application note

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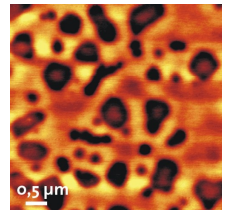
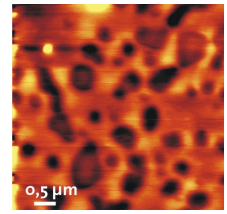
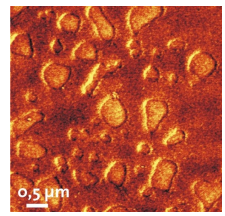
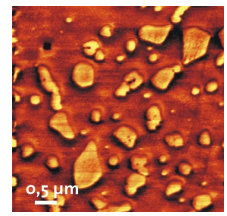
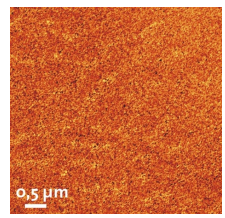
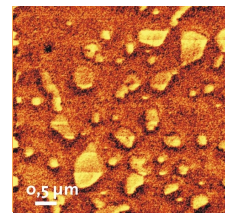
Atomic Force Microscopy - Temperature Dependent Properties of Polymers

Atomic force microscopy (AFM) is one of the most powerful methods available for surface characterisation. The forces which interact between tip and sample can be used to map surface topography on the nanometer scale. In addition, if equipped appropriate, an AFM is also able to detect material properties based on tip - sample interaction forces. The Digital Pulsed Force Mode allows imaging of all surface properties that can be extracted from pulsed force and force distance curves. For many technical applications, temperature dependent studies on such properties are crucial.

PS-PMMA blend

The combination of Digital Pulsed Force Mode AFM with a variable temperature sample stage is capable of imaging phase separations in polymer blends as a function of the glass transition temperature. The Digital Pulsed Force Mode evaluates and stores the complete force distance cycle, which allows a detailed analysis of the interactions between tip and sample. In these studies, not only the material properties of PS-PMMA (Polystyrol-Polymethylmetacrylat) blend were measured, but also their variations with temperature (25°C and 140° C; Scan range: 5x5 µm; Adhesion and cantilever oscillation damping images reconstructed from Pulsed Force Curves).

	25° C	140° C	
Topography	 Fig. 1	 Fig. 2	The topography image at 25° C (Fig. 1) is similar to the one at 140° C (Fig. 2). However the total height increases at 140° C indicating a swelling of the polymer film at elevated temperature. Scan range at 25° C (Fig. 1): 5x5 µm, z=20 nm; scan range at 140 ° C (Fig. 2): 5x5 µm, z=30 nm.
Adhesion	 Fig. 3	 Fig. 4	Large differences can be seen in the adhesion image. The variation of the adhesion at increasing temperature indicates, that the holes in the topographical picture correspond to PS, whereas the higher domains correspond to PMMA.
Damping of the cantilever free oscillation	 Fig. 5	 Fig. 6	These were recorded immediately after snap-off from contact with the sample, clearly showing the material contrast at elevated temperatures. At room temperature, a thin water layer is present on the sample masking material contrast. At 140° C a clear material contrast between PS and PMMA can be seen, because the water layer is no longer present.

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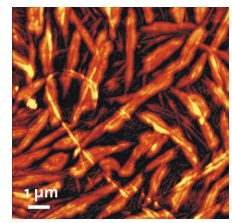
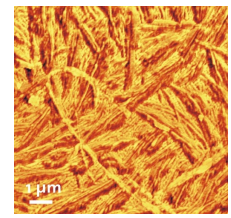
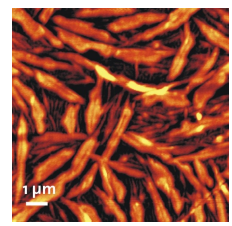
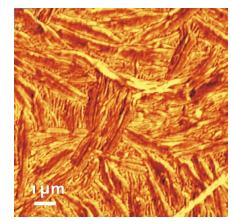
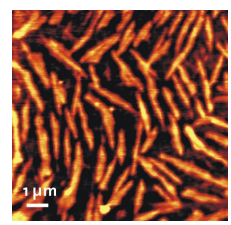
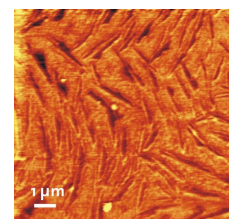
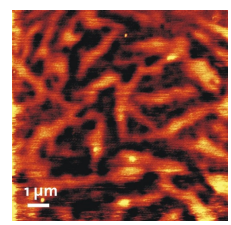
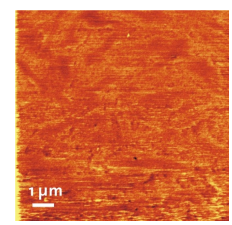
Paraffin

Thin paraffin films were deposited onto a silicon substrate. The variable-temperature sample stage allowed easy temperature-controlled measurements up to 200° C. Along with the topography, the adhesion of the paraffin was evaluated at increasing temperatures with the Digital Pulsed Force Mode.
(Scan range: 10x10 µm, 25°-90° C: z= 35 nm; 130° C: z= 20 nm)

At room temperature, there are two types of crystalline phases, with large crystals separated by finer structures. The adhesion image shows predominantly topographical features, due to larger tip-sample interaction surfaces.
Upon heating, the thin fine structures disappear first at about 100° C. The images taken at 120° C show mainly the presence of the large structures. They display lower adhesion (dark area in adhesion image) compared to the area where the fine structure was previously.
The melting of the large crystals becomesvisible at 130° C in the topographical image. They can be still recognized although the adhesion shows an almost uniform picture.

	Topography	Adhesion
25° C	 Fig. 7	 Fig. 8
90° C	 Fig. 9	 Fig. 10
120° C	 Fig. 11	 Fig. 12
130° C	 Fig. 13	 Fig. 14