CONTROLLED SURFACE RIPPLING ON GLASS AND POLYMER SURFACES

Mechanics of
Functional Materials

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OUTLINE

1. Surface rippling inside silica glass scratches

   (Gnecco, E., J. Hennig, E. Moayedi and L. Wondraczek (2018). "Surface rippling of silica glass surfaces scraped by a diamond indenter." Physical Review Materials)

2. Surface rippling on polystyrene surfaces
   1. Boundary effects

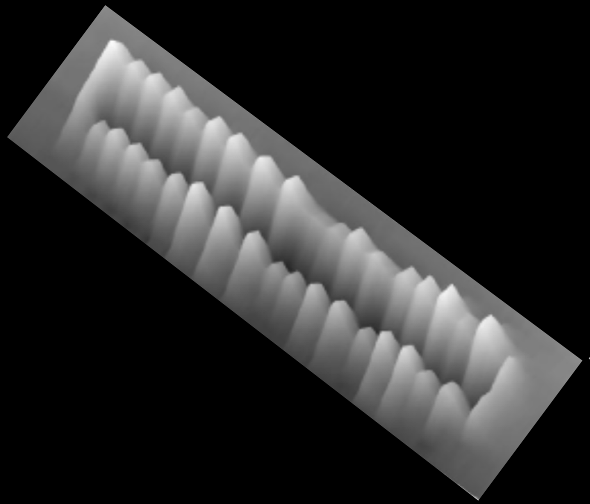
      (Hennig, J., V. Feller, P. J. Martinez, J. J. Mazo and E. Gnecco (2022). “Locking effects in plowing-induced nanorippling of polystyrene surfaces”, in progress)

   2. Nanoparticle release

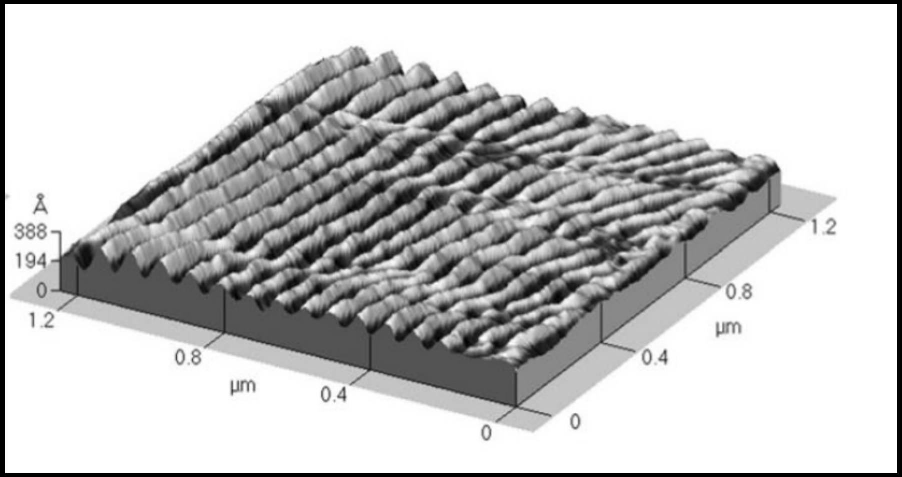
      (Hennig, J., A. Litschko, J. J. Mazo and E. Gnecco (2021). “Nucleation and detachment of polystyrene nanoparticles from plowing-induced surface wrinkling”, Applied Surface Science Advances)

SURFACE RIPPLING

- suface rippling due to sliding contacts is a frequently observed but not clearly understood phenomenon
- known from different materials



Topography image of a scratch groove formed on KBr crystal
(Socoliuc, A., E. Gnecco, R. Bennewitz and E. Meyer (2003). "Ripple formation induced in localized abrasion." Physical Review B)



3D topography image of ripples on PET
(D´Acunto, M., Napolitano, S., Pingue, P., Giusti, P. and Rolla, P. (2007). "Fast formation of ripples induced by AFM. A new method for patterning polymers on nanoscale." Materials Letters)

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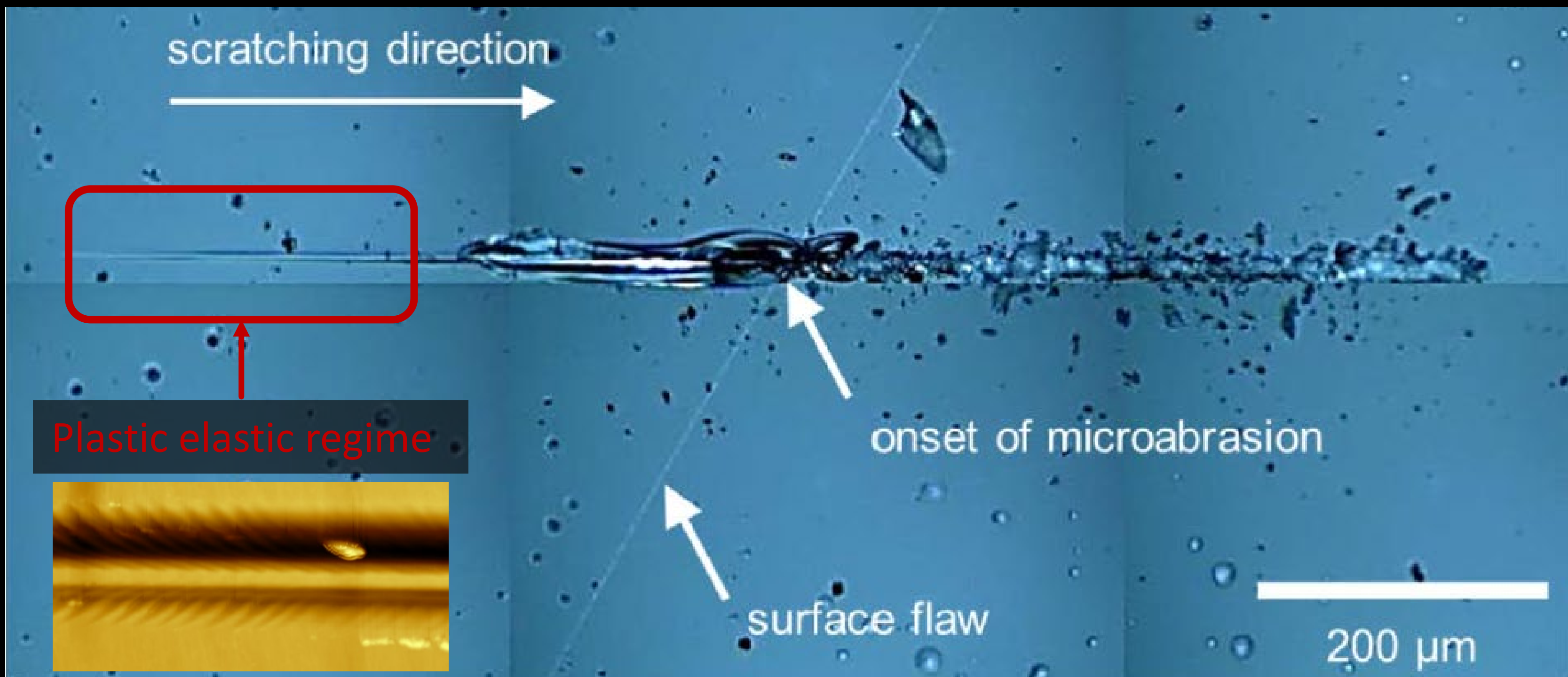
MOTIVATION

- What is known for scratch tests on glasses?

