AFM analysis of a PS-PB blend

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Sample and experiment description

- Polymer blend consisting of stiff polystyrene (PS) and soft polybutadiene (PB)

- PS:PB ratio: 1:1 (w/w) on silicon

- Unmixing and phase separation occurs upon evaporation of organic solvent during sample preparation.

- AFM modes used to characterize the sample:
  - Static mode (Nanosurf NaioAFM, PPP-ContSCR)
  - Dynamic mode phase imaging (Nanosurf NaioAFM, PPP-XYNCSTR)
  - Force spectroscopy (Nanosurf NaioAFM, PPP-XYNCSTR)
  - Lateral force (Nanosurf FlexAFM, Nanosurf Easyscan2 controller, PPP-ContSCR)
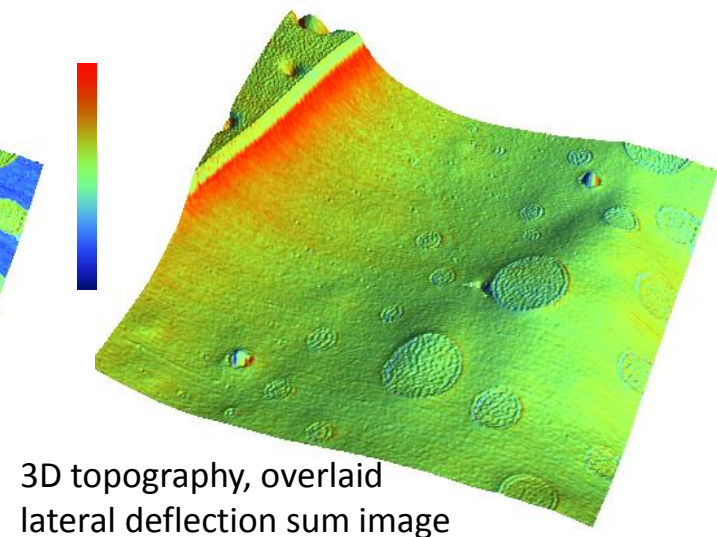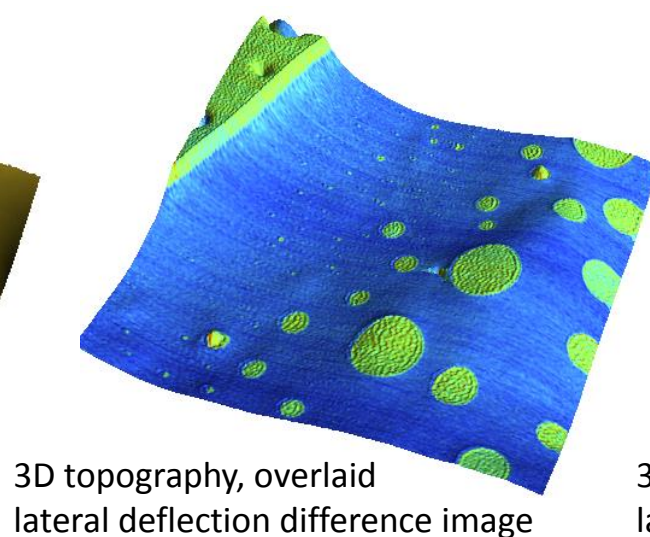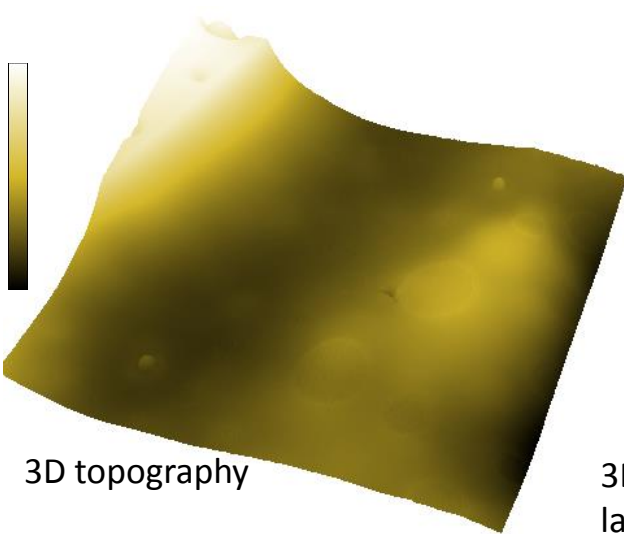
Static mode imaging

