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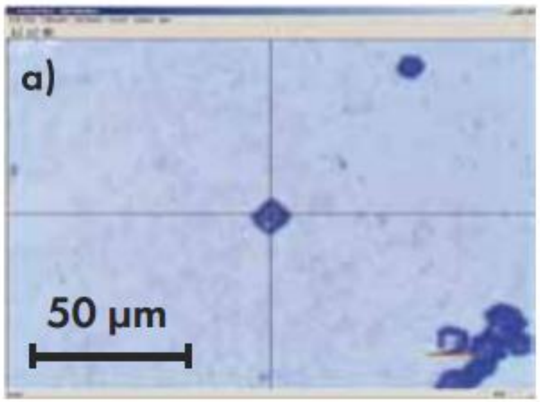
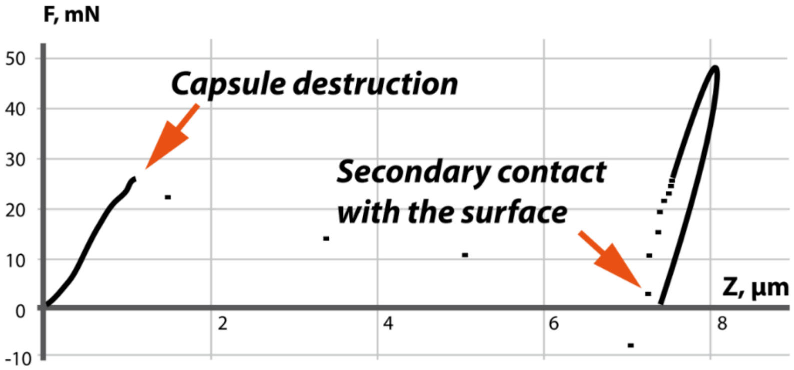
NIOS Nanomechanical Testers:

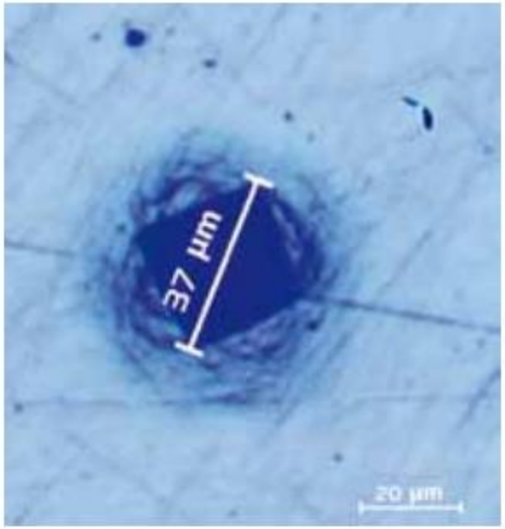
Metal-ceramic and polymer applications

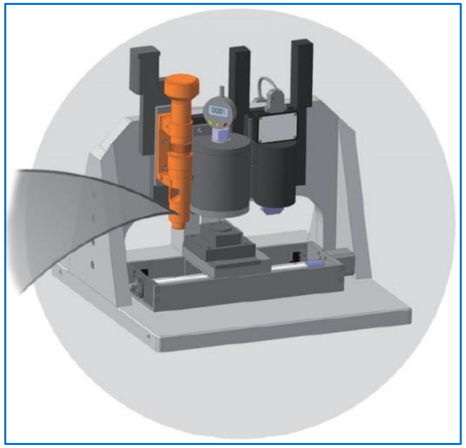
Optical microscopy





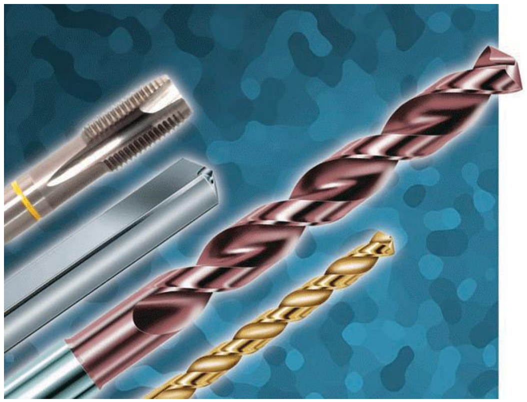
Indenter positioning – displacement between microscope focus point and indenter tip is calibrated with 1 um accuracy