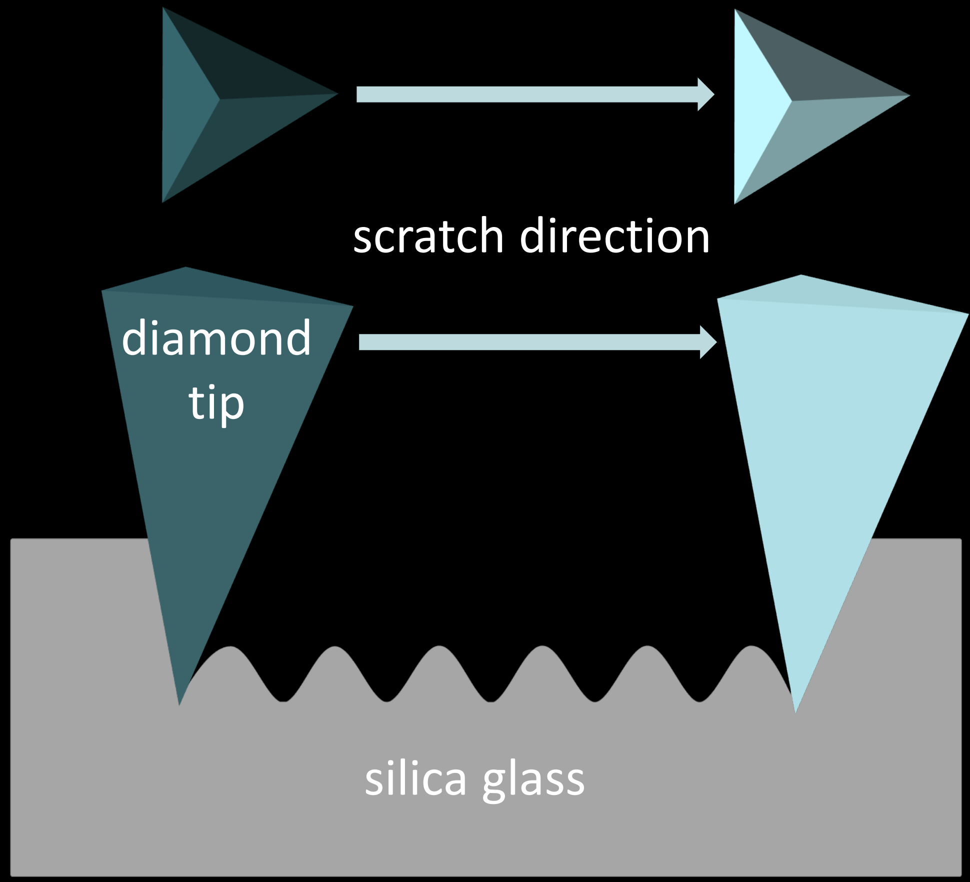


Typical scratch pattern observed on silicate glasses during scratching under increasing normal load

(Moayedi, E. and L. Wondraczek (2017). "Quantitative analysis of scratch-induced microabrasion on silica glass." Journal of Non-Crystalline Solids)

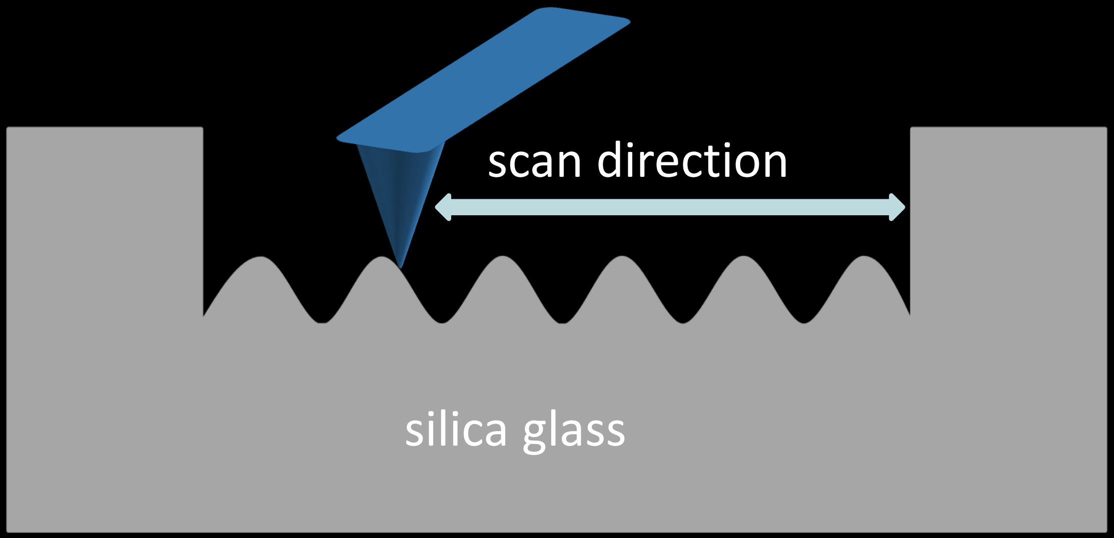
NANOINDENTER SCRATCH TESTS

- scratch test with nanoindenter with diamond tip (Berkovich tip, edge forward)
- normal load 30 mN (plastic elastic deformation)
- scratch velocities from 10 µm/s up to 500 µm/s

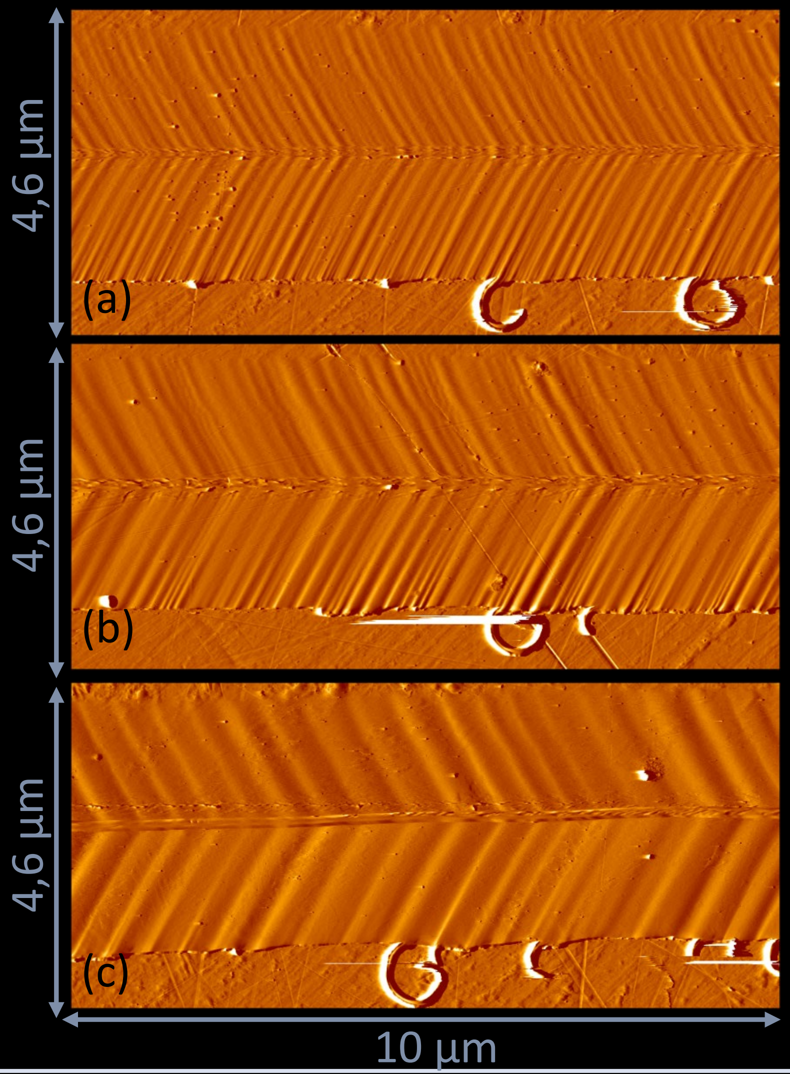


NANOINDENTER SCRATCH TESTS

- imaging of scratch groove with AFM
- scanning along the scratch direction
- herringbone pattern inside the scratch



