

AFM Tapping Mode Phase-Imaging of Carbon Nanotubes

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Abstract: In the meantime AFM-imaging of carbon nanotubes has become a usual tool to get detailed information about the samples. This work introduces some new aspects in Tapping Mode Phase Imaging that may support the proper identification of SWNT samples.

For most applications one can think about employing Single Walled Carbon Nano Tubes (SWNTs) it is necessary to manipulate them in stable suspensions at high weight fractions with defined length scale (1-13). There exist different techniques for solution phase separation chemical derivatization, controlled dispersion, high volume fraction suspensions, self assembly in complex fluids and even fiber- or composit production (14-28). But because of high Van der Waals interactions, it is not easy to separate single SWNT's (29) to analyse them properly with surface analytical methods. Islam et al. and others (30) showed that it is possible to separate single tube fragments in Dispersion and that Scanning Force Microscopy AFM can be a helpful tool to get detailed information about diameter's length dimensions and aggregation of SWNT's. In this work we report the AFM analysis of differently produced SWNT's. The AFM experiments were carried out at a Nanoscope Multimode AFM with IIIA controller in Tapping mode. Micromesh NSC15 cantilevers were used at setpoints between 0,6 and 0,8. The tip sharpness of about 5 nm - 15 nm tip radius and the used 130 μm scanner lead to a maximum resolution in x and y of about 25 nm (see beyond). The SWNT's were either spin coated from bucky paper solutions on native oxide covered silicon surfaces or CVD grown on native oxide covered silicon surfaces which were doped with catalyst particles before. In both cases it turned out to be difficult to obtain clear information about size and shape of the SWNTs. Analysing the first kind one has to overcome the influence of buffer material which has to be used for the separation and the spinning process. As Islam (30) demonstrated one can overcome this problem partly with heating. In case of the other samples even after applying cleaning procedures there are a lot of rests of the CVD process which contaminate the cantilevers or give misleading AFM information. During the described investigations it turned out that especially the phase information can give good additional information about the nanotube material, because the contrast between SWNTs and the other material is rather high in the phase images.

Fig 1. shows an overview image (Scansize 10 μm) of a sample which was made from a "bucky paper" solution. The height information in the right picture shows three or four small wire

like structures that are carbon nanotubes. From the height image alone it is not clear if this all is carbon nanotube material. Comparing the height information with the corresponding phase image one can find out that all wires have the same height of about 2 nm and show the same phase contrast of about 8 degrees.

