AFM investigation of carbon nanotubes

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Purpose of investigation

Carbon nanotubes were discovered by high resolution transmission electron microscopy (HRTEM) in 1991 by Iijima (Nature 354 (1991) 56) [1].

A carbon nanotube is constituted from one (single wall nanotube) or several (multi-wall nanotube) atomic planes with a graphite-like arrangement of C atoms, which are rolled into a cylinder (see Fig. 2).

A multi-wall carbon nanotube may contain between 2 and 30 concentric layers of graphite; the outer diameter can range anywhere from 10-50nm while the length can reach more than 10 microns. A single wall nanotube can have the diameter between 1 and 1.4nm and length up to 100um.

Carbon nanotubes have unique electric and mechanical properties that make their investigation one of the key trends in different fields of science and technology. They may have metallic or semiconducting behavior depending on the way the graphite sheet is rolled to form the tube [2].

Some recent experimental results showed the possibility to develop nano-sized electronic devices based on carbon nanotubes. However, to make technological applications of carbon nanotubes possible, it is just essential to find such tool that can accurately characterize the properties of individual nanotubes.

The purpose of the current work is the following:

- To demonstrate that atomic force microscopy can be considered as one of the most appropriate characterization tool for investigation of carbon nanotubes properties;
- To outline the capability of AFM for such kind of investigation;
- To offer carbon nanotubes deposited on a quasi-plane substrate (e.g. HOPG, gold) as a test sample for AFM.

Sample preparation

To minimize a number of nonstable nanostructured compounds such as C-C complexes, graphite planes and amorphous carbon, the original carbon nanotubes had been thermally annealed for several times. As a result, we obtained the composition with the increased number of nanotubes, which was poured on a clean and smooth surface of highly oriented pyrolytic graphite (HOPG). The advantage of using HOPG as a substrate is that it increases the nanotubes adhesion and at the same time is a well-known test structure for atomic resolution imaging.

Measuring techniques

For our investigation of carbon nanotubes we used the Solver P47 Scanning Probe Microscope (NT-MDT). All measurements were done in ambient environment at the room temperature. The measuring head with the cantilever displacement detection system was installed on the corresponding piezotube scanner. Moreover, to achieve a better vibration isolation of the probe and substrate during the scanning process, the active vibration isolation table (Halcyonics, Germany) and acoustic hood were additionally used.

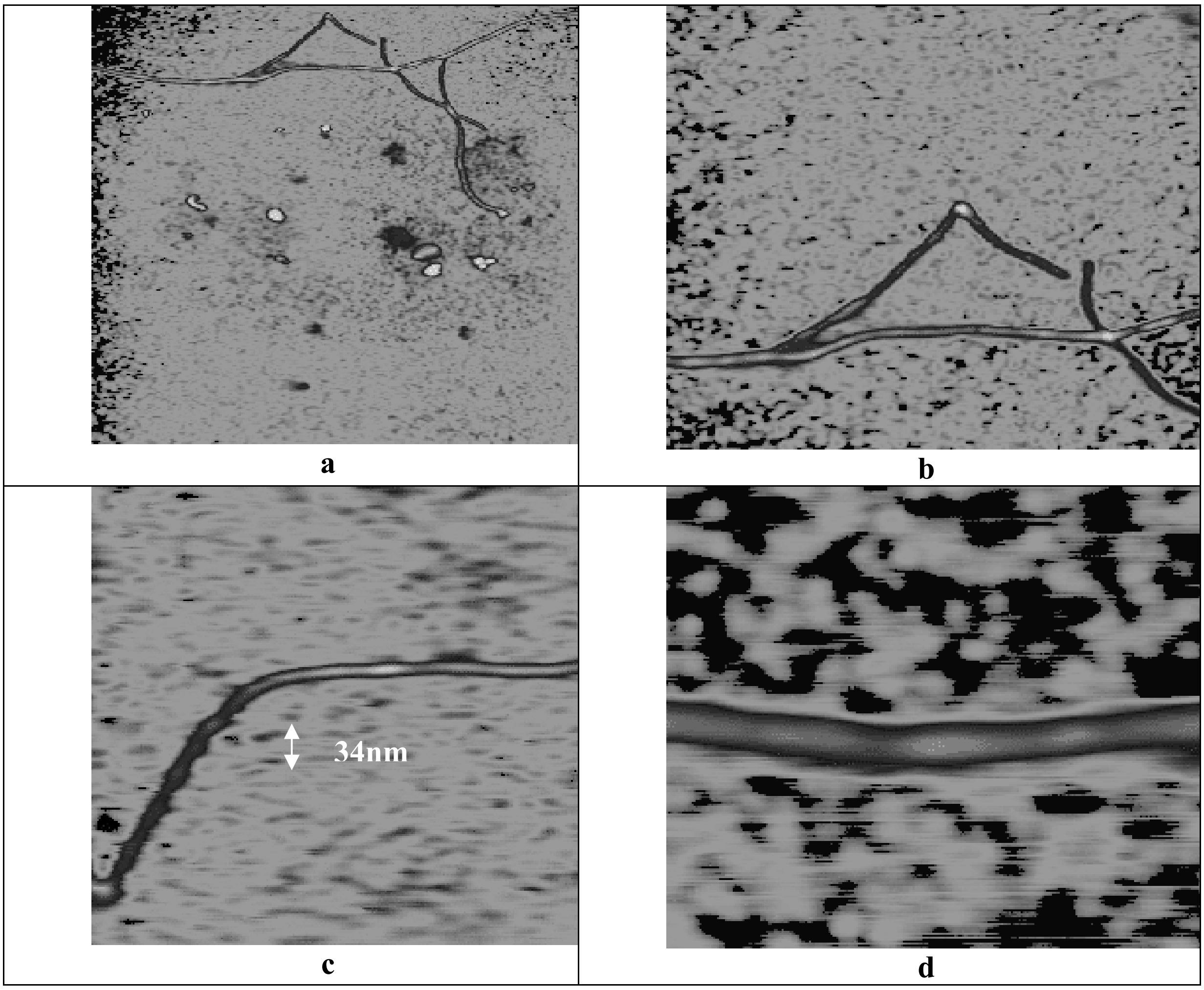
For topography imaging of carbon nanotubes placed on a HOPG substrate, the semicontact AFM technique was selected. The reason of our selection was that this technique shown a quite obvious advantage against the contact AFM if any poorly fixed nanometer sized objects like carbon nanotubes was measured by AFM. This is because the tip-sample interaction force is few times less than in the contact AFM mode.