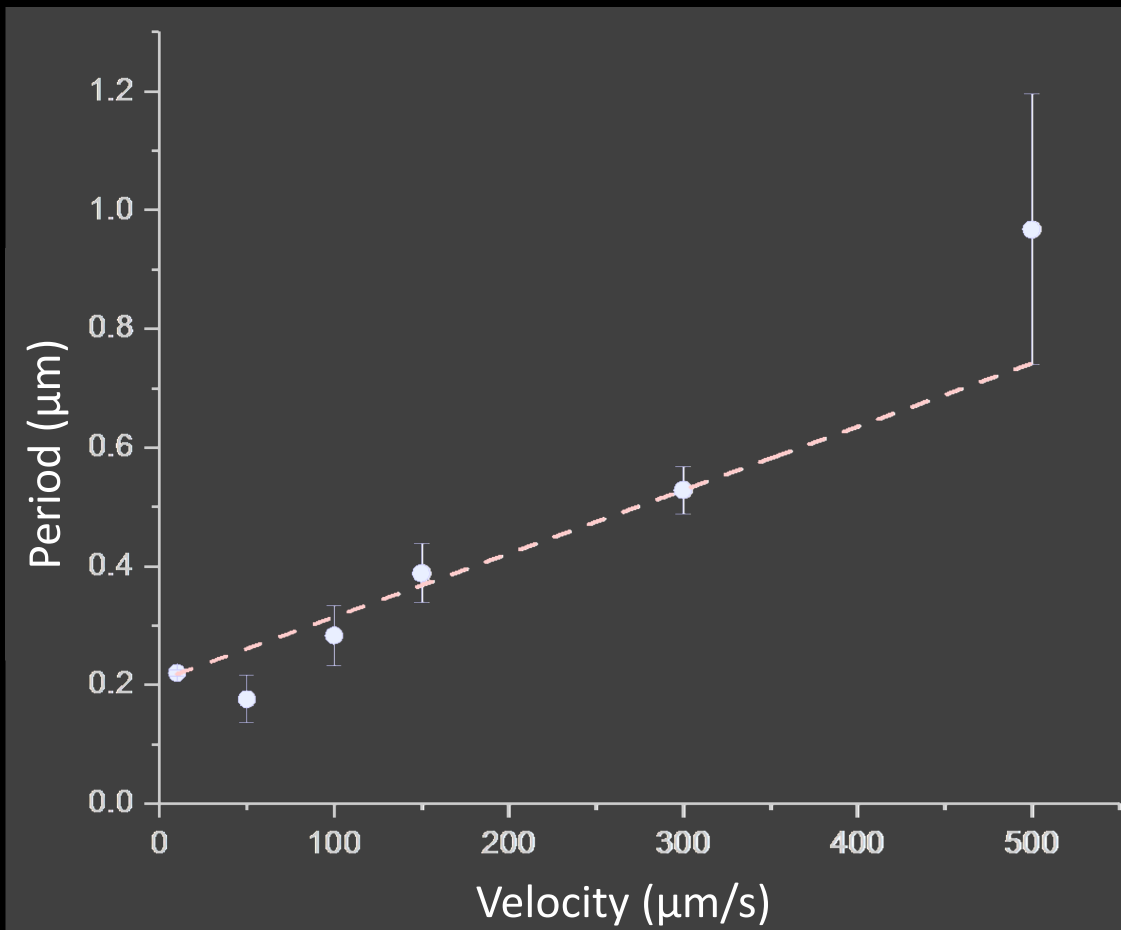
HERRINGBONE PATTERN



- periodic herringbone pattern in all scratches and allover the whole scratch
- periodicity changes with scratch velocity

AFM topography of silica glass surfaces scratched from left to right with a normal load of 30 mN and scratch velocities of
(a) 10 µm/s
(b) 100 µm/s
(c) 300 µm/s.

VELOCITY DEPENDENCE OF RIPPLE PERIOD



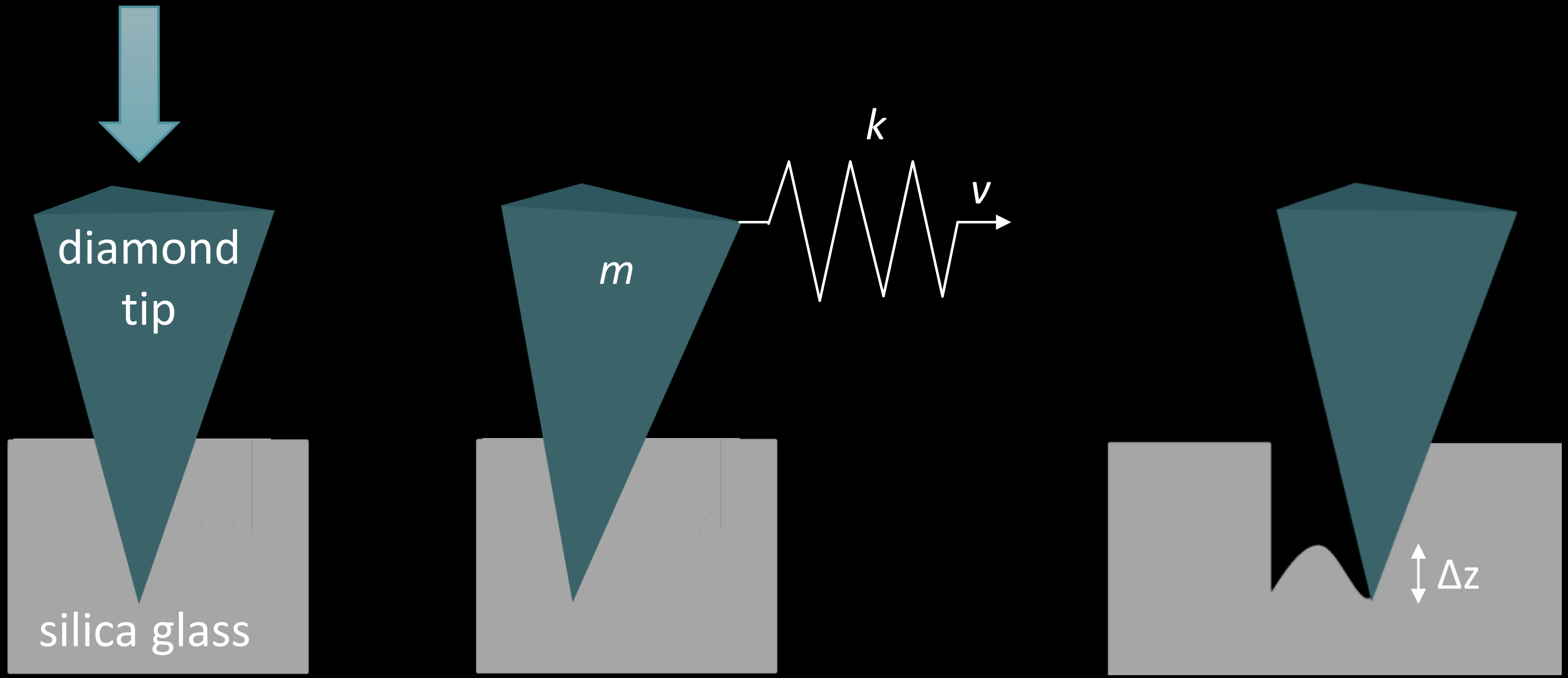
$\lambda = \lambda_0 + t_0 v$

$\lambda_0 = (207 \pm 8)$ nm
$t_0 = (1{,}07 \pm 0{,}15)$ ms

Velocity dependence of the ripple period as measured by AFM *ex situ* after scratching with a normal load of 30mN and linear fit of the experimental data points.

(Gnecco, E., J. Hennig, E. Moayedi and L. Wondraczek (2018). "Surface rippling of silica glass surfaces scraped by a diamond indenter." Physical Review Materials)

