Ultrahigh interlayer friction in multiwalled boron nitride nanotubes
Nanotubes production and characterizations

Two kinds of nanotubes have been used during this work: Chemical Vapour Deposition (CVD) grown Boron Nitride nanotubes (BNNT) and Arc Discharge (AD) Carbon nanotubes (CNT).

BNNT have been realized at the Laboratoire de Multimateriaux et Interfaces (LMI) in Lyon, France. CVD induces the decomposition, on the surface of a catalyst (metallic nanoparticles), of precursor gas: borazine ($\text{B}_3\text{N}_3\text{H}_6$) in flowing NH$_3$ gas. The precursor dissolves in the catalyst, diffuses through and recombines to form bonds which gradually form the NT. Growth of NTs is initiated at the precise spot where the catalyst is deposited and the diameter of NTs is determined by that of the catalyst particle [1].

CNT have been realized at the Ecole Polytechnique Federale de Lausanne (EPFL) in Switzerland. Nanotubes are grown with 100 A, 20 V dc arc between a 6.5 mm diameter graphite anode and a 20 mm graphite cathode in a 500 torr He atmosphere for 20 min. Nanotubes were found on the cathode, where they were encapsulated in a cylindrical shell 1 cm long [2].

Nanotubes have been characterized by Transmission Electron Microscopy, with a TopCon microscope at 120 kV and 200kV, see figure 1. In agreement with what observed by Chui et al. [3] and as clearly shown in the pictures, AD-CNT and CVD-BNNT present a very high structural purity with no evident defect in a spatial region attending the microns range, comparable to the telescopic length of the experiments. No clear difference is present between CNT and BNNT used during the experiments.
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**Figure 1:** Ultra-high-resolution TEM images of Nanotubes: a) and b) Carbon Nanotube; C) and D) Boron Nitride Nanotube. Picture in d) is courtesy of Arnaud Brioude [1].
Experimental Methods

Before any manipulation, the nanotubes are prepared by gluing them individually on a tungsten tip electrochemically etched. BNNT are first culled under optical visualisation and beforehand glued with carbon tape; bonding is then reinforced before manipulation by electron beam induced deposition (EBID). CNT are directly select under SEM visualisation and glued by EBID.

A tungsten tip etched as described in [4] is glued with conductive epoxy glued on one arm of the tuning fork (TF) as used in scanning probe microscopy. On the sample holder of the SEM, the TF and the prepared nanotubes are put face to face on a dedicated nanomanipulation station. While the TF is place on the motionless part, the tip-nanotube is mounted on the X-Y-Z step motors (Attocube N51xyz). A first step consist in bring together the free part of the nanotube and the tip apex of the TF-tip. Then nanostructure and TF tip are combined using local EBID. As a piezo excitator excites the oscillator at his own resonance frequency ($\approx 32kHz$), a lock-in and a phase locked loop (Specs Nanonis) maintain constant the amplitude of oscillation and the phase between the tuning fork and the excitator respectively. In order to stretch the strucure and measure properly the displacement, the stretching direction of the nanomanipulator is used in continuous mode providing displacement with sub-nanometer resolution. During the displacement (speed 25 nm s$^{-1}$) both the position of the mobile tip, recorded through the voltage applied, and the signals provided from the TF, usually excited by means of piezoelectric dithering allowing a natural mechanical excitation, are simultaneously recorded.

Post mortem characterization of the nanotubes

After the tensile load experiment, nanotubes have been imaged with Transmission Electron Microscope working at 200 kV. Intershell diameter have been obtained by the TEM pictures. Only the nanotube that have been glued on the tungsten wire can be imaged because of the dimension of the tuning fork and the access room inside the microscope column. In figure 2, we report TEM pictures of the nanotubes whose data have been presented in the main text.
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**Figure 2: Post mortem images of Nanotubes:** (A) BNNT1, intershell radius $R=25$ nm; (B) BNNT2, intershell radius $R=25$ nm; (C) BNNT1, intershell radius $R=10$ nm; (D) CNT, intershell radius $R=12$ nm.

Science and Technology B: Microelectronics and Nanometer structures, 9, 601 (1991);