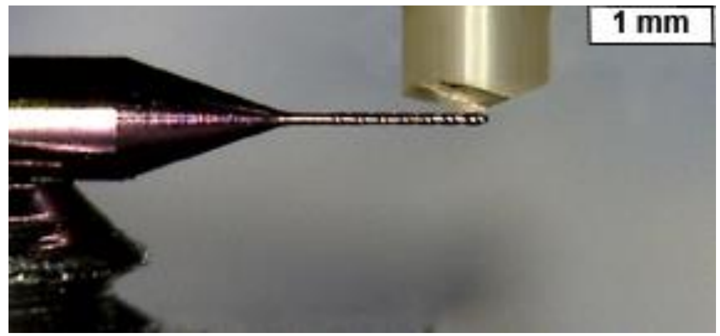


Measuring linear size (and area) over the optical image

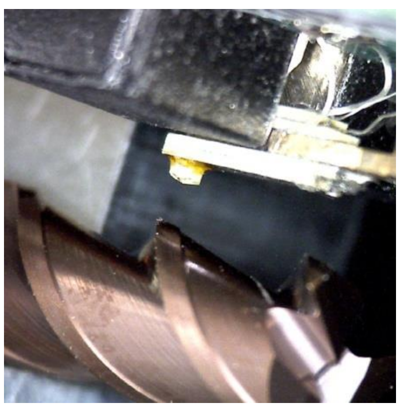
Example: coated cutting tools



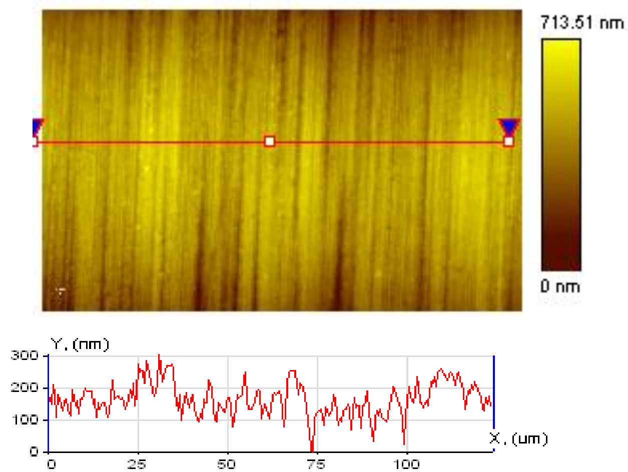
Sample: micro-drill
for electronics industry applications



R_a = 70 nm
RMS = 86 nm
R_z = 232 nm
H = 30,8±6,5 GPa
E = 675±105 GPa
k = 2,15±0,05 kN/m