Cantilever selection

In the semicontact AFM mode silicon cantilevers NSG10S (NT-MDT) were used. The typical resonance frequency of such cantilevers is 225kHz, force constant 11.5H/m, and the tip curvature radius 10nm. It is important to understand that the smaller tip curvature radius is the better results can be achieved. To make sure our tip curvature radius is small enough, we used its image obtained by scanning electron microscope.

Results

A lot of AFM topography images of carbon nanotubes were obtained on Solver P47. The illustrative example of physical zooming of the individual nanotube is shown on Fig. 3.



a

b

c

d

Fig. 3. Semicontact AFM topography images of the individual carbon nanotube. (a) – scan size 4x3um, average height 6nm; (b) – scan size 2x1,4um, average height 5nm; (c) – scan size 1,2x1nm, average height 3nm; (d) – scan size 500x400nm, average height 5nm.

As the initial approximation the tip shape can be estimated by its curvature radius **R**. Our experimental results shown that **R** is few times bigger than the nanotube typical diameter. In this case an individual nanotube is scanned across, the following estimation can be used: $\mathbf{R} = \mathbf{L}^2/8\mathbf{H}$, where **L** is a cross-section length of the scanned nanotube, **H** is a cross-section height of the scanned nanotube [3]. As shown on Fig. 3, the tip curvature radius is about

10nm as per the initial approximation, which points to the high quality of the used cantilever tip. Furthermore, it was found that the exact 3D upper estimation of the tip shape can be reconstructed from AFM image using the “blind reconstruction” method [4].

Therefore, carbon nanotubes uniformly deposited on a substrate with the sufficient concentration can be used as a test structure for cantilever tip shape estimation.

The possibility to manipulate the nanometer sized objects, such as carbon nanotubes, as well as to modify surface structure and properties (nanolithography) in air and liquid environment significantly expands the standard AFM capability and its scientific application. The example of carbon nanotube nanomanipulation is shown on Fig. 5.

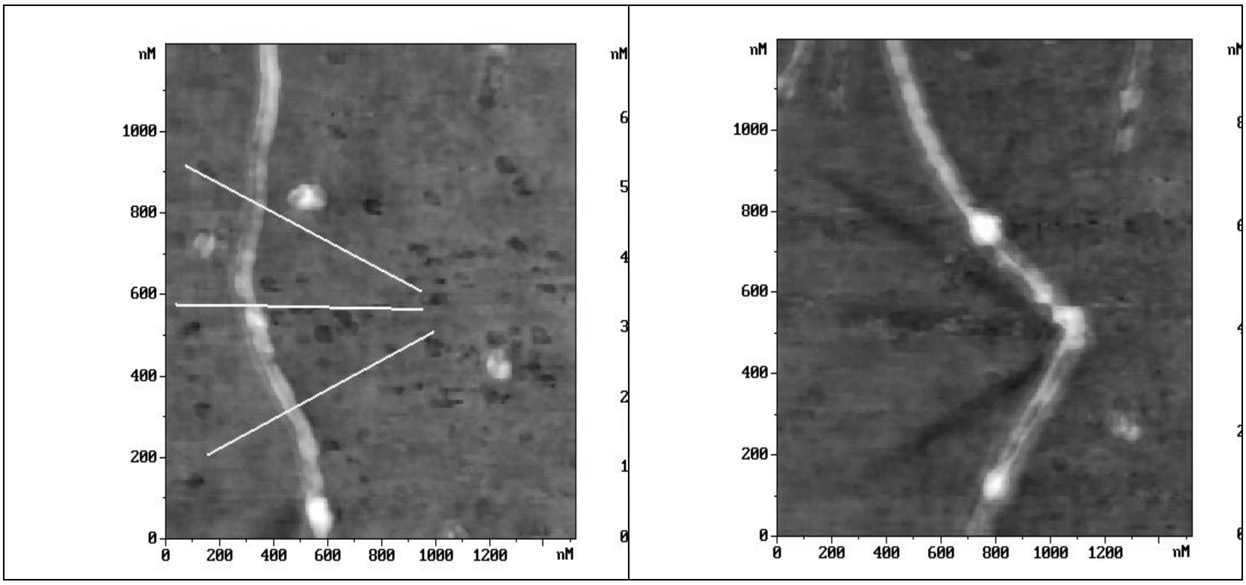


Fig. 5. Nanomanipulation of the carbon nanotube (AFM image, scan size: 1,5x1,2um)

In conclusion, AFM is a powerful tool for the investigation of carbon nanotubes properties and development of nanometer sized electronic devices. However, the interpretation of AFM data should be seriously considered and new metrological methods are to be developed.

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