RIPPLE PATTERN CREATED BY STICK SLIP MOTION



estimated time spent in dip: λ/v = 22,0 ms

additional surface indentation Δz = 5,5 nm

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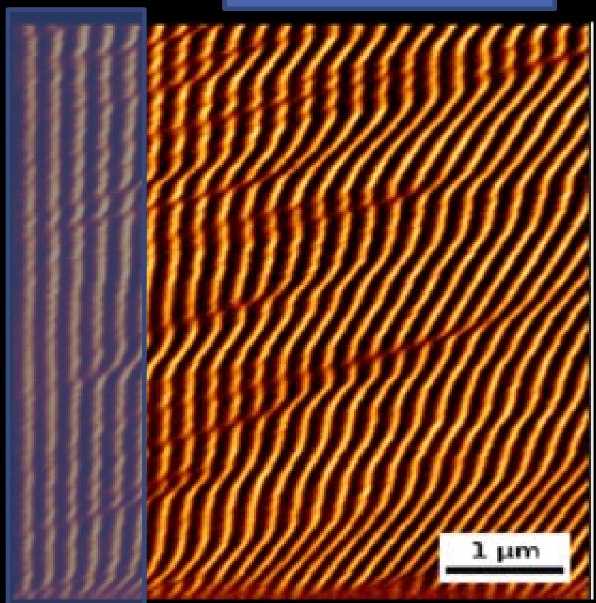
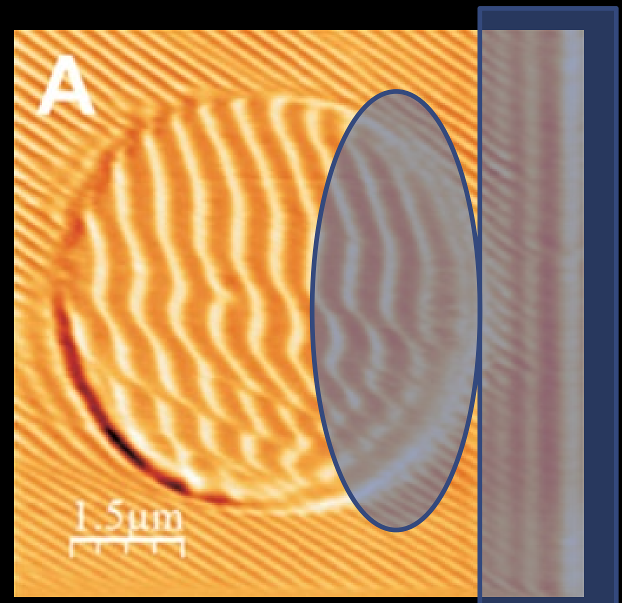
MOTIVATION

- Surface rippling induced by a sharp tip (e. g. AFM tip)
- 2 regions: boundary region and steady region



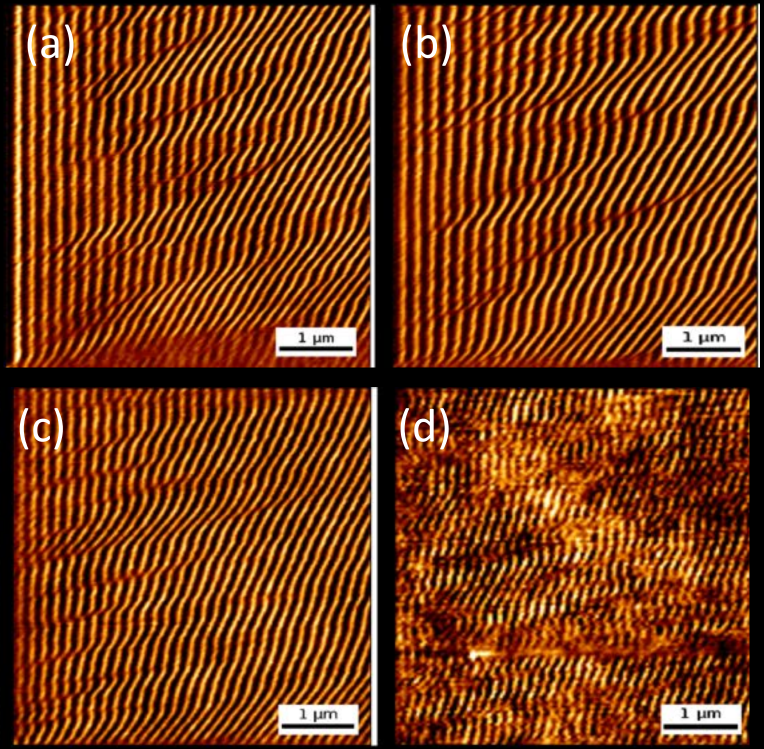
Wavy pattern formed on a PS surface during scanning with AFM with constant load
(Mazo, J., P. Martínez, P. Pedraz, J. Hennig and E. Gnecco (2019). "Plowing-Induced Structuring of Compliant Surfaces." Physical Review Letters)



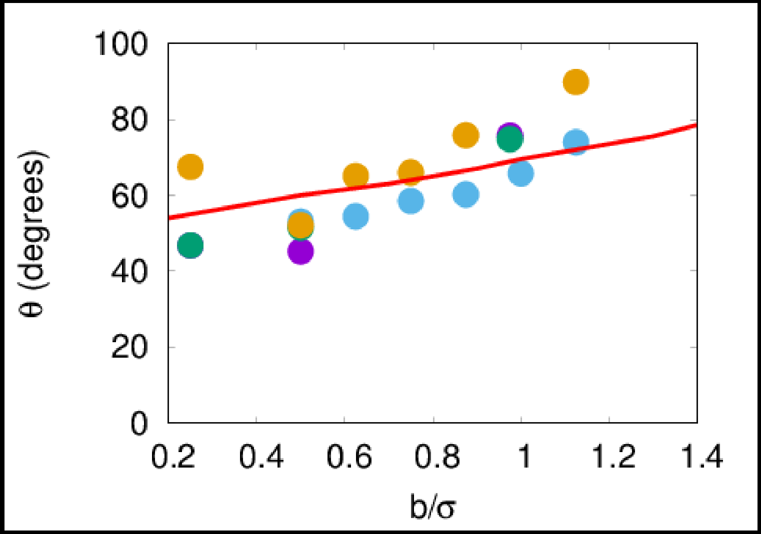
Wavy pattern formed on a PET surface during scanning with AFM with different loads
(Napolitano, S., M. D'Acunto, P. Baschieri, E. Gnecco and P. Pingue (2012). "Ordered rippling of polymer surfaces by nanolithography: influence of scan pattern and boundary effects." Nanotechnology)

MOTIVATION

- Ripple orientation in steady region defined by scan line distance (at ambient conditions and loading forces about 100 to 200 nN)



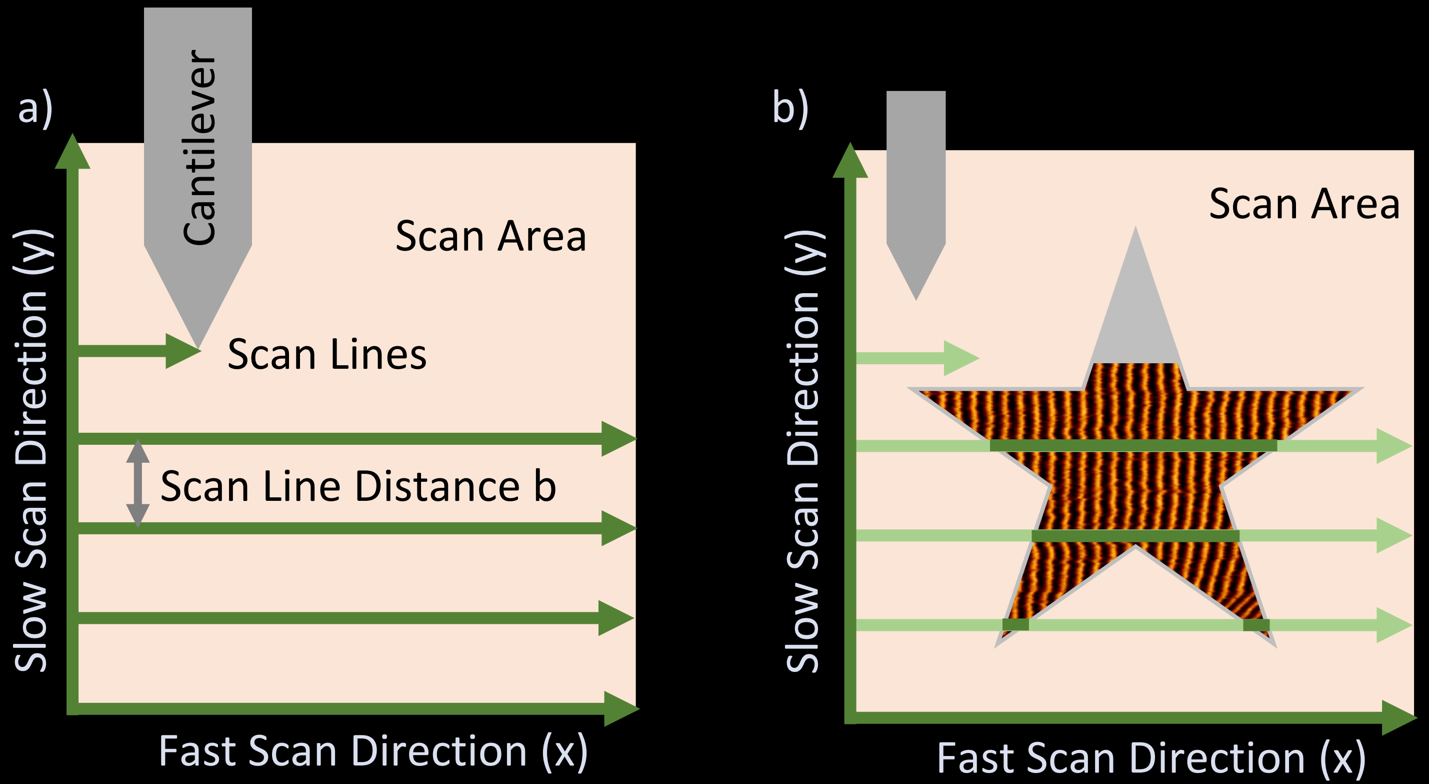
Experimental ripple patterns: AFM topography images of scrapped poystyrene surface. scan line distances of (a) b=10 nm, (b) b=20 nm, (c) b=30 nm and (d) b=35 nm.



