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Probing from Both Sides: Reshaping the Graphene Landscape via Face-to-Face Dual-Probe Microscopy

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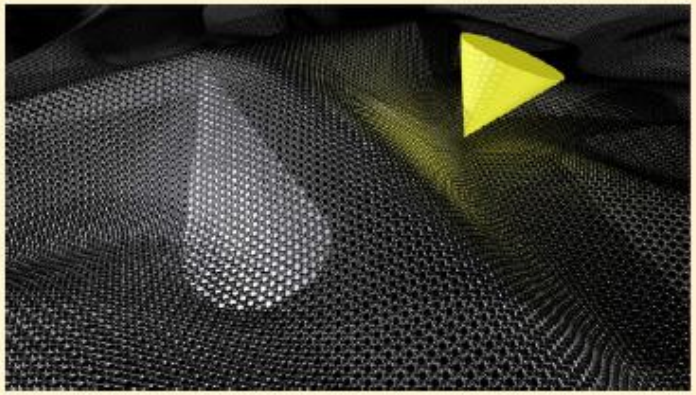
ABSTRACT: In two-dimensional samples, all atoms are at the surface and thereby exposed for probing and manipulation by physical or chemical means from both sides. Here, we show that we can access the same point on both surfaces of a few-layer graphene membrane simultaneously, using a dual-probe scanning tunneling microscopy (STM) setup. At the closest point, the two probes are separated only by the thickness of the graphene membrane. This allows us for the first time to directly measure the deformations induced by one STM probe on a free-standing membrane with an independent second probe. We reveal different regimes of stability of few-layer graphene and show how the STM probes can be used as tools to shape the membrane in a controlled manner. Our work opens new avenues for the study of mechanical and electronic properties of two-dimensional materials.



KEYWORDS: *Graphene, graphene membrane, scanning tunneling microscopy, multiprobe STM, mechanical deformation, molecular dynamics simulation*

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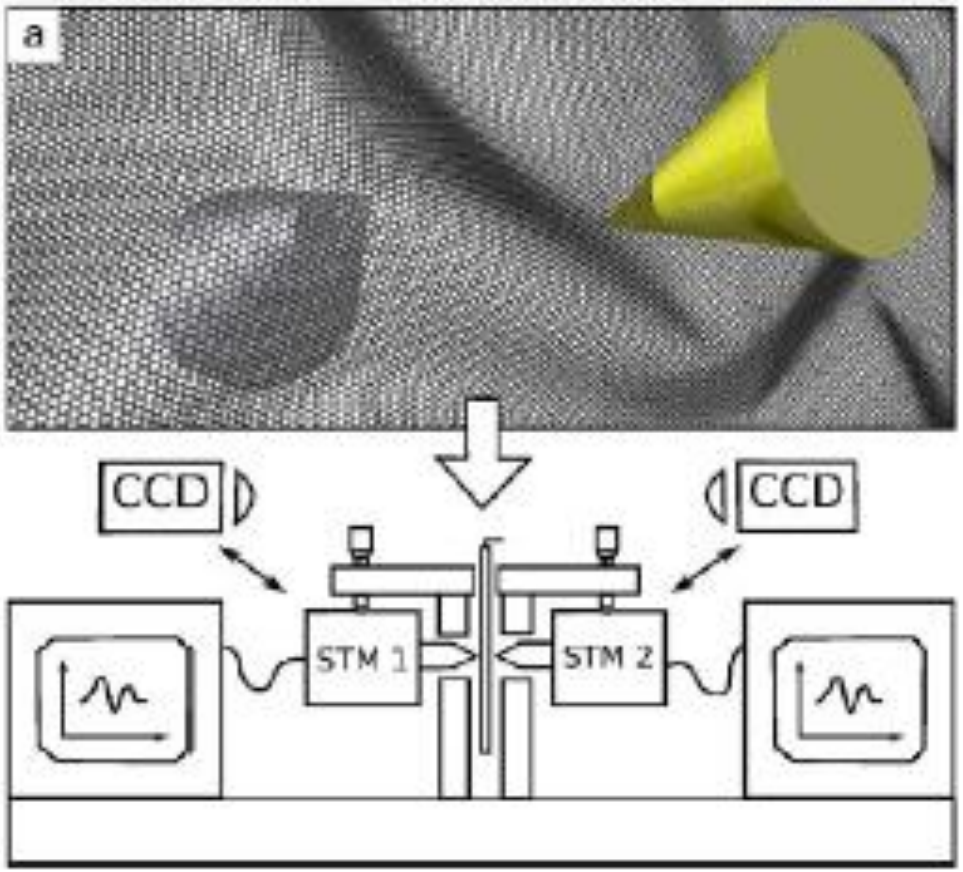
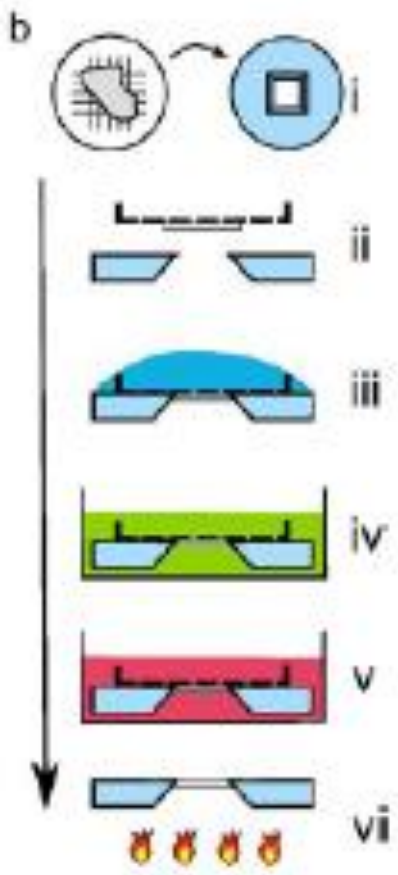
Experimental setup