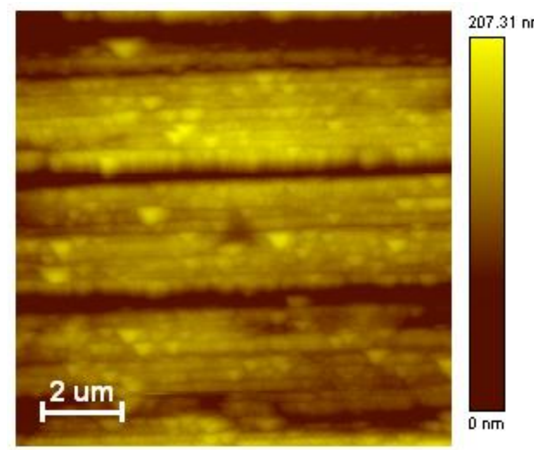


Sample: cutting
edge of the fraise



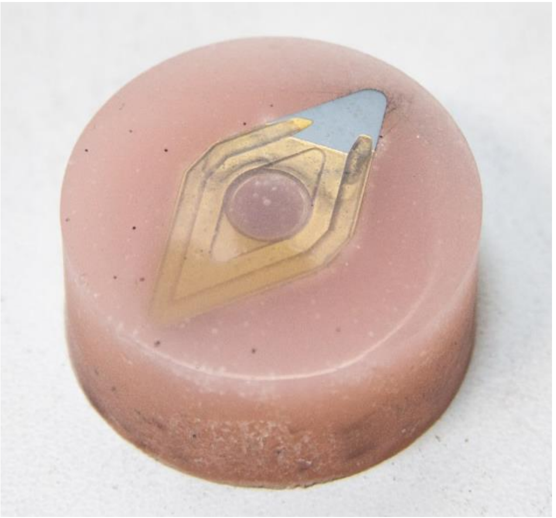
Surface topography

Image of the residual imprint

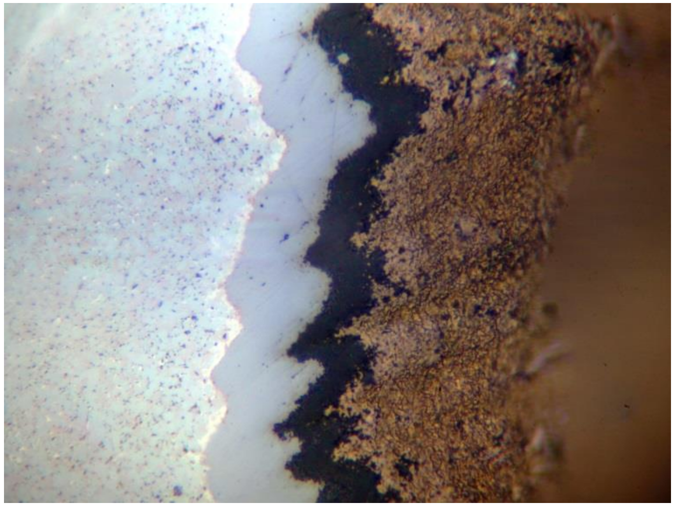


Example: multilayer coating on cutting tools

Sample



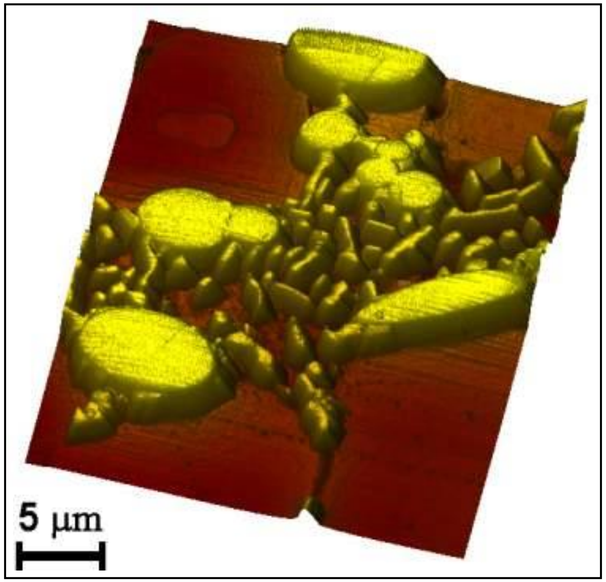
Sample angle polish



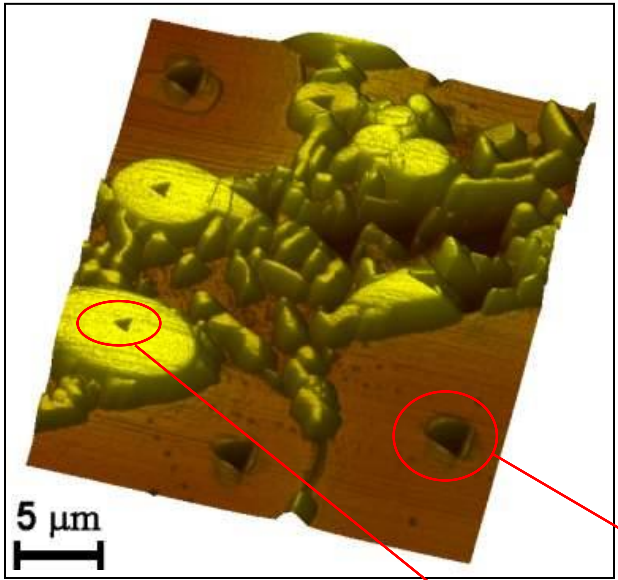
Optical image of layered structure

Material	Hardness, GPa	Elastic modulus, GPa
Hard Alloy	19.0 ± 5.4	410 ± 140
TiCN	17.7 ± 4.5	340 ± 80
Al_2O_3	20.4 ± 4.0	340 ± 35
TiN	10.9 ± 3.3	360 ± 200