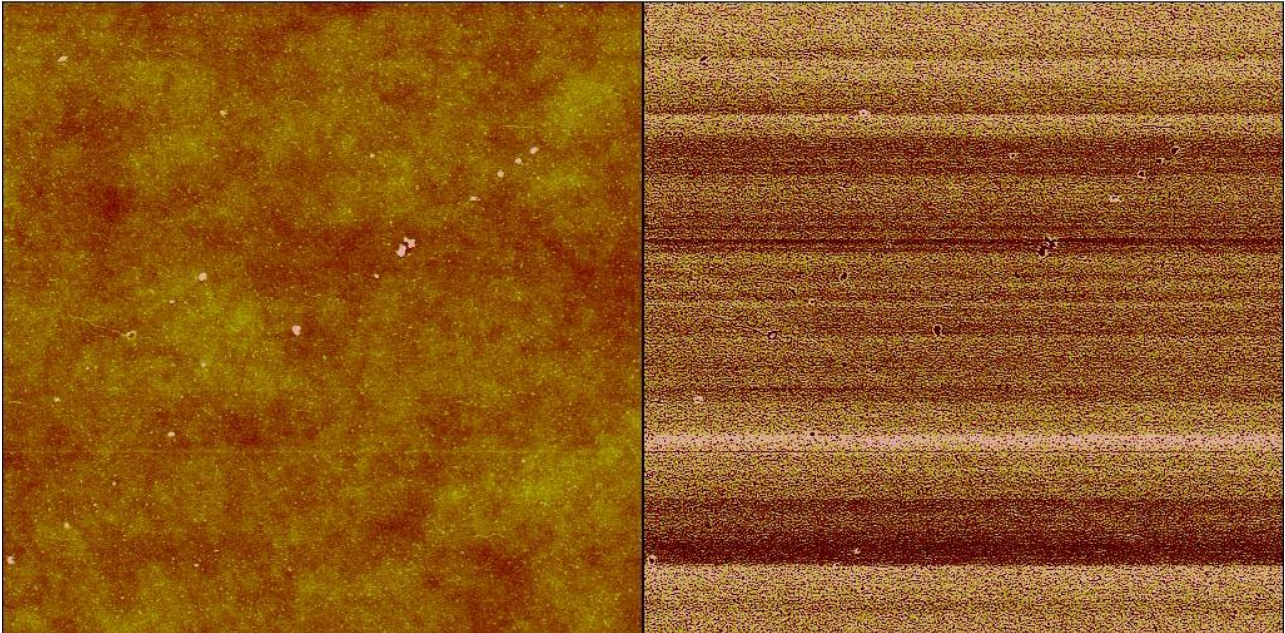


Fig.1 AFM Tapping Mode image (left) and corresponding Phase image (right) of a solution of carbon nanotubes spincoated on a silicon surface; scan size 10 μm x 10 μm ; z-scale 10 nm, phase angle 10 degrees; phase information at the position of the wire like structures in the height image shows the same strong contrast for all wires.

Which means that the interaction between tip and wire at all wires shows the same change in the phase of the cantilever vibration. From the thickness and the phase contrast together one can conclude that the wires in the image are SWNT material. Of course a lot of other not wire like structures in the phase image show the same contrast. This could be also nanotube material which is partly hidden in the buffer material of the coated layer or which are single or agglomerated shorter pieces of SWNTs. Because of the treatment during the preparation of the tube solution it is reasonable that there are a lot of short pieces of SWNTs which can not be imaged as wires anymore because their length is in the range of the tip radius (25 nm). Because of the mathematic folding of the tip shape and the sample information such parts can be seen as islands only. This is supported by Fig 2 where a lot of Islands can be seen in the phase image. At the bottom of the image one longer tube segment can be identified. The topography image shows some island structures along the wire which can be small SWNT parts or contaminations from the solution material. Therefore the corresponding phase information along the wire shows thicker and thinner passages. But still a constant phase shift can be seen along the wire. This obviously results from the same Tip sample

interaction at this passage of the image. For this sample finally one can assume that phase imaging gives helpful arguments for the identification of SWNT material.