- DMA (Danish Micro Engineering)
- 2× independent STM head
  - 5×5 µm scan range
  - Scan heads can be replaced by CCD cam with an accuracy < 5 µm
  - A stationary and a scannig tip
- Alignment process:
  - Position of the sample is marked with a CCD camera
  - Sample removed, opposite to the camera a tip is aligned to the previously marked position
  - Repeat the above on the other side
  - The 2 probes are now within scanning range

Sample preparation



- Free-standing graphene membrane, 5-8 layers

I. Sample transfer
  - Mechanical exfoliation
  - Transfer to dissolvable plastic TEM grid
  - Transfer to TEM window frame (2 µm, gold coated Si)

II. Alignment

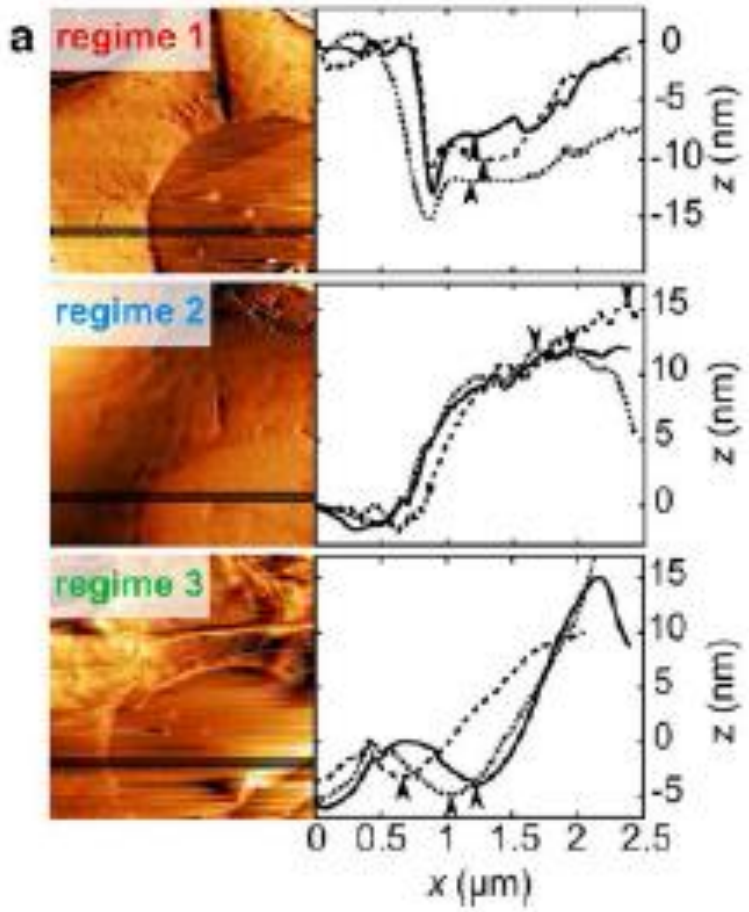
III. Isopropanol – adhesion increases between flake and substrate