- PS islands of varying size in a PB matrix
- PS island protrude the PB matrix
- Image dimensions: 15 µm x 15 µm
- Height range 250 nm
- Measured with NaioAFM and PPP-ContSCR cantilever
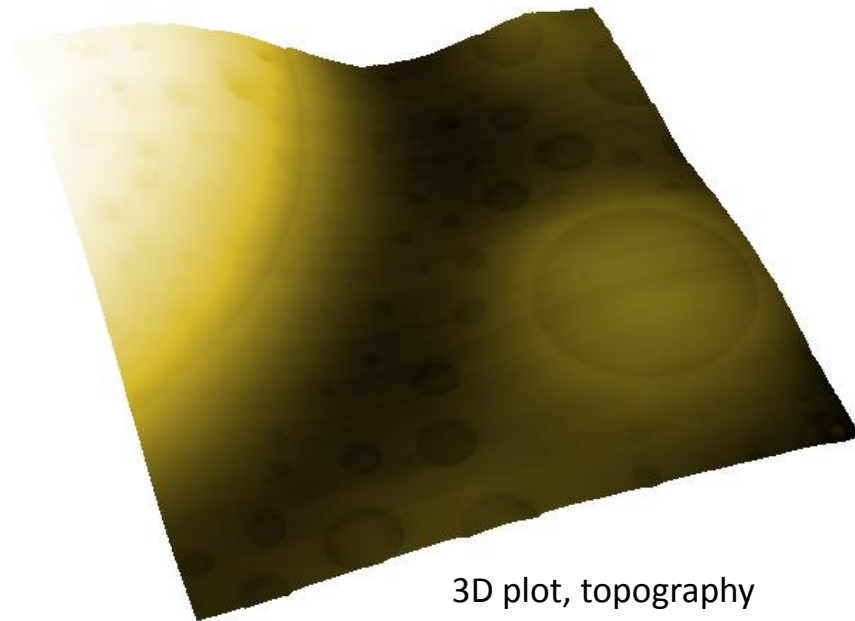
topography