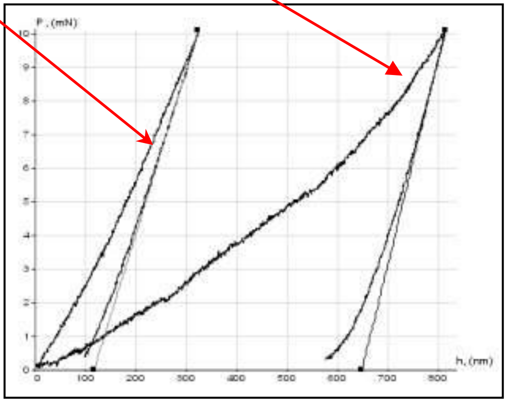
Localized nanomechanical tests



Topography:
before indentation

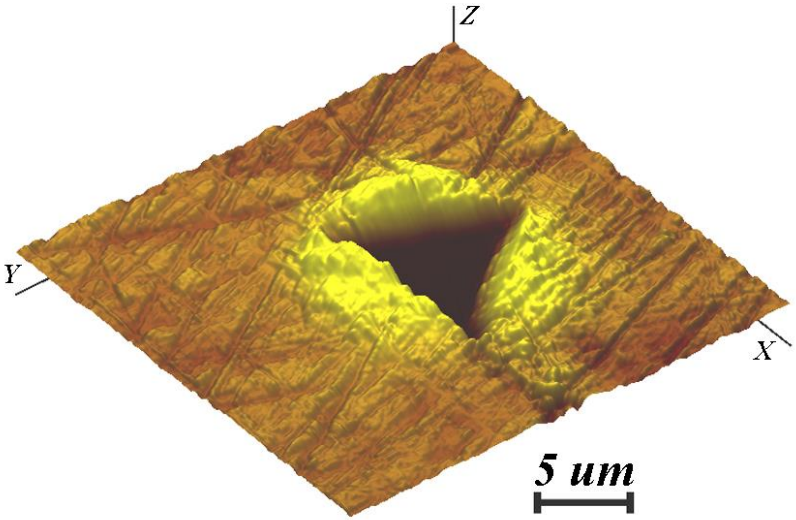


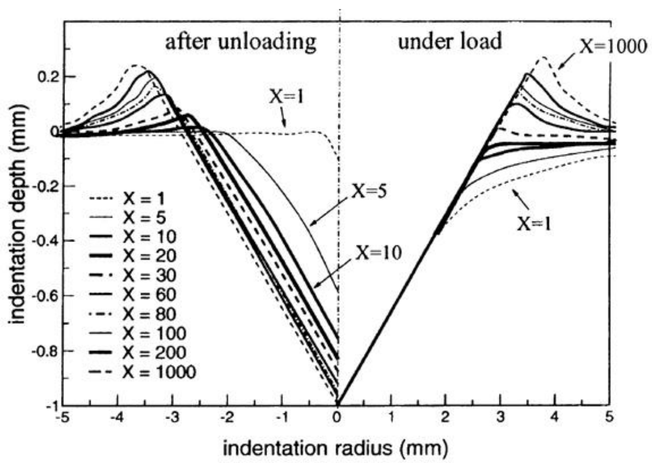
Topography:
after indentation



Sample: aluminum alloy D16

SPM aids indentation: pile-up analysis

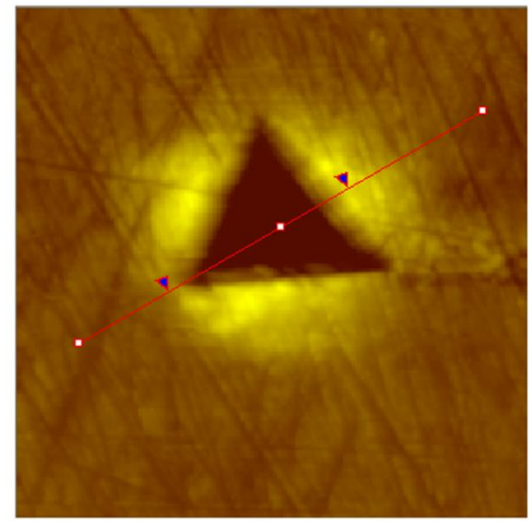


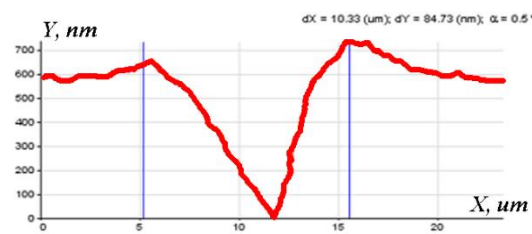


$$X = \frac{E}{\sigma_0}\cot\theta$$

X – rheological factor
E – Young's modulus
σ_0 – yield stress
θ – semi-apical angle of indenter