Ripple orientation far from the left boundary as a function of the scan line distance b. The circles correspond to distinct series of AFM measurements The red line corresponds to the results of the numeric simulations.

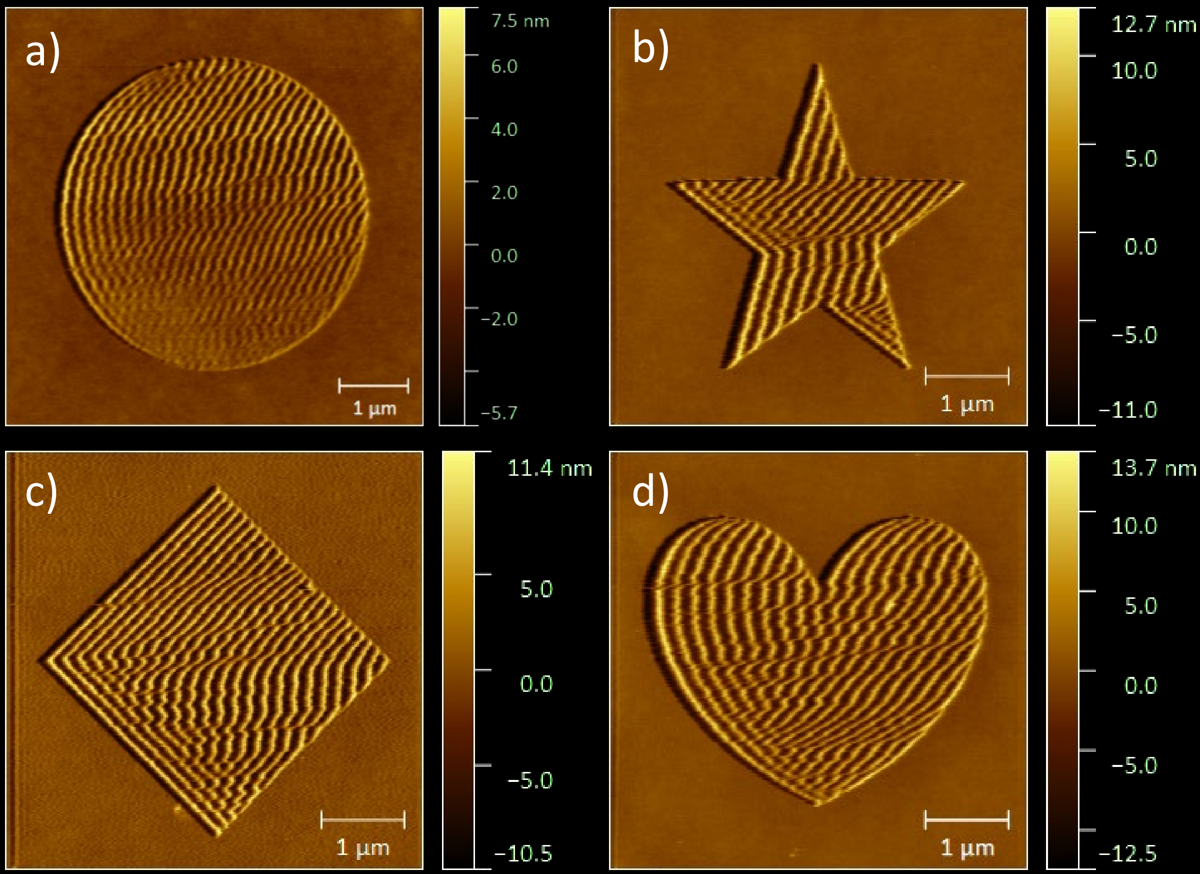
(Mazo, J., P. Martínez, P. Pedraz, J. Hennig and E. Gnecco (2019). "Plowing-Induced Structuring of Compliant Surfaces." Physical Review Letters)

EXPERIMENTAL SETUP FOR SURFACE RIPPLING INSIDE DEFINED GEOMETRIES



a) Scrapping PS surfaces without setpoint variation used as reference
b) Scrapping PS surface inside a defined geometry applying a multi setpoint script for AFM scan. Normal load outside the star: 20 – 30 nN, inside the star: 100 – 150 nN.

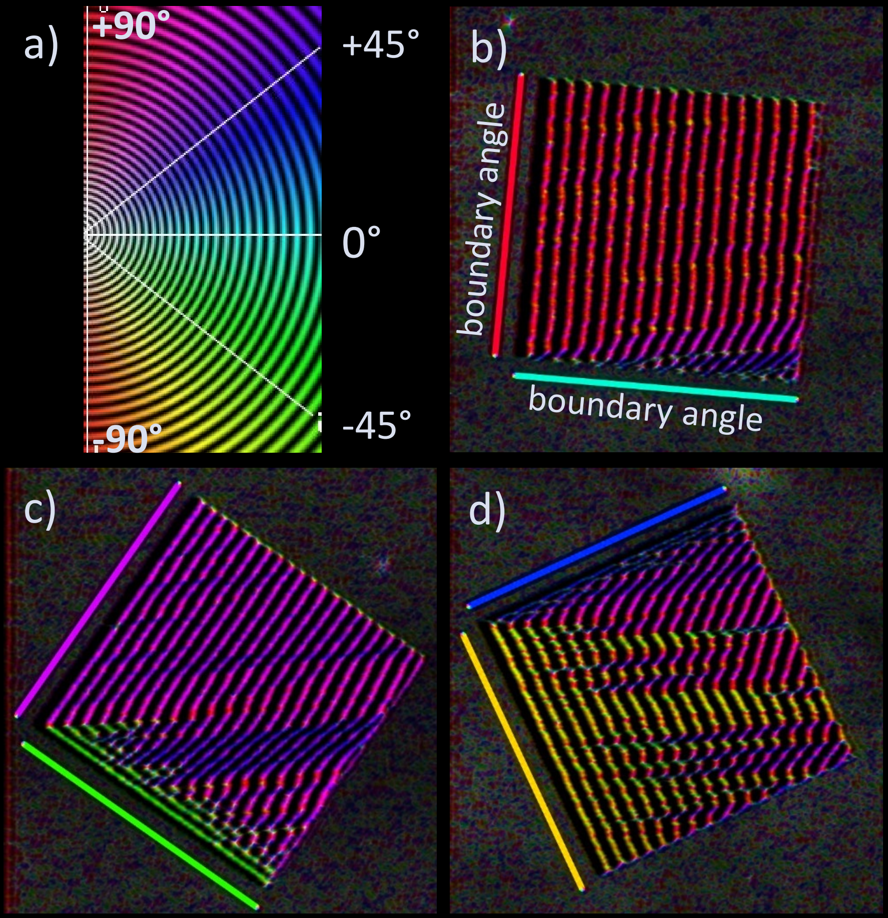
SURFACE RIPPLING INSIDE DEFINED GEOMETRIES



a) circle, b) star, c) square rotated by 45° and d) heart with rippling structure showing different ripple orientations depending on left boundary.