IV. Chloroform bath – 10 min, softens the support foil of the grid

V. Dissolution of the grid foil

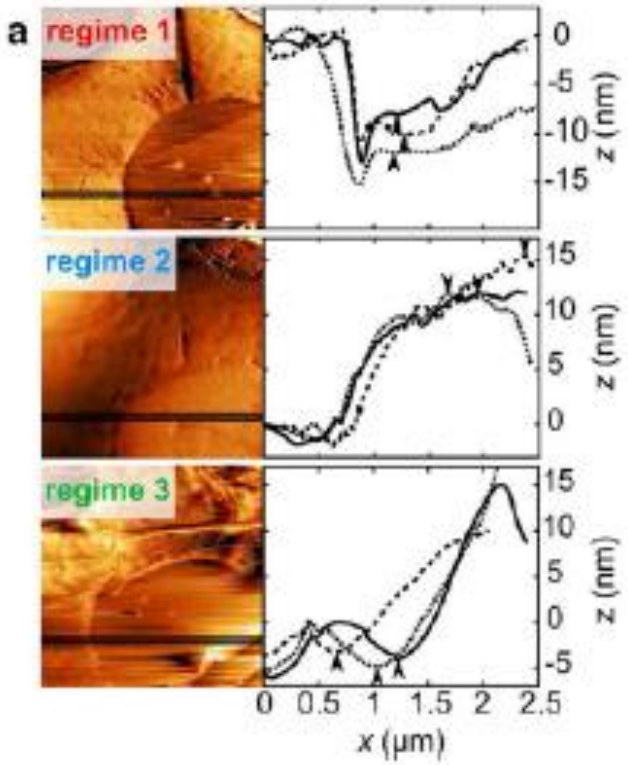
VI. Heat treatment – 350 °C, 1h in vacuum