3D plot, topography
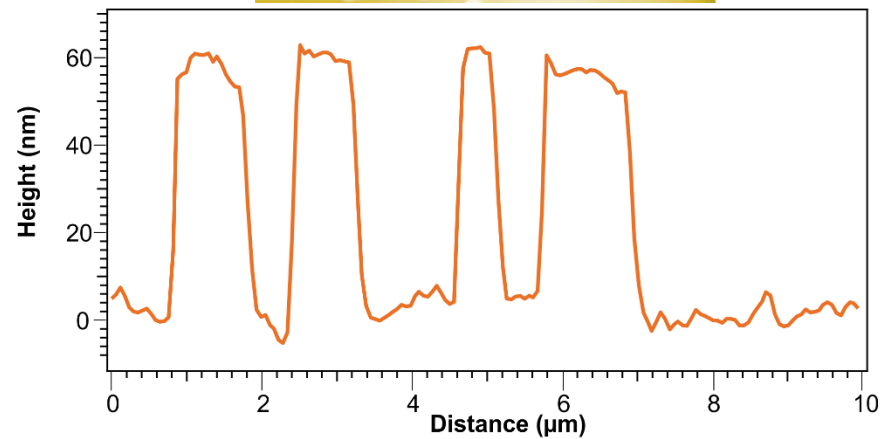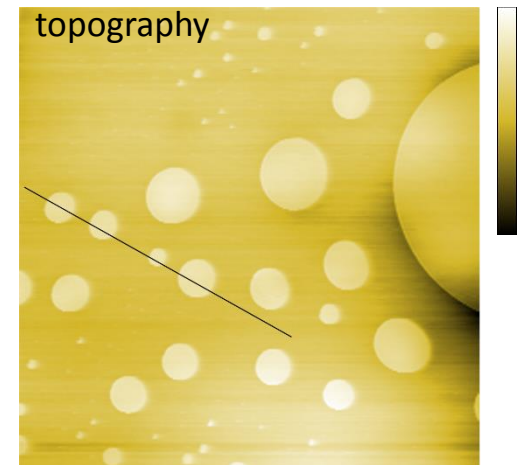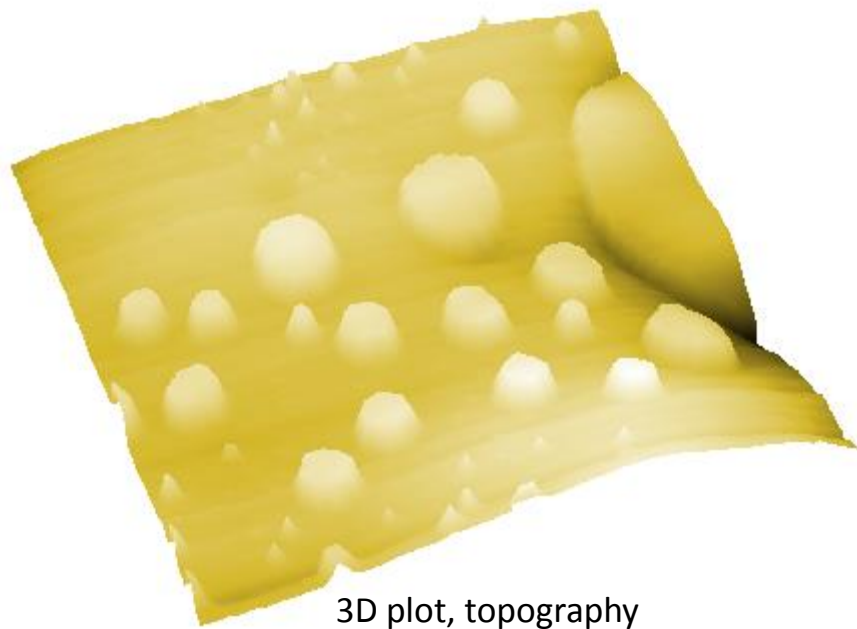
Lateral force mode imaging

- Lateral force mode imaging provides information about the friction of surface.
- During static mode imaging topography (left)) and lateral deflection are measured in parallel
- The difference image highlights friction characteristics (middle)
- The sum highlights topographic characteristics, especially steep edges (right)
- Measured with FlexAFM, ES2 controller, PPP-ContSCR cantielver
- Image dimensions: 9 µm x 9 µm
- Height range 350 nm; lateral deflection range: 40 mV

3D topography

3D topography, overlaid
lateral deflection difference image
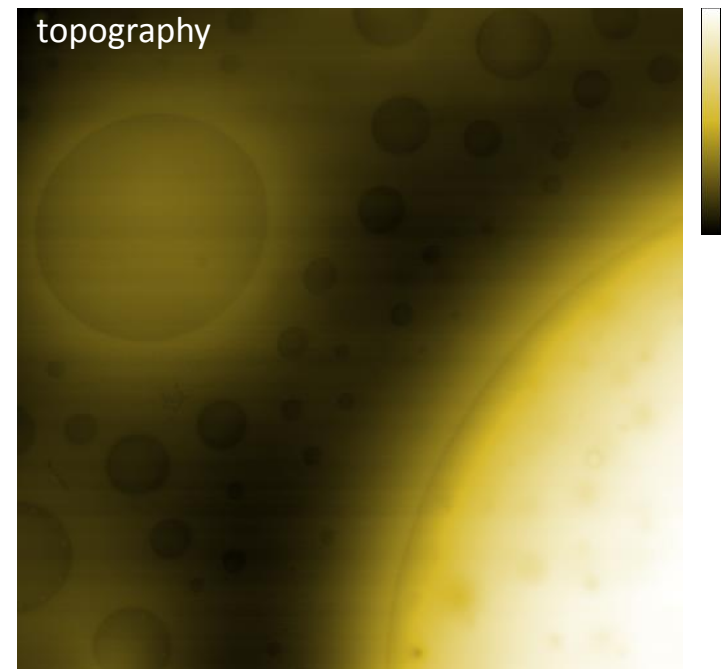
3D topography, overlaid
lateral deflection sum image