(Hennig, J., V. Feller, P. J. Martinez, J. J. Mazo and E. Gnecco (2022). "Locking effects in plowing-induced nanorippling of polystyrene surfaces", in progress)

RIPPLE ORIENTATION INSIDE A ROTATED SQUARE



Rippling structures inside rotated squares with a color analysis for angles.

a) color scale of angle
(Z. Püspöki et al., Transforms and Operators for Directional Bioimage Analysis: A Survey, Advances in Anatomy, Embryology and Cell Biology (2016) Springer International Publishing),

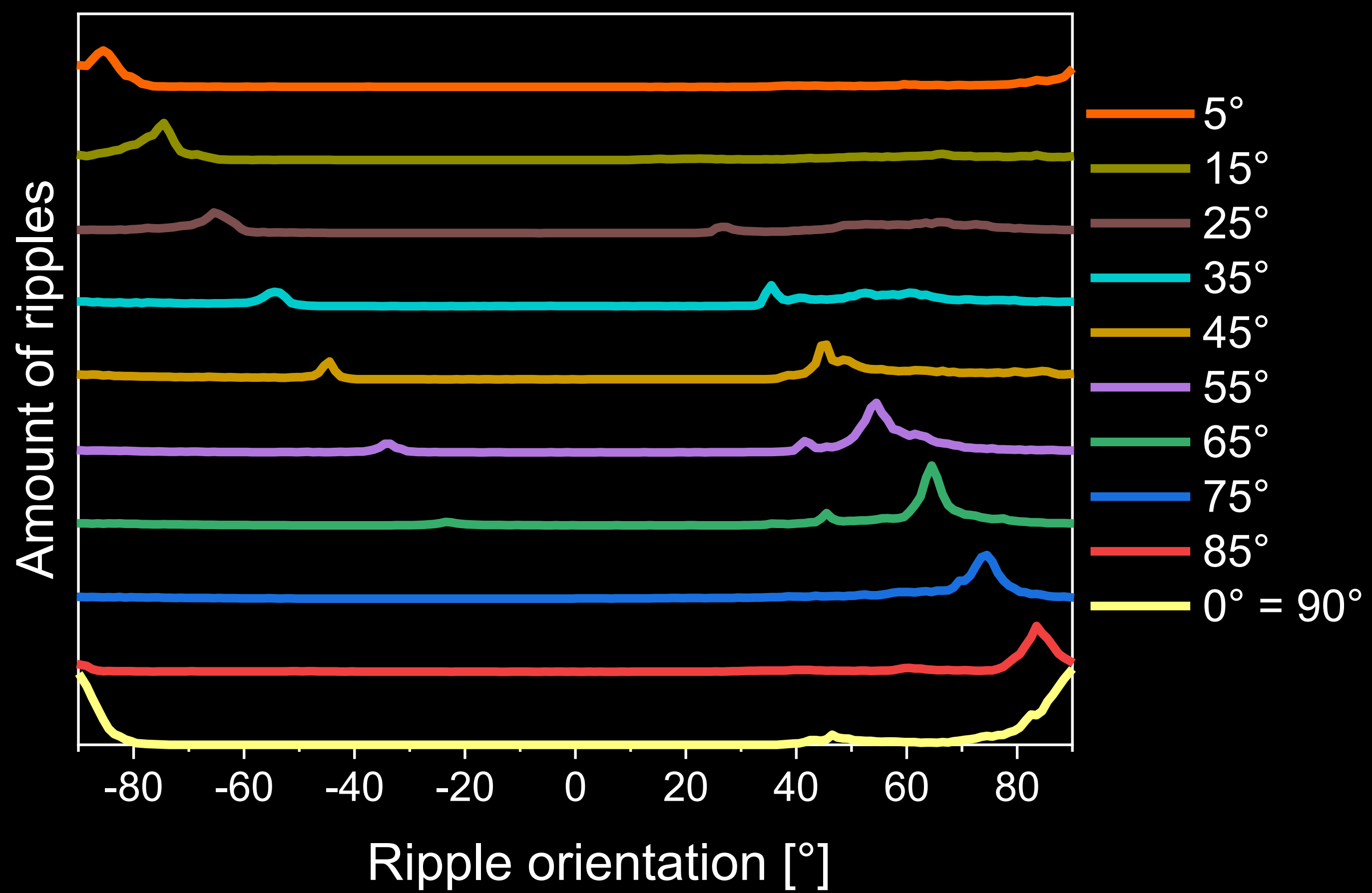
b) structured square rotated by 85°,

c) by 55° and

d) by 25°.

(Hennig, J., V. Feller, P. J. Martinez, J. J. Mazo and E. Gnecco (2022). "Locking effects in plowing-induced nanorippling of polystyrene surfaces", in progress)

RIPPLE ORIENTATION INSIDE ROTATED SQUARES



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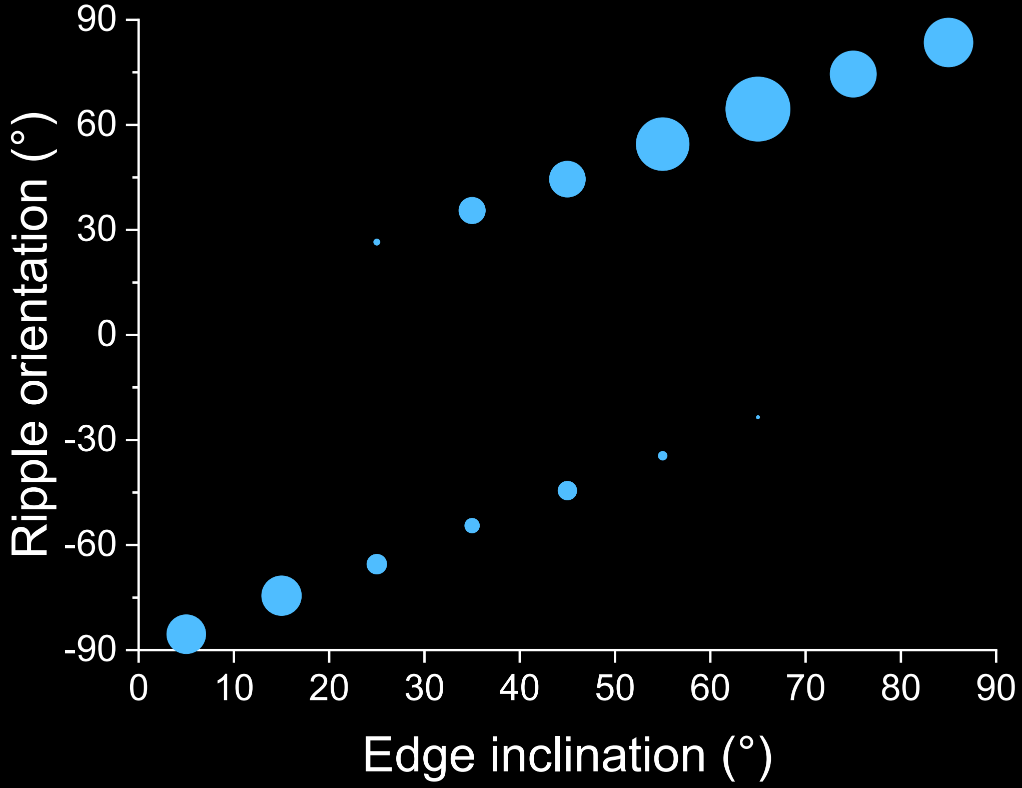

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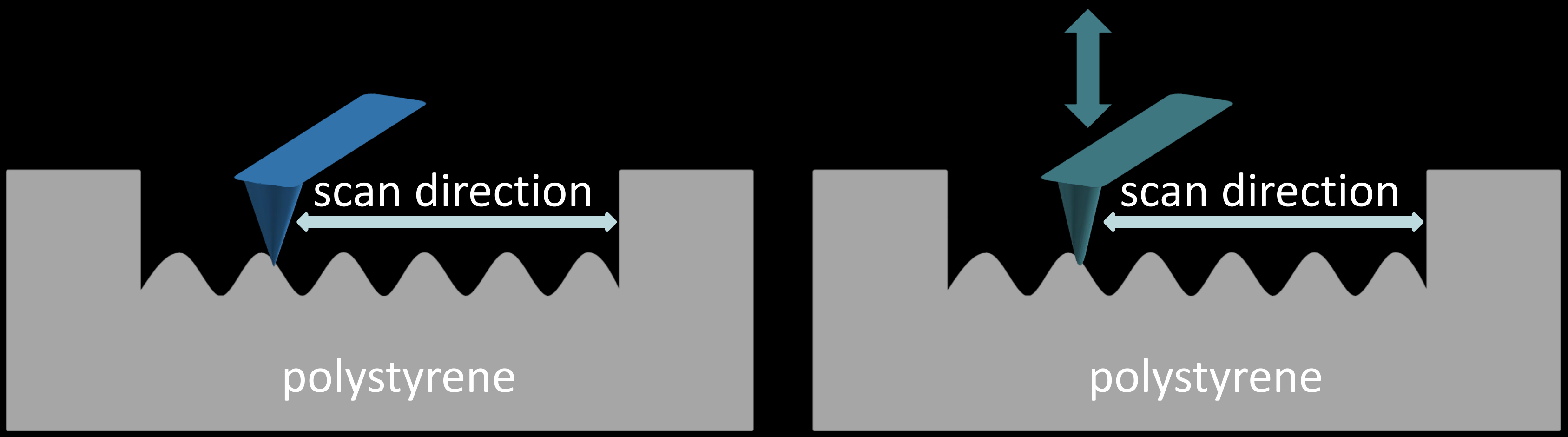
EXPERIMENTAL SETUP

