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Material Sensitive Microscopy on the Nanometer Scale

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Scanning Probe Microscopy (SPM) introduced new possibilities for materials characterization on the nanometer scale. The primary advantage of SPM is its capability for the real scale 3D characterization of surfaces of materials. Especially surface defects can be imaged without averaging over hundreds of nanometers. No specialized sample preparation is required and samples can be analyzed in their natural environment.

By investigating the tip-sample interaction, one can obtain not only the high resolution topographic structure of the surface but also information about the local mechanical properties of the sample components. Figure 1 shows Digital Pulsed Force Mode (DPFM) images of polymer blends consisting of poly-methyl-methacrylate (PMMA) and styrene-butadiene (SB). The topography and stiffness maps of the PMMA-SBS blend are represented in Figure 1 A. In this case, the styrene-butadiene forms a triblock copolymer. The topography image shows the formation of a netlike PMMA structure filled with SBS. The simultaneously recorded stiffness map reveals a contrast of about 0.6 N/m where the stiffer material is the PMMA. Figure 1 B shows the topography and stiffness maps of the PMMA-SBR blend. The styrene-butadiene is a statistical copolymer in this blend. The topography image shows the formation of PMMA islands surrounded by SBR. The contrast in stiffness between PMMA and SBR, shown in the stiffness map, is approximately 2.5 N/m, indicating that the difference in the chemical structure of the styrene-butadiene results in different stiffness properties.

If the standard AFM cantilever is replaced by a micro-machined SNOM cantilever, not only is the imaging of topographic and mechanical contrast on surfaces available, but also optical images with resolution below the diffraction limit. Figure 2 shows the simultaneously recorded topography (A) and fluorescence SNOM (B) images of a two phase polymer blend used for LED applications. The excitation wavelength for the fluorescence images was 457 nm, whereas the detection was above 490 nm.

A more complete characterization of materials can be achieved if the chemical information is linked to the high resolution SPM images. This can be realized by combining extremely sensitive Raman spectroscopy with high resolution optical microscopy. Figure 3 shows an AFM image of nanotubes spread on a silicon substrate. The same sample area was imaged with the Confocal Raman Microscope (CRM). Raman spectra from various sample areas are shown in Figure 3 B, such as fluorescence from the lithographic marker (top spectrum), a characteristic nanotube peak at 2650/cm (middle spectrum), and silicon (lower spectrum). Figure 3 C, imaged in horizontal polarized light, shows that only the horizontal nanotube (marked as 1) is present in the CRM-image. By changing to vertical polarization, the “missing” nanotube 2 becomes visible. The nanotubes are visible only if the polarization axis is parallel to the nanotube.

The aim of this paper is to present not only examples of various material characterizations on the nanometer scale, but also to introduce the instrumentation required for such studies.