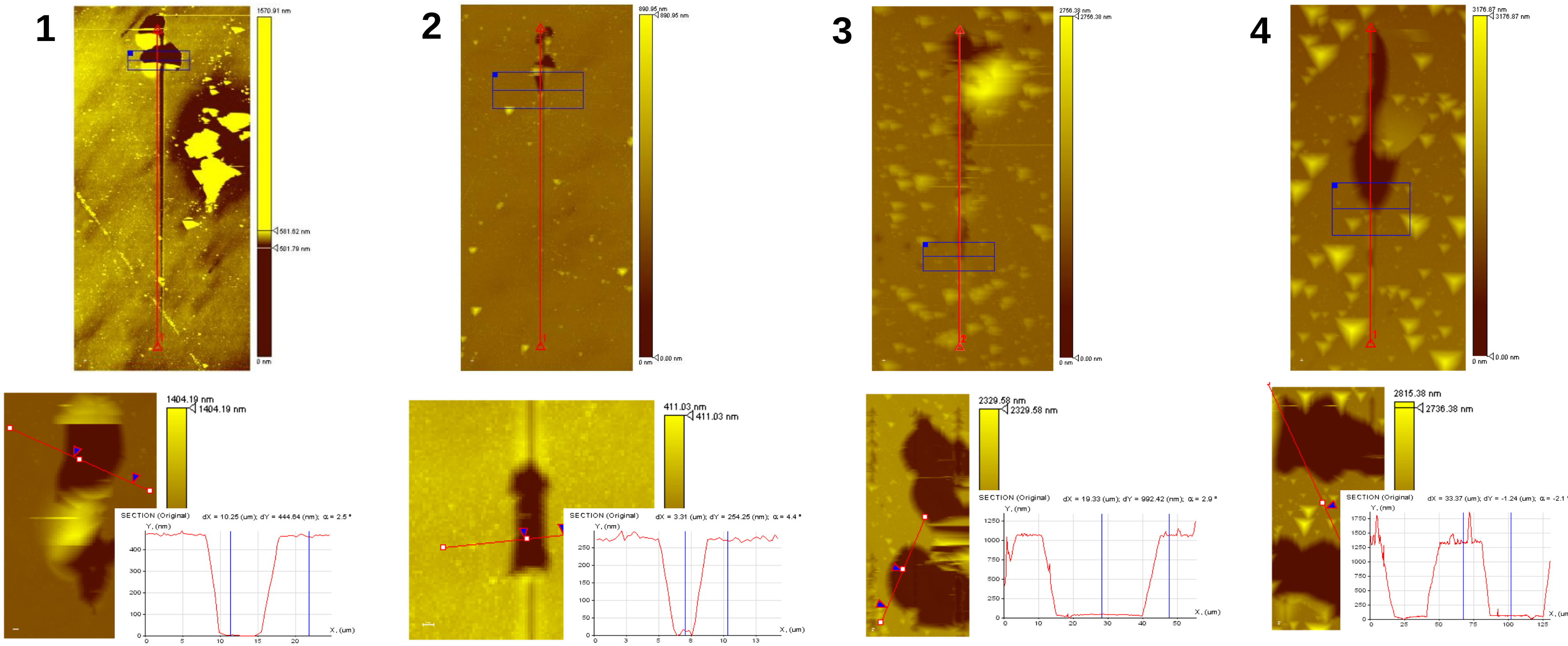
Pile-up analysis on steel 254 reference block HV 0.05





