application note

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Imaging of Carbon Nanotubes Combining Confocal Raman and Atomic Force Microscopy

Carbon Nanotubes are unique nanostructures with remarkable mechanical and electrical properties. Due to their tremendous potential for future innovations, great efforts are made to characterize these structures.

In this study, carbon nanotubes were investigated with Confocal Raman Microscopy and Atomic Force Microscopy using only one single instrument.

Carbon Nanotubes on Silicon

An image was obtained by using the Raman Spectral Imaging Mode of the CRM200, which means that a complete spectrum was acquired at every pixel. The image was generated by evaluating the intensity of all Raman spectra. After the measurement, the spectrum at each pixel can be displayed. Fig. 1 shows a 100 x 100 micron area (200 x 200 pixels), including 40 000 spectra (acquisition time: 100 ms per spectrum, laser power: 100 mW @ 532 nm). In this sample the nanotubes are deposited onto a specially treated silicon substrate, which forces the nanotubes to assemble in rows. The regular and easily observable arrangement is illustrated with the corresponding spectra as designated by the arrows: Fig. 2 shows the Raman lines at 1600/cm and 2690/cm which are characteristic for the carbon nanotubes and fig. 3 is the spectrum of silica with the two Raman lines at 520/cm and 950/cm.

By simply rotating the objective turret of the microscope, AFM measurements can be performed on the same sample without touching it.
Fig. 4 shows an overview of the sample with a scan range of 20 µm x 20 µm and 256 x 256 pixels. The image was obtained at 1.5 sec/line. The images in fig. 5 and fig. 6 are zoom-ins of 5 µm x 5 µm and 1.5 µm x 1.5 µm, respectively.
The size of a single tube measures between 15 nm and 60 nm.

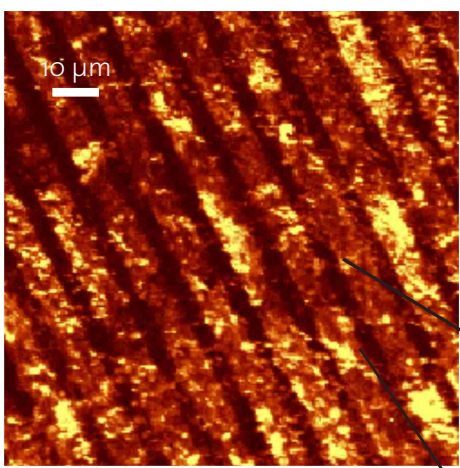

Fig. 1: Image of the integral intensity of all Raman lines: Carbon nanotubes assembled in rows with corresponding Raman spectra.
Scan range: 100 µm x 100 µm

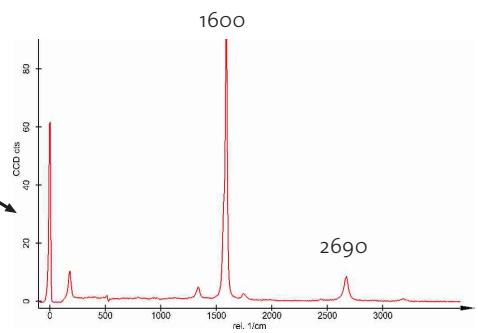

Fig. 2: Spectrum of carbon nanotubes

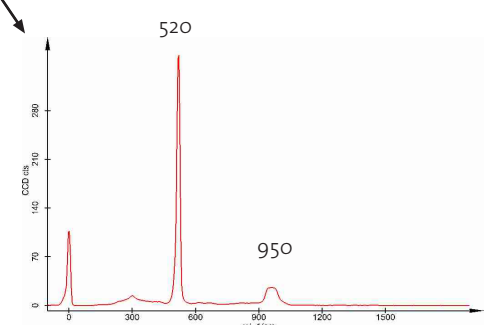

Fig. 3: Spectrum of Silica

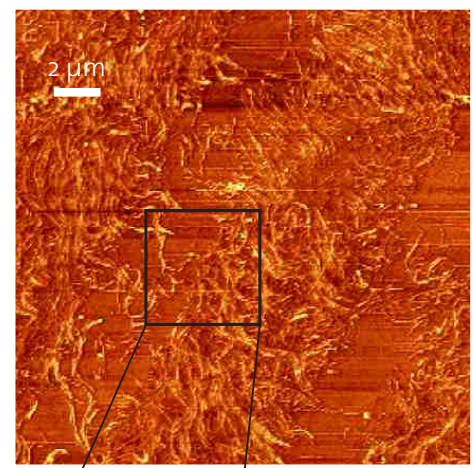

Fig. 4: AFM measurement, 20 µm x 20 µm

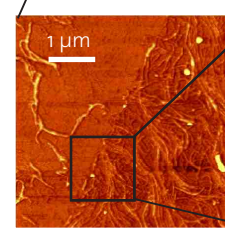

Fig. 5: Zoom-in, 5 µm x 5 µm

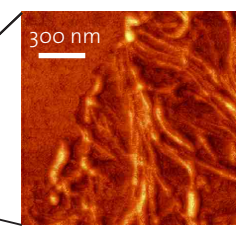

Fig. 6: Zoom-in, 1,5 µm x 1,5 µm

Samples courtesy of Prof. Tsukruk, Iowa State University, USA

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Individual Bundles of Single Wall Carbon Nanotubes