Dynamic mode imging

- Smaller PS islands are 10-30 nm below the level of the PB matrix.
- Different imaging modes probe surfaces differently; e.g. PS islands protrude PB in static mode
- Measured with NaioAFM and PPP-XYNCSTR cantilever
- Image dimensions: 20 µm x 20 µm
- Height range 560 nm

3D plot, topography
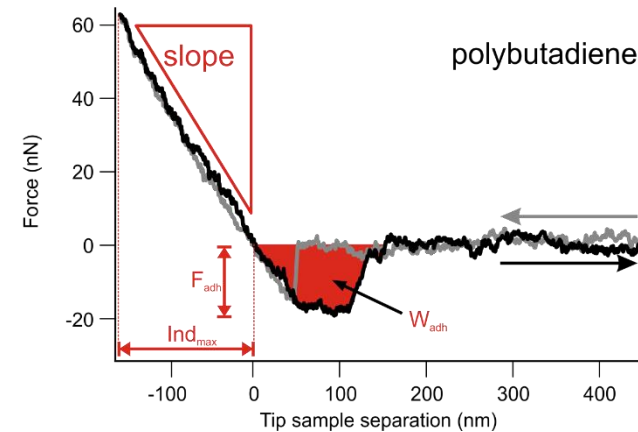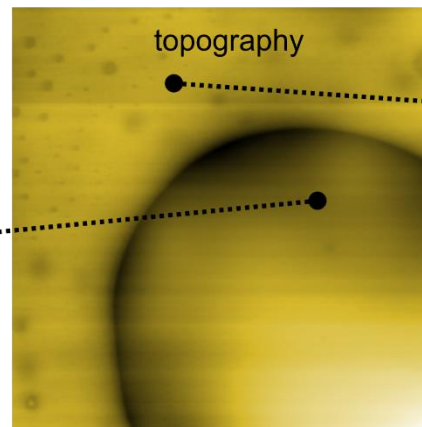

topography

Dynamic mode imaging with phase contrast

- Phase shift in dynamic mode reveals qualitative information about sample properties (e.g. stiffness or adhesion).
- Topogaphy (left) and phase (middle) are measured simultaneously.
- PS-PB blend shows well defined phase contrast (here ~10°) between the PS islands (dark) and PB matrix (bright)
- Topography and phase signal can be correlated (right) to identify specific regions
- Measured with NaioAFM, PPP-XYNCSTR cantielver
- Image dimensions: 20 μm x 20 μm
- Height range 560 nm; phase range 15°

