Characterization of Multi-Component Materials on the Sub-Micrometer Scale
A Topographic 3D Raman and AFM Study

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Knowledge about the morphology and chemical composition of heterogeneous materials on a sub-micrometer scale is crucial for the development of new material properties for highly specified applications. Such materials can either have mono-atomic flat surfaces or a roughness of several hundred micrometers or millimeter. In the past two decades, AFM (atomic force microscopy) was one of the main techniques used to characterize the morphology of nano-materials spread on nanometer-flat substrates. Fig. 1a shows the high spatial resolution AFM image of carbon nanotubes spread on a silicon wafer. From such images it is possible to gain information about the physical dimensions of the material on the nanometer scale, without additional information about their chemical composition, crystallinity or stress state. On the other hand, Raman spectroscopy is known to be used to unequivocally determine the chemical composition of a material. By combining the chemical sensitive Raman spectroscopy with high resolution confocal optical microscopy, the analyzed material volume can be reduced below 0.02 μm^3, thus leading to the ability to acquire Raman images with diffraction limited resolution from very flat surfaces [1, 2]. The combination of confocal Raman microscopy with Atomic Force Microscopy (AFM) is a breakthrough in microscopy. Using such a combination, the high spatial and topographical resolution obtained with an AFM can be directly linked to the chemical information provided by confocal Raman spectroscopy [3]. Fig. 1b shows the intensity distribution of the G-band obtained from the same sample position as in Fig. 1a using Raman imaging. In this imaging mode a complete Raman spectrum is acquired in every image pixel, leading to a 2D array of single Raman spectra. The evaluation of peak intensity and peak position results in different Raman images, representing various properties of the carbon nanotubes. The Raman spectrum of a SWCNT with its unique Raman bands is shown in Fig. 1 (right side). Characteristic for SWCNT only are the radial breathing modes (RBM), providing information about the diameter of the tube. The position and width of the G-band is used to distinguish between metallic (bright color in Fig. 1c) and semiconducting (bright color in Fig. 1d) CNT and to probe the charge transfer arising from doping CNT`s.

Topographic Raman imaging, a new imaging technique developed for measurements on rough surfaces over large areas, allows confocal Raman imaging guided by the surface topography obtained by an integrated profilometer. Large-area topographic coordinates from the profilometer measurements can be precisely correlated with the large area confocal Raman imaging data. This allows true surface Raman imaging on heavily inclined or rough surfaces, with the true sample surface held in constant focus, while maintaining highest confocality. Fig. 2a shows the topography of a pharmaceutical tablet on the order of several hundred micrometer. In Fig. 2b the true surface Raman image is presented, showing the distribution of the API (red) in the various excipients (green and blue color).

In summary, the combination of confocal Raman microscopy and topographic Raman imaging allows the characterization of materials at high, submicron resolution, as well as on mm-rough surfaces across large areas. Examples from various fields of applications will be presented.

References

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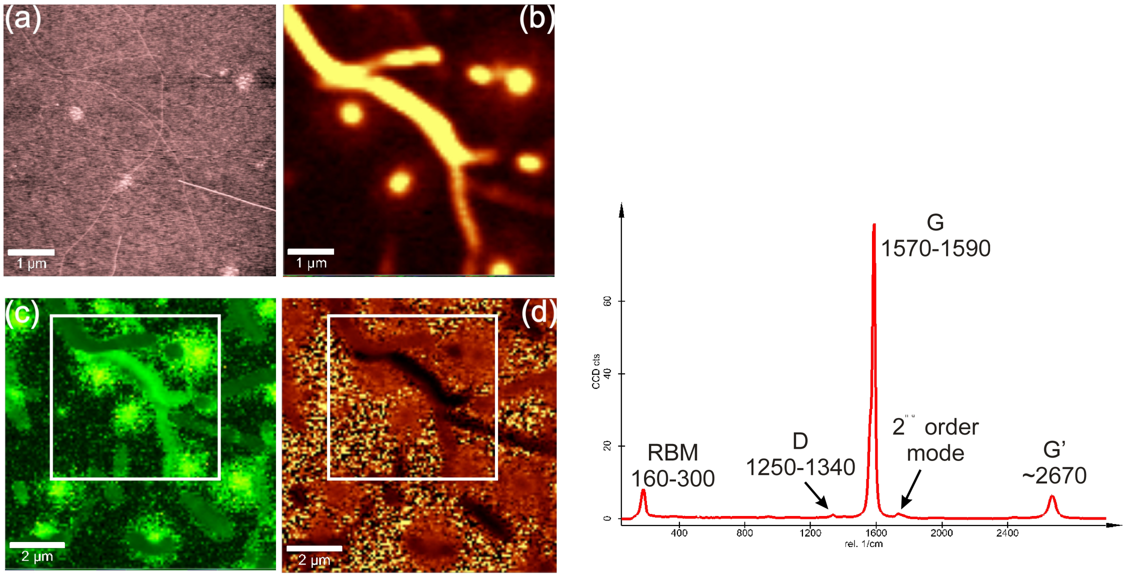

Figure 1: *AFM image of CNT`s spread on a Si substrate (a), confocal Raman image showing the intensity of the G-band, thus the distribution of CNT`s on the Si substrate (b), enlarged scansize confocal Raman images showing the width (c) and center of mass (d) of the G-band revealing crystalline defects and stress states of the different CNT`s.*
Right side: Characteristic Raman spectrum of a SWCNT.

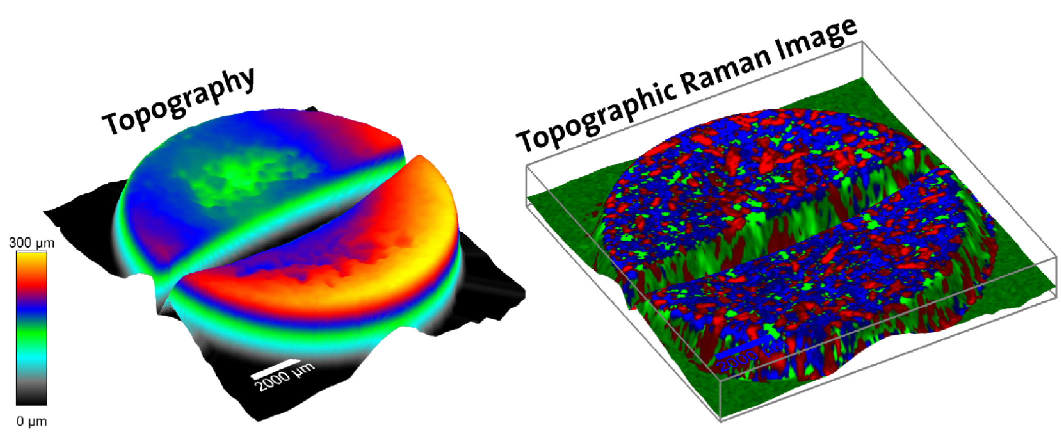

Figure 2: *Topography and True Surface Raman Image of a pharmaceutical tablet.*