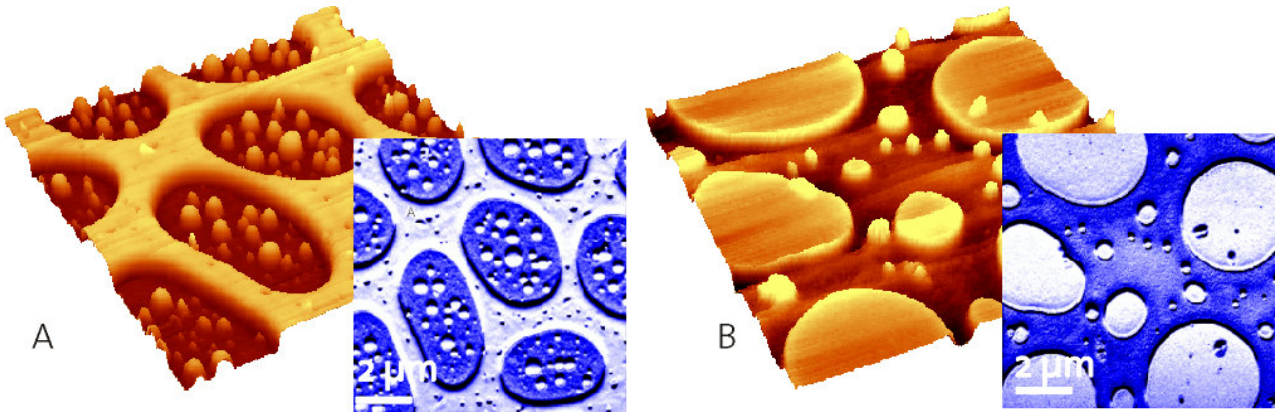

Fig. 1: DPFM images (topography and stiffness maps) of thin films of polymer blends: PMMA-SBS (A) and PMMA-SBR (B).

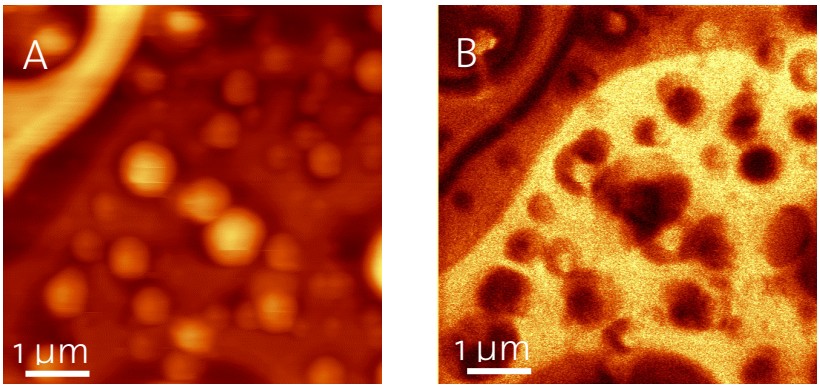

Fig. 2: Simultaneously recorded AFM image (A) and fluorescence SNOM image (B) of a two phase polymer blend used for LED applications. Excitation wavelength: 457 nm, detection above 490 nm.

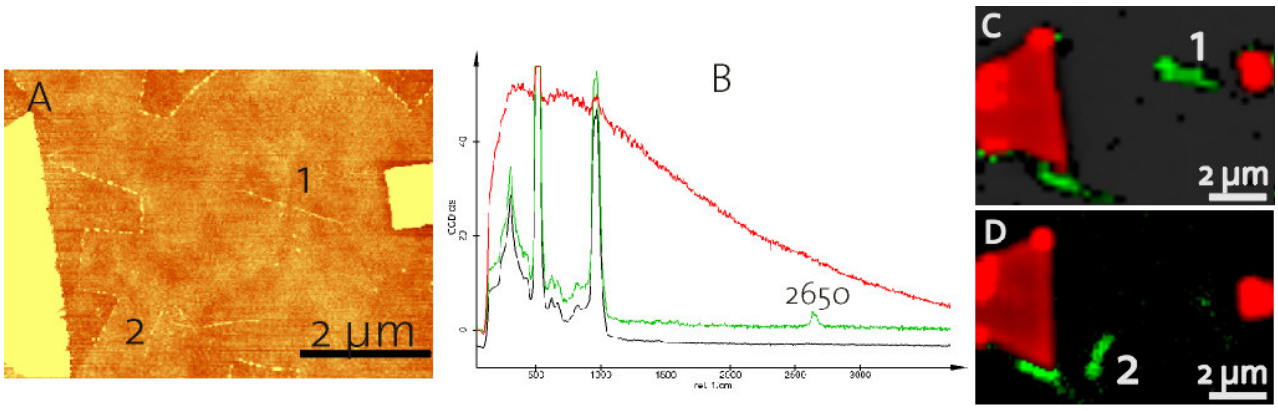

Fig. 3: Nanotubes spread on a silicon wafer: AFM topography image with two nanotubes marked as 1 and 2 (A), color-coded Raman spectra from various areas of the same sample: fluorescence – red, nanotube – blue, silicon – black (B), color coded Raman spectral images of the same area as (A) with horizontal (C) and vertical (D) polarized excitation. The nanotubes are only visible if the polarization axis is parallel to the nanotube.

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