3D topography with phase shift overlay
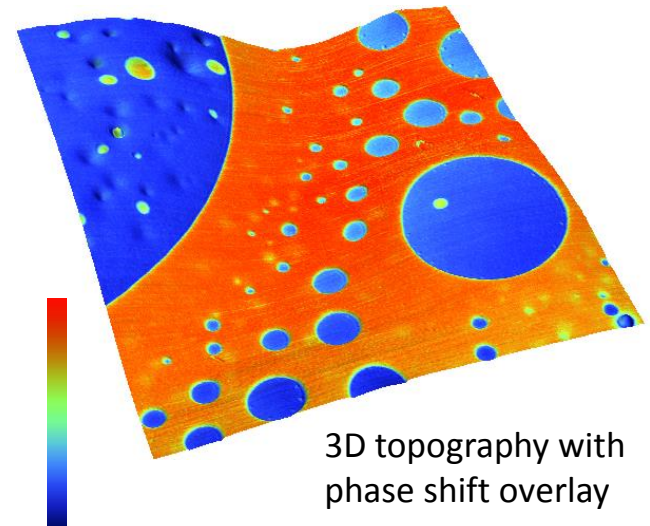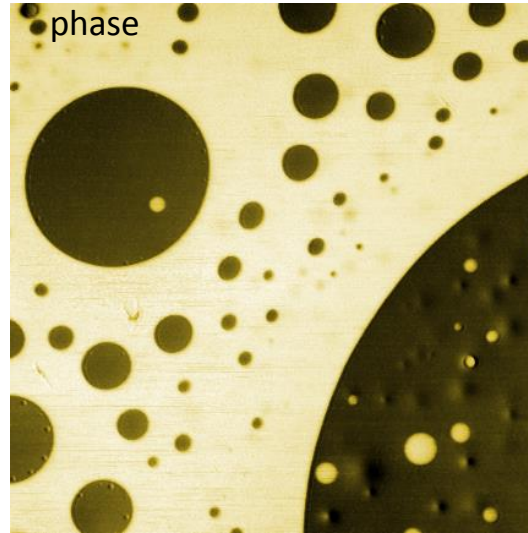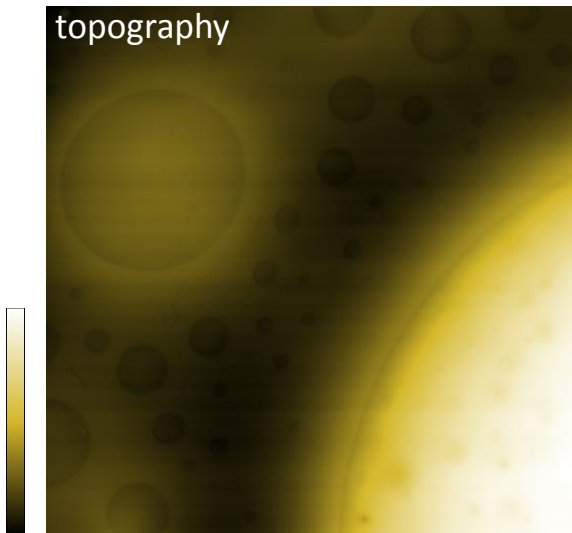
Force spectroscopy

- Force distance (FD) curves provide quantitative information about the components of the PS-PB blend
  - The slope of the contact part of the FD curve (red triangles) is a semiquantitative measure for the local sample stiffness.
  - The peak force during the backward movement provides information about the adhesive properties of the sample (F_{adh}).
  - The area under the adhesion peak is a measure for the irreversible work performed (W_{adh}).
  - The indentation into the sample at maximal loading force is indirectly also a measure for the stiffness.
- Measured with NaioAFM, PPP-XYNCSTR cantielver
- Image dimensions: 20 µm x 20 µm
- Height range 560 nm; force setpoint 60 nN

