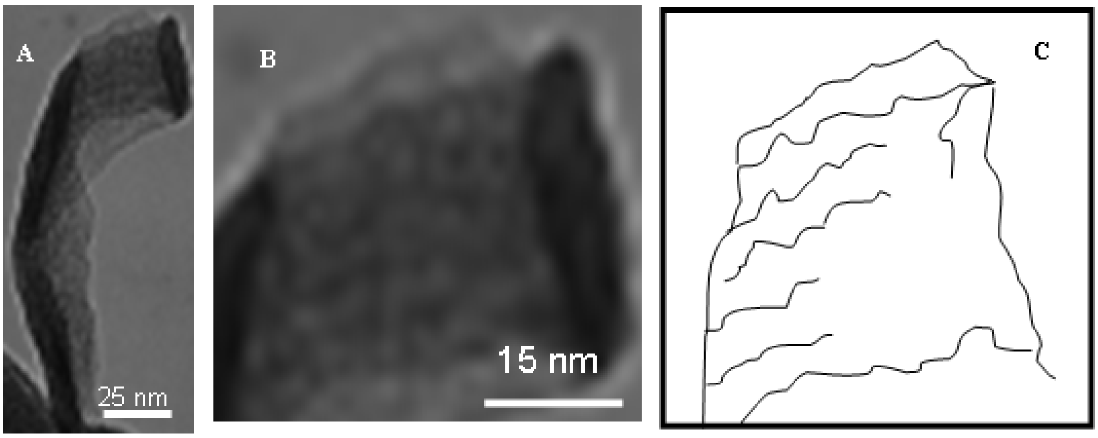

Figure 3. High-resolution bright field TEM image (a) deformed NanoUrchin tip. (b) Magnification of the Fig 3a showing the presence of topological defects and organic cover on the tube-ended. (c) organic formation scheme of NTs tip of the NanoUrchin.