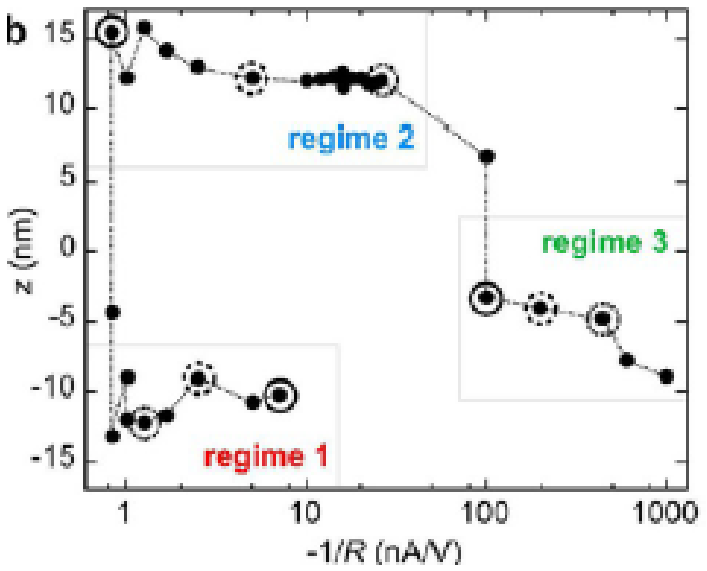
Push/pull the membrane



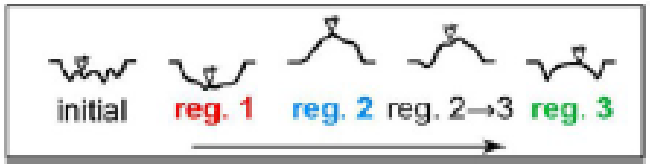
- With a single tip
- STM tip can be used to push/pull the membrane, creating permanent deformations
- Initial shape: random (-0.2 V, 1nA)
- Increase the bias voltage, sudden change at -1.2 V
- Increase the current, lower the voltage → Pauli repulsion becomes dominant.

Push/pull the membrane



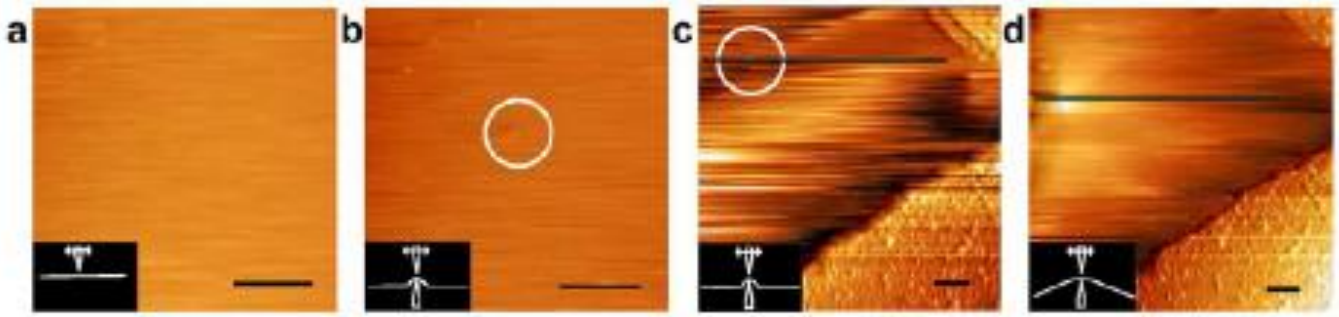


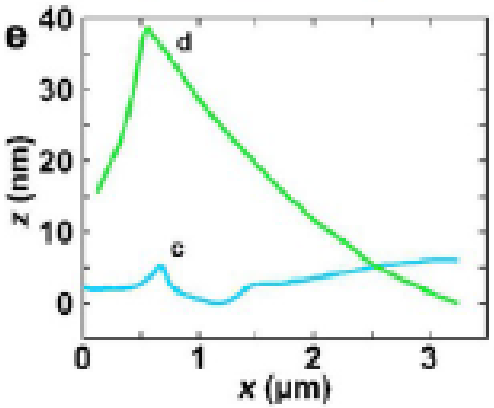
Inverse tunneling resistance



- *regime 1* → *regime 2* fast transition
- *regime 2* → *regime 3* slow transition