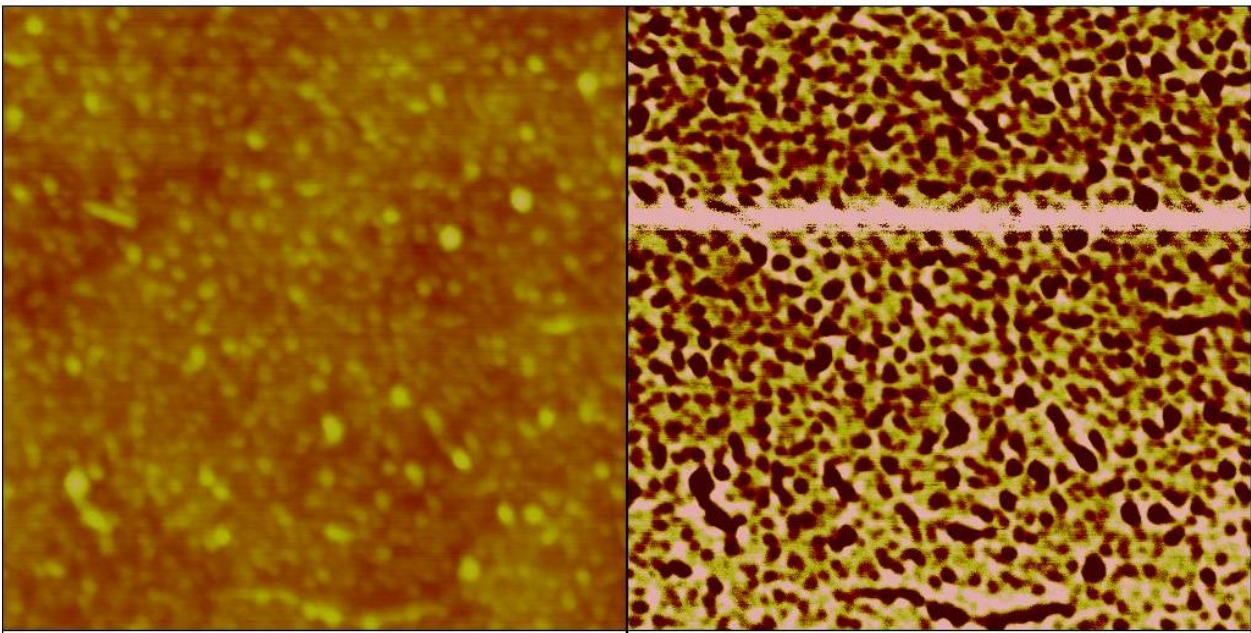


Fig. 2. AFM Tapping Mode image (left) and corresponding Phase image (right) of a solution of carbon nanotubes spincoated on a silicon surface; scan size $1\ \mu\text{m} \times 1\ \mu\text{m}$; z-scale 10 nm, phase angle 10 degrees; Comparing topography with phase information at least five partly contaminated segments of SWNTs can be identified.

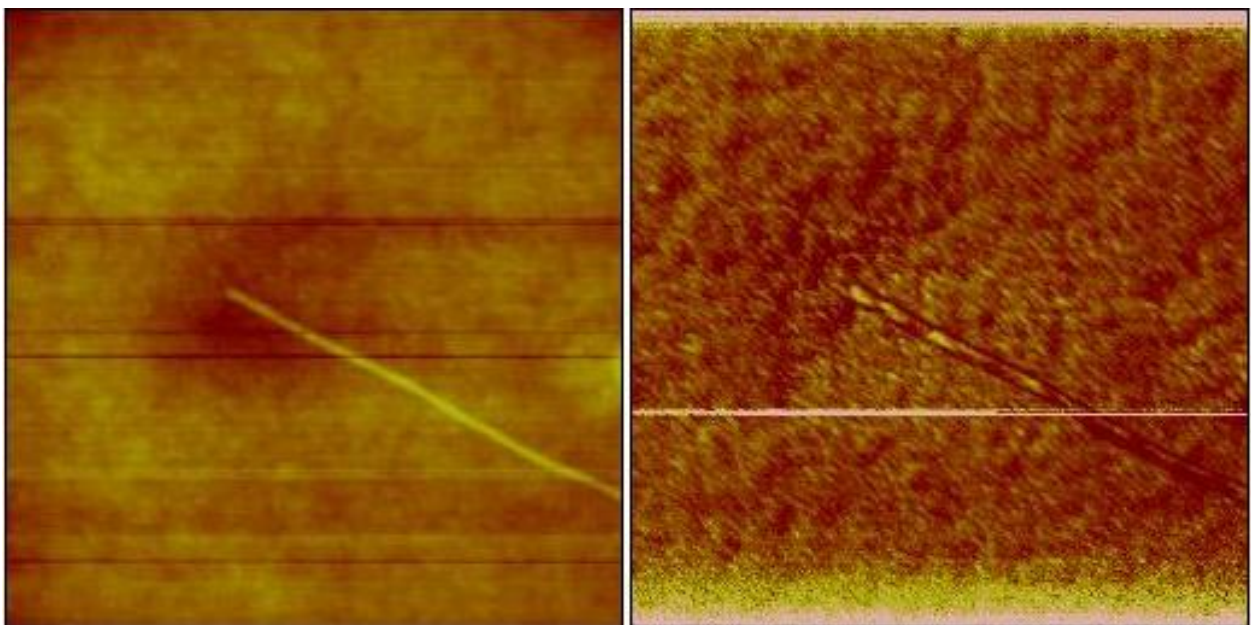


Fig. 3. AFM Tapping Mode image (left) and corresponding Phase image (right) of a SWNT, which was CVD-grown on a silicon surface which was doped with catalyst particles; scan size $1\ \mu\text{m} \times 1\ \mu\text{m}$; z-scale 10 nm, phase angle 10 degrees; Images show the beginning/end of the SWNT; in the phase image the tube is clearly surrounded by a dark line;

Looking at completely different prepared SWNT samples, which were made with CVD on with catalyst islands doped silicon surfaces one can find similar behaviour of the imaging of carbon nanotubes which supports the upper described results. With this method one is able to achieve single already separately growing SWNT's on the sample. In the phase image of Fig. 3 one can clearly see that the imaged tube is surrounded by a dark line, which is caused by a dramatic phase shift because of the changing tip tube interaction at the sides of the tube.

So in both cases the similar effects lead to contrast enhancement of the AFM Phase information. This way phase imaging can be a powerful tool to analyse SWNT Samples. Only very short pieces (length < 25 nm) cannot be imaged properly.

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