- Structuring of surface in contact mode with contact mode tip (tip radius < 10 nm)
- Imaging in tapping mode with tapping mode tip (tip radius < 7 nm)

Structuring in contact mode

Imaging in tapping mode



TOPOGRAPHY IMAGES OF NANOPARTICLE RELEASE

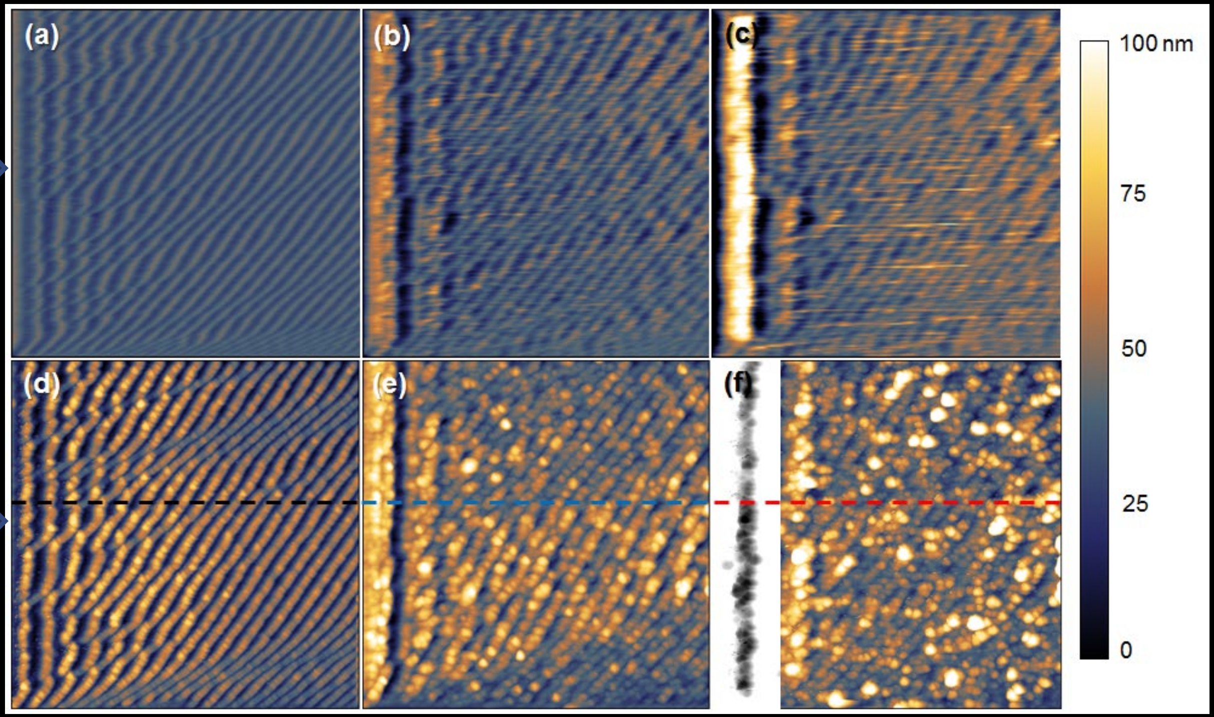
1 x

3 x

5 x

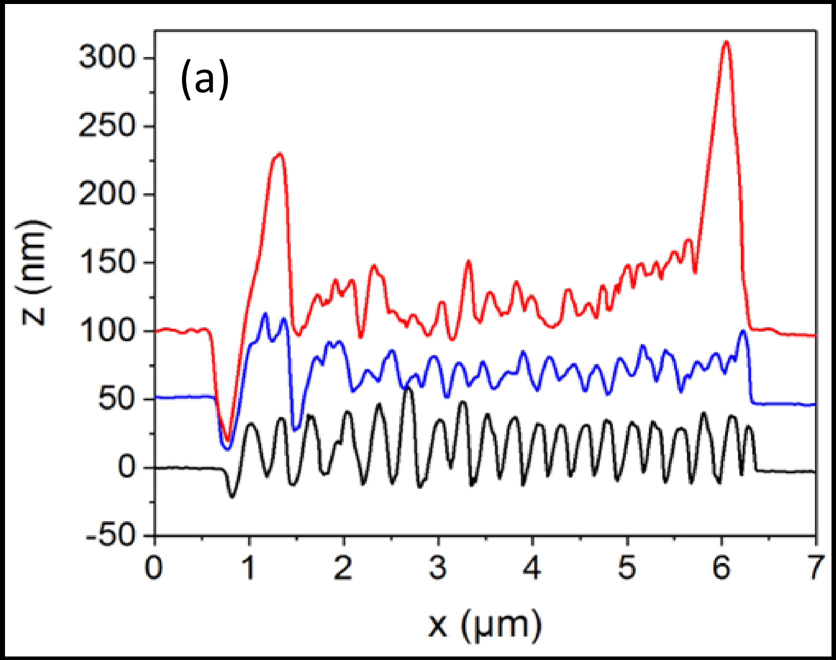
while scraping in contact mode

imaging afterwards in tapping mode

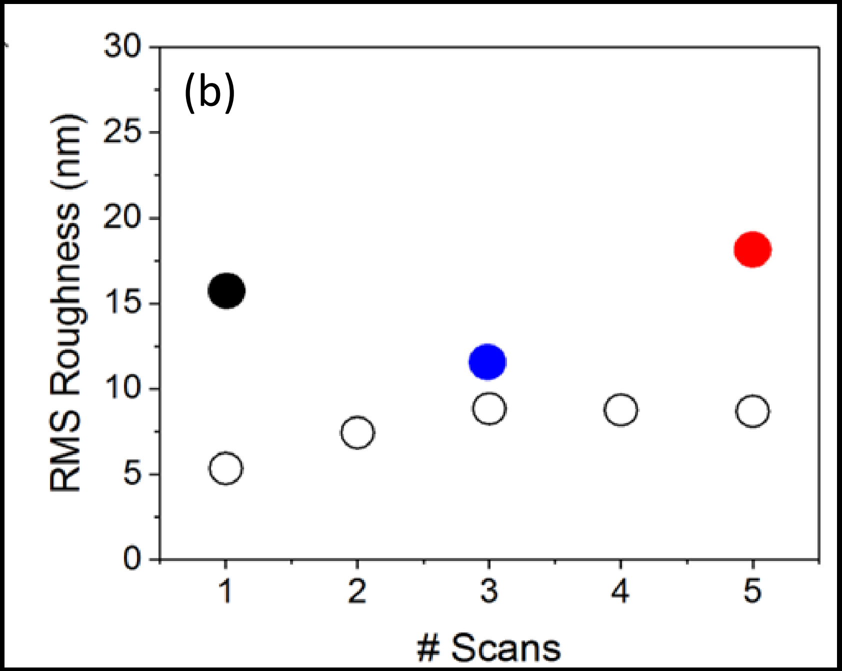


(Hennig, J., A. Litschko, J. J. Mazo and E. Gnecco (2021). “Nucleation and detachment of polystyrene nanoparticles from plowing-induced surface wrinkling”, Applied Surface Science Advances)