	Slope	F_{adh}	W_{adh}	Ind_{max}
PS	↑	↑	↓	↓
PB	↓	↓	↑	↑

polystyrene

topography

polybutadiene
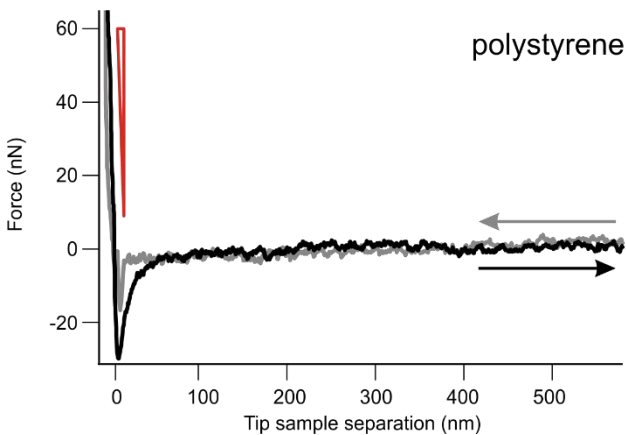
Force mapping

- Record an array of FD curves, e.g. after recording a topography image
- Correlate AFM topography (left) to parameters extracted from FD curve analysis.
- Image processing allows overlaying the topography with color-coded elastic modulus (middle) or max. adhesion (right) information.
- Measured with NaioAFM, PPP-XYNCSTR cantielver
- SPIP data analysis software
- Image dimensions: 15 µm x 15 µm
- Height range: 210 nm
- Elastic modulus range: 0-5 GPa, plotted on a logarithmic scale
- Max. adhesion force scale: 20-40 nN
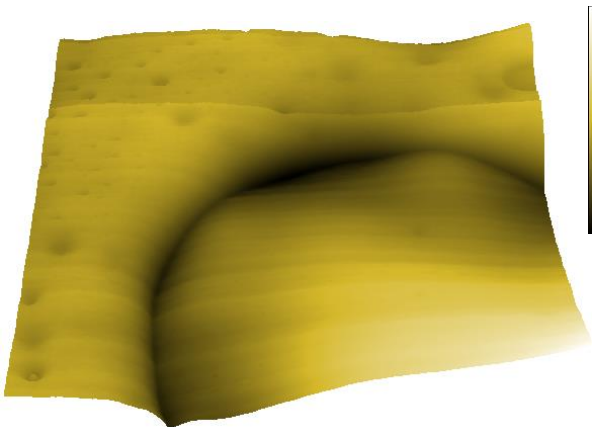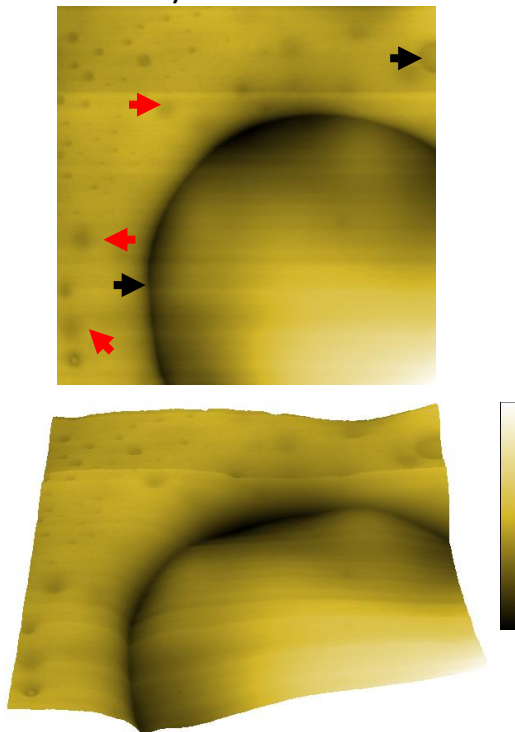