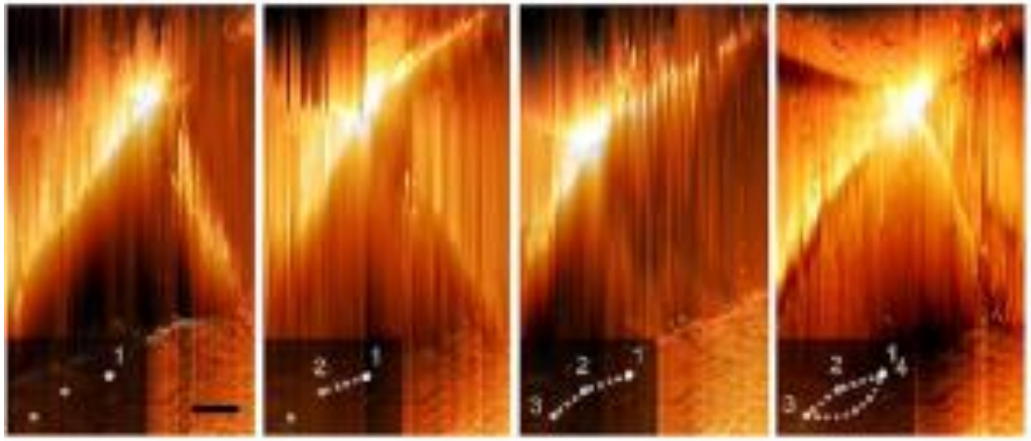
Stretching of the membrane





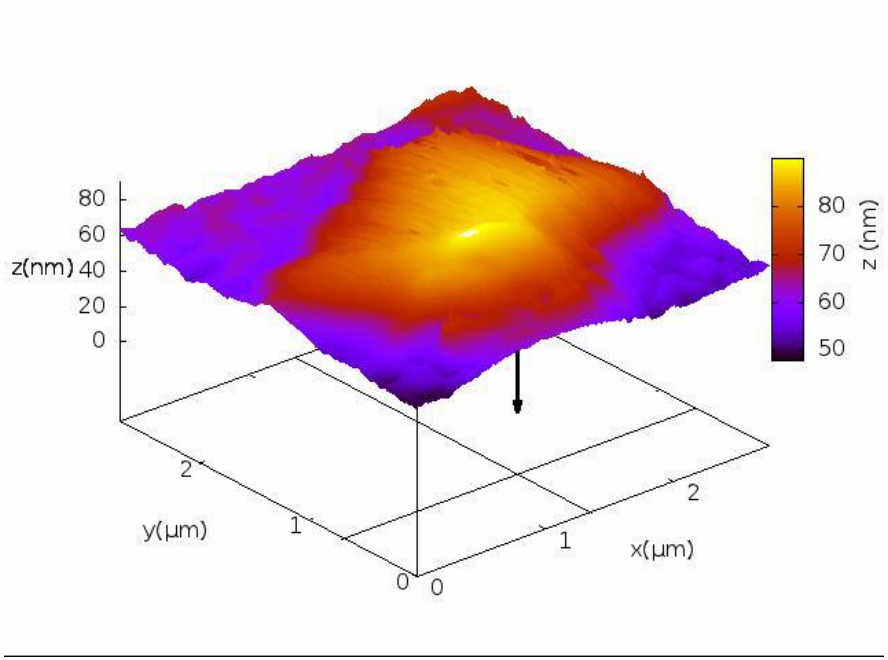
- Stationary tip: constant current → constant force between tip and sample
- (b), (c): point feature
- (d): mountain feature

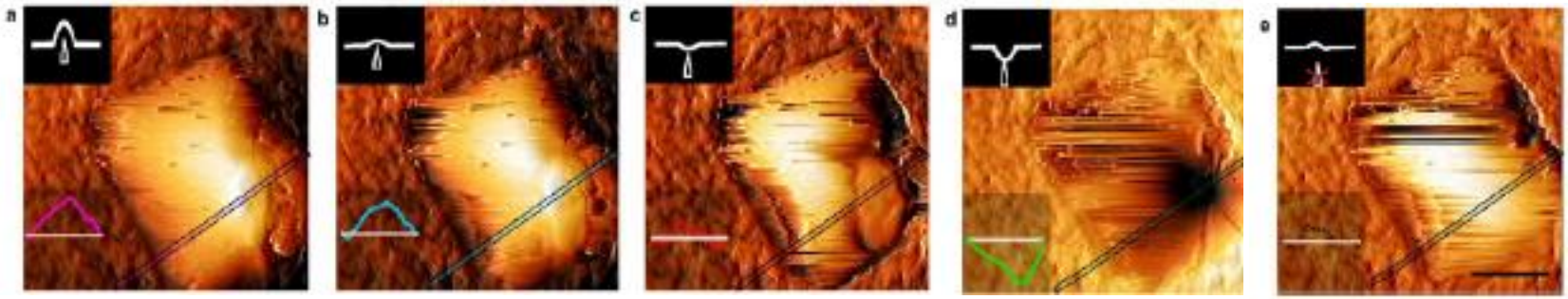
- Moving the stationary tip:



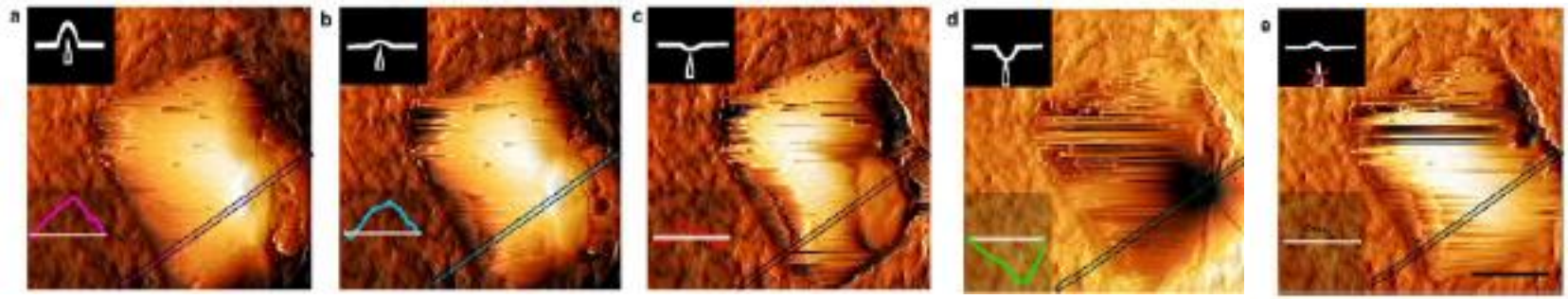
Stretching of the membrane

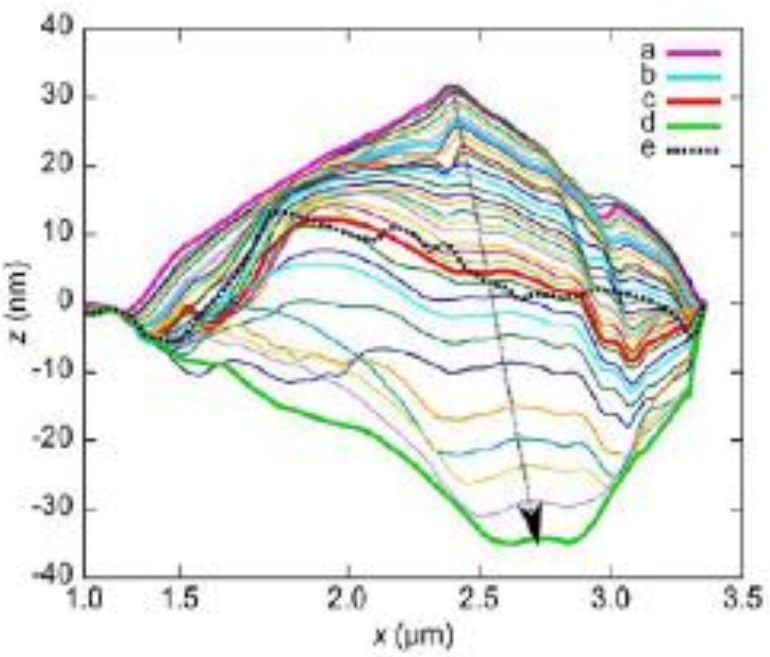
- Adjusting the vertical position of the stationary tip:
  - Feedback off
  - Slowly pulling the probe away
- Mountain - > valley
- After probe breaks away, membrane snaps back to a freely suspended configuration