Study of delamination, adhesion and thickness

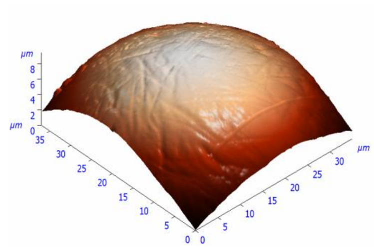
Sample: plasma sputtered diamond-like coating

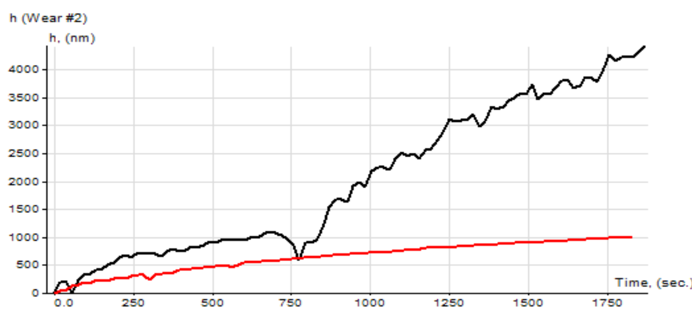


	Thickness, nm	Load at which film is pulled, mN
1	460±20	6,2±0,3
2	265±10	5,9±0,3
3	960±70	11,0±2,6
4	1255±20	20,8±6

Reciprocating wear tests

Spherical Al_2O_3 tip



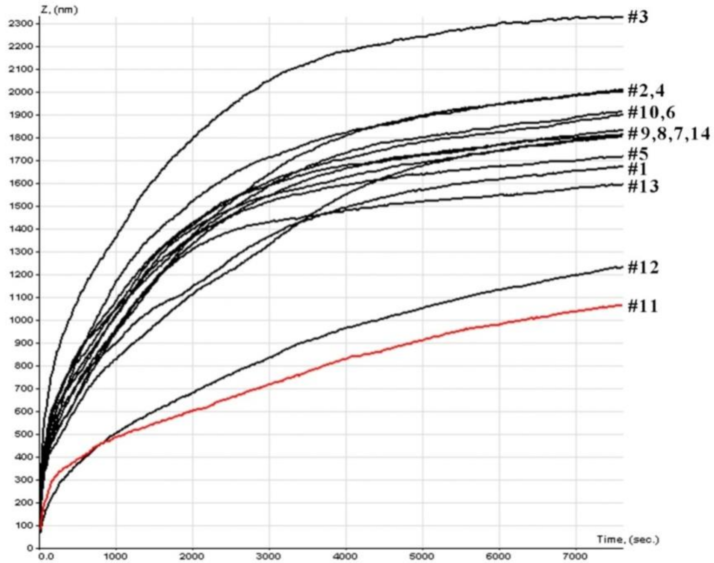


Linear wear rate

Sample	Δh,nm
Duralumin (untreated)	**75±15**
Duralumin with protective mineral coating	**5.9±1.2**

Wear estimation of coatings

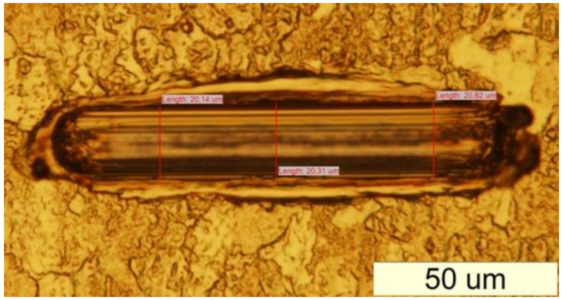
Wear time diagram



$$J = (L \times t \times v)/h$$

L=100 um — stroke length;
v=0,13 Hz — reciprocating test frequency;
t=7600 sec — testing time;
h — indentor penetration depth;

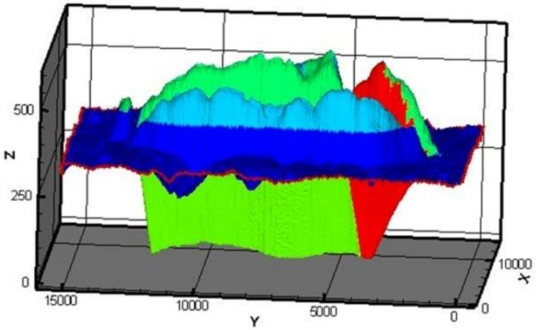
Indirect volume estimation over the optical micro-photograph of the groove