In the following study, laser vaporization-grown single-wall carbon nanotubes (SWNT) produced at Oak Ridge National Laboratory are imaged. SWNTs were deposited on a Si substrate using a spin-coating technique. Fig. 7 shows an AFM measurement with a scan size of 14 µm x 14 µm and 256 x 256 pixels. Fig. 8 shows the corresponding measurement in the Spectral Imaging Mode of the CRM200 on the same sample position. A complete spectrum is obtained at every pixel. Scan range also 14 µm x 14 µm with 150 x 150 pixels (= 22500 spectra) and an integration time of 50 ms. The image was obtained by integrating over all Raman lines. Using both images the specrtal data can be clearly linked to dedicated nanotubes observed in the AFM image.
The orientation of the tubes can be determined by measuring the intensity of the Raman spectrum depending on the polarization. The signal is always strongest when the laser light is polarized along the nanotube axis. Therefore two measurements were performed, one initial measurement, and another with the sample rotated by 90 degrees (compare fig. 9 and fig. 10). Depending on the polarization of the incident light, different carbon nanotubes are visible.
A detailed evaluation of the obtained spectral data of one carbon nanotube is shown in Fig. 11. Different Raman lines are visible. Each part of the spectrum, the radial breathing mode (RBM) at around 180/cm, the D-band at around 1330/cm, the G-band at around 1580/cm, second-order-modes and the G`-band can be used to differentiate properties of carbon nanotubes.

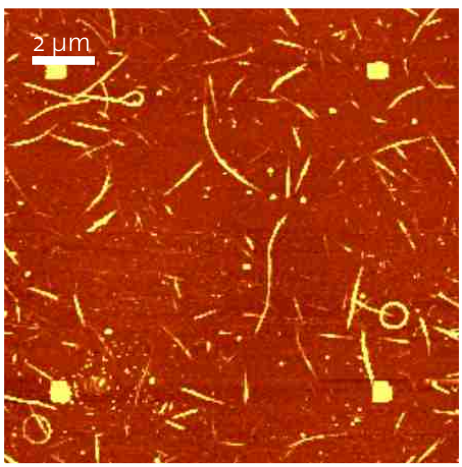

Fig. 7: AFM measurement, 14 µm x 14 µm

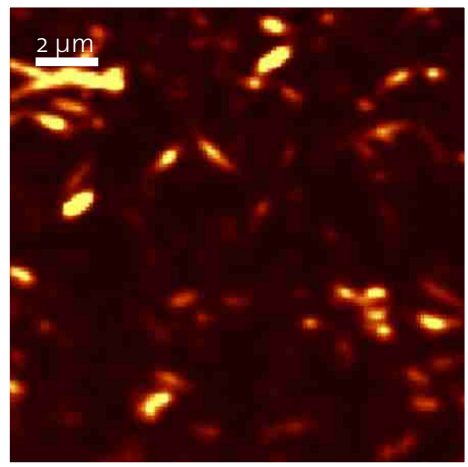

Fig. 8: Raman measurement, on the same sample position, 14 µm x 14 µm

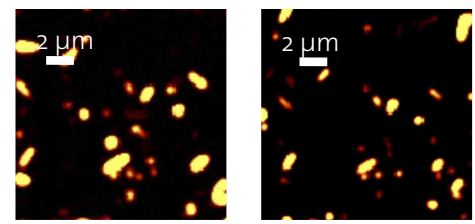

Fig. 9+10: Different carbon nanotubes are visible, depending on the polarisation of light. Polarisation plain in Fig. 10 is rotatet by 90 degrees.

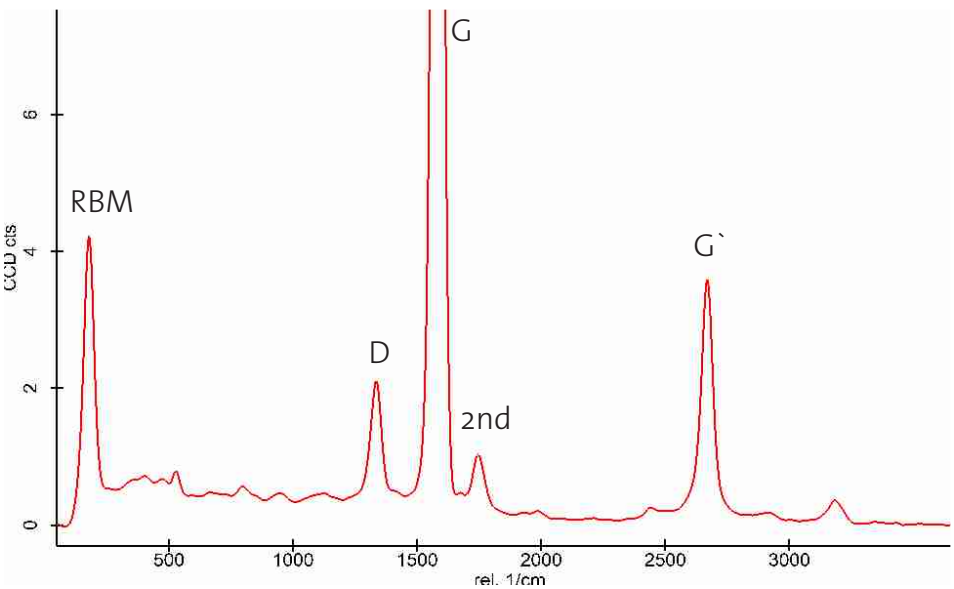

Fig. 11: Spectrum of carbon nanotubes.

Samples courtesy of Dr. D. B. Geohegan, Prof. A. A. Puretzky and Dr. D. W. Austin, Oak Ridge National Laboratory, USA