EVOLUTION OF ROUGHNESS



Cross sections acquired from tapping mode images after one, three and five scans



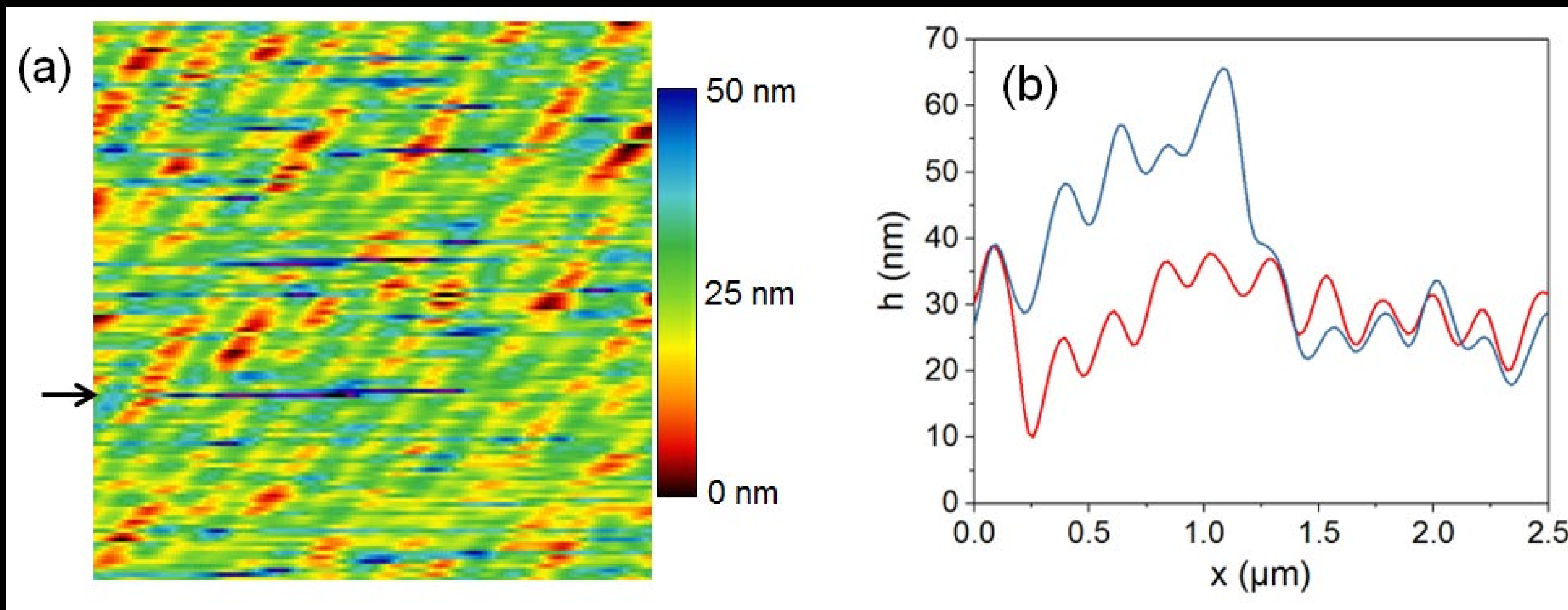
Surface roughness

● while scraping, after

○ one, ● three and ● five scans

(Hennig, J., A. Litschko, J. J. Mazo and E. Gnecco (2021). "Nucleation and detachment of polystyrene nanoparticles from plowing-induced surface wrinkling", Applied Surface Science Advances)

DETACHMENT OF NANOPARTICLES



Topography image (2.5×2.5 µm) acquired while 5th pass of scraping

Cross-section corresponding to the arrow in (a) (in blue) and to the previous scan line (in red).

(Hennig, J., A. Litschko, J. J. Mazo and E. Gnecco (2021). “Nucleation and detachment of polystyrene nanoparticles from plowing-induced surface wrinkling”, Applied Surface Science Advances)

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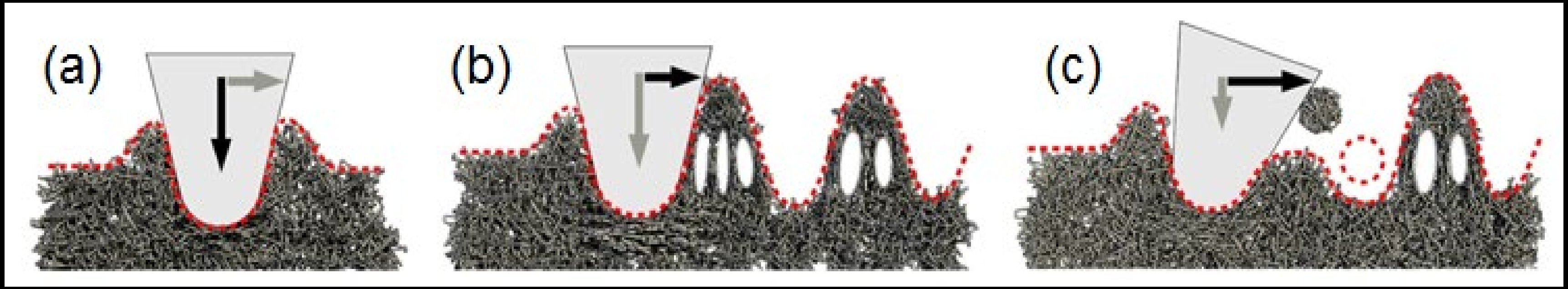

Three phases of the proposed mechanism for particle formation and detachment: (a) Extrusion of material all around the location initially indented by the AFM tip, (b) Crazing process and particle nucleation caused by the increasing lateral force on a (previously formed) ripple pattern; (c) Sphere detachment.

(Hennig, J., A. Litschko, J. J. Mazo and E. Gnecco (2021). "Nucleation and detachment of polystyrene nanoparticles from plowing-induced surface wrinkling", Applied Surface Science Advances)

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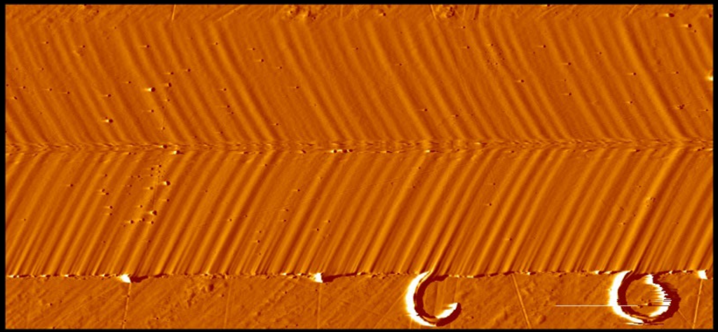
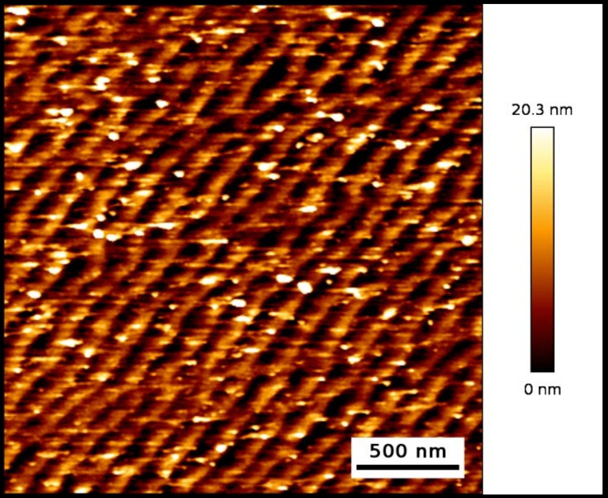


herringbone pattern inside a scratch on silica glass



PS surface scraped by AFM tip several times resulting in surface rippling and detachment of particles

PS surface structured in the shape of a heart by scraping with AFM tip and coloured by angles

ACKNOWLEDGMENT

- Scientific colleagues
  - Enrico Gnecco
  - Elham Moayedi
  - Lothar Wondraczek
  - Juan Mazo
  - Pedro Martínez
  - Patricia Pedraz
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  - Susanne Sandkuhl
  - Alexander Litschko
  - Valentin Feller

- References

  - *Gnecco, E., J. Hennig, E. Moayedi and L. Wondraczek (2018). "Surface rippling of silica glass surfaces scraped by a diamond indenter." Physical Review Materials*
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