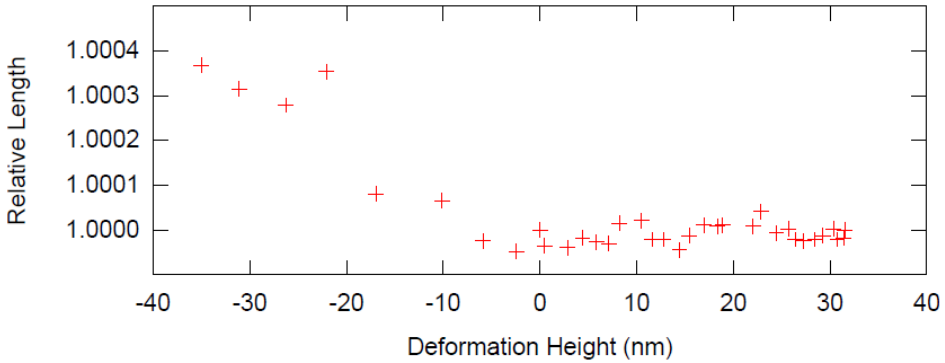


Stretching of the membrane





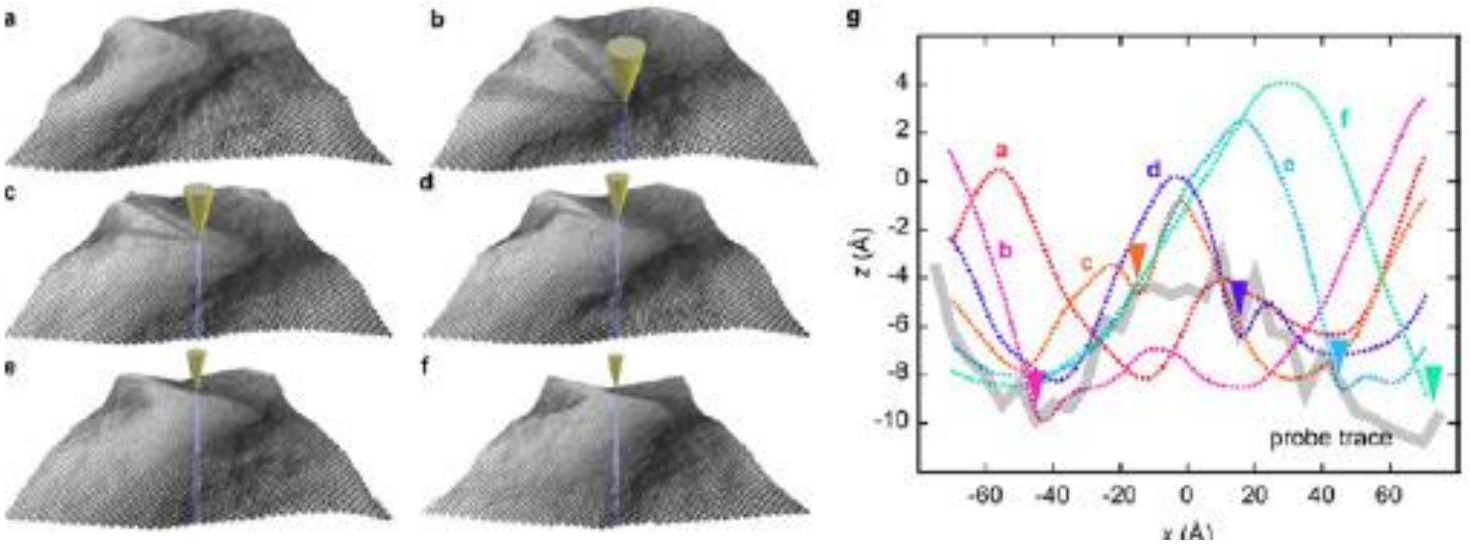
Length of the line profiles:



- Maximum strain is only 0.04%
- Carbon bonds are not significantly stretched, only the shape of existing deformations is changed

Molecular Dynamics simulations

- STM images effected by tip induced elastic deformations → difficult to interpret the results



- 17x17 nm graphene, 2% compression, MD simulations (300K, 5ps) → rippled surface

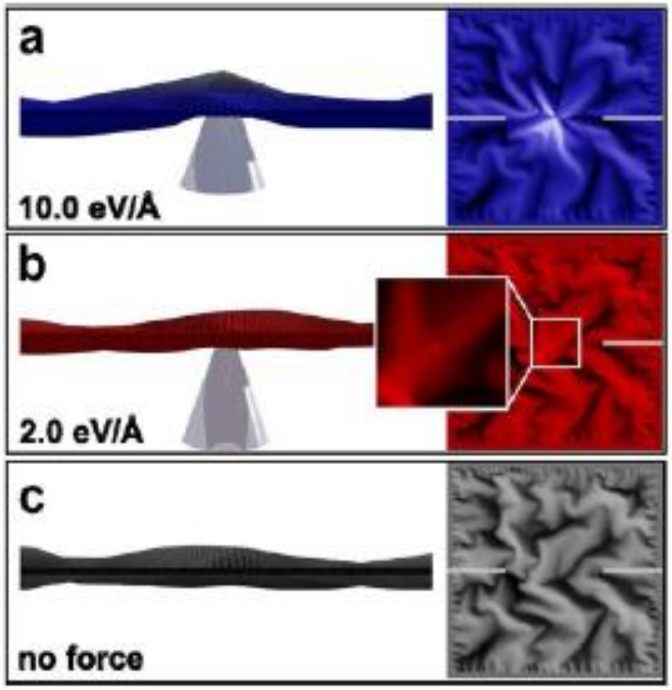
- Simulated scan:
  - scan line differs from the membrane shape at any time
  - the amplitude of the measured ripples is less than simulated
  - Scanning tip can locally turn concave locations to convex ones and inversely

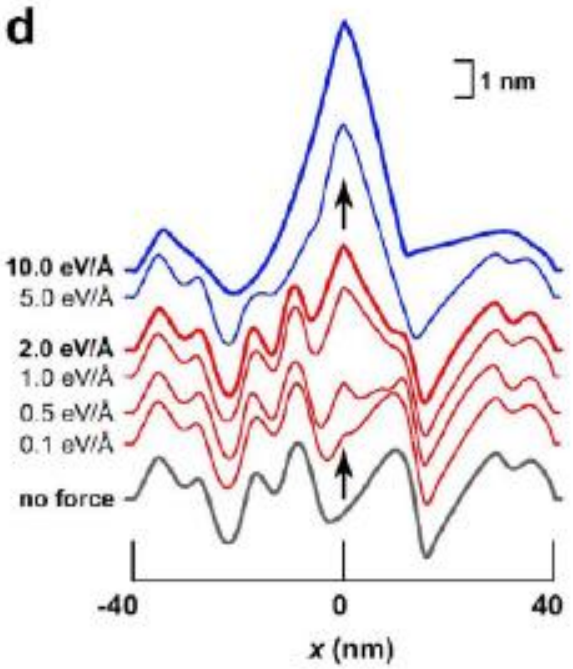
Molecular Dynamics simulations

- Fixed edges, starting from rippled surface:
  - Forces > 0.05 eV/Å → mountain like configuration
  - Pushed back only by several full scans

- Low force is enough to stretch out the initially random deformations
- Pushing/pulling the membrane in the opposite direction imposing a strain to the material requires a significantly higher force

- 84x84 nm:
  - Lower forces: point feature
  - Higher forces: mountain feature
  - Deformation depends on:
    - imposed force
    - initial structure





- When the applied force is close to the deformation threshold, continuous scanning can trigger the transition from point to mountain feature.

Thank you for your attention!