3D topography (dynamic mode)
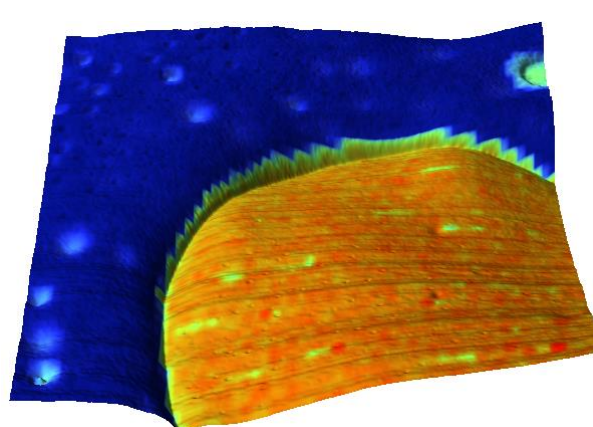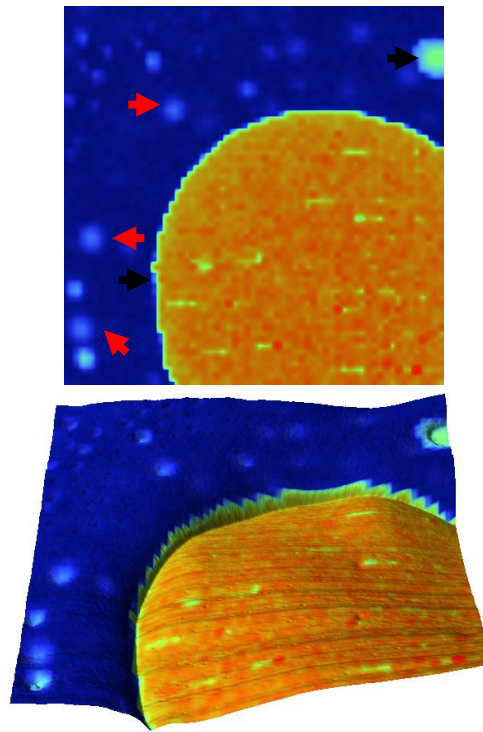
3D topography with elastic modulus overlay
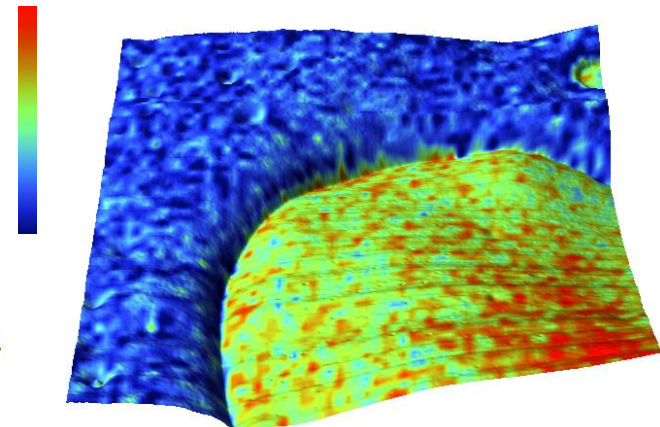
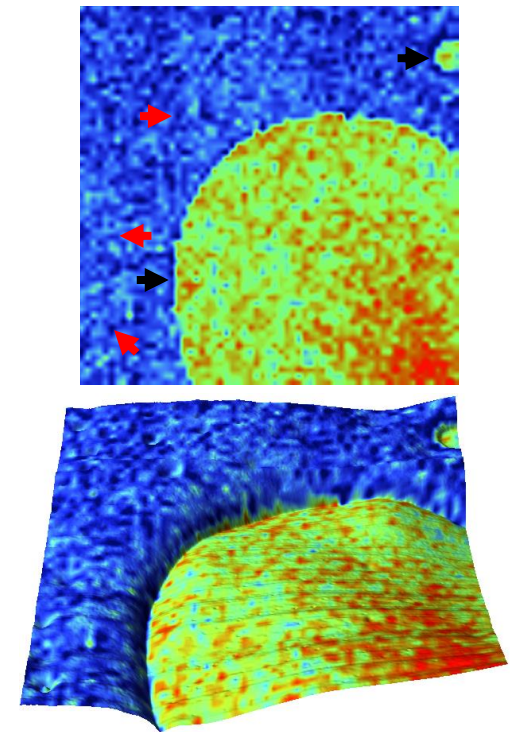3D topography with max. adhesion force overlay

Force mapping

- Areas of high modulus (green-red) correlate with larger and smaller PS islands that are also visible in topography (black arrows). The low modulus area (dark blue) corresponds to the PB matrix. The light blue regions in the PB matrix also reveal slight depressions in topography (red arrows).

- Adhesive properties of the sample correlate with the high elastic modulus regions and identify the PS islands (black arrows) but not with the areas of higher modulus in the PB matrix (red arrow).

3D topography (dynamic mode)

3D topography with elastic modulus overlay

3D topography with max. adhesion force overlay

Force mapping

- Force maps provide large amounts of data.

- Histograms of the extracted parametes can be calculated and statistically evaluated.
  - Adhesion PS: 32±3 nN
  - Adhesion PB: 23±2 nN
  - El. Modulus PS: 1.2±0.2 GPa
  - El. Modulus PB: 3.7±0.2 MPa

- Modulus calculated using the DMT model.

- The accuracy of the modulus calculation depends on the stiffness of the cantilever relative to the stiffness of the material probed.

3D topography with elastic modulus overlay
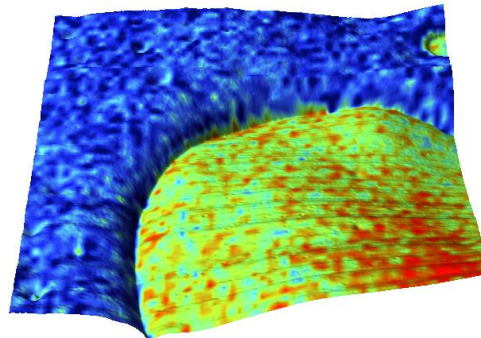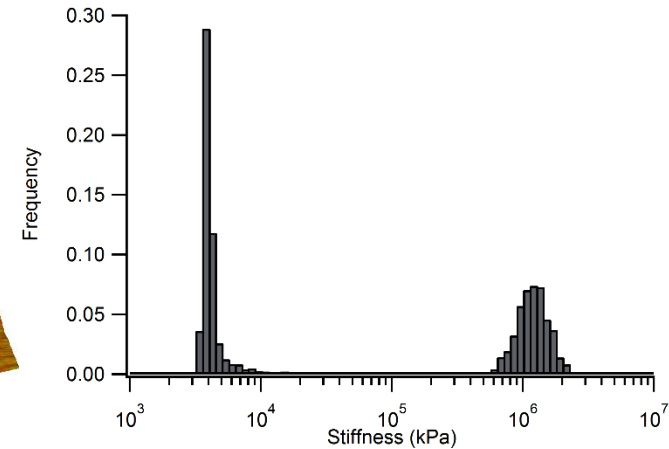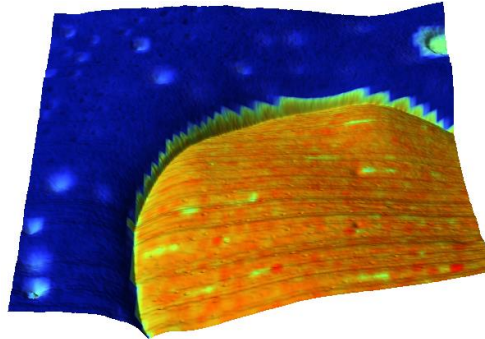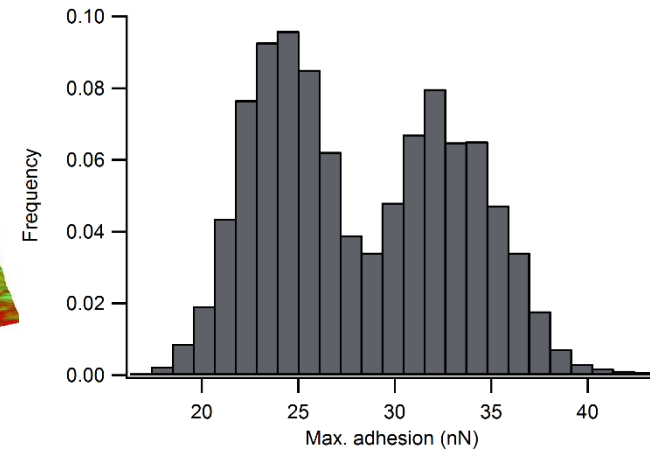

3D topography with max. adhesion force overlay

Force mapping and phase imaging

- Force mapping and phase imaging of the same area

- Comparison of phase signal with force mapping results:

  - Phase response on PS strongly correlates with the results of the elastic modulus and max. adhesion measurements (black arrows).

  - In the PB matrix some areas with only a slight negative phase shift (light green, red arrows) co-locate with a slightly increased elastic modulus. The max. adhesion force does not show significant response in these regions. Also in the topography only minor depressions can be observed.

- phase range 15°

3D topography
(dynamic mode)

3D topography with
phase shift overlay

3D topography with
elastic modulus overlay

3D topography with max.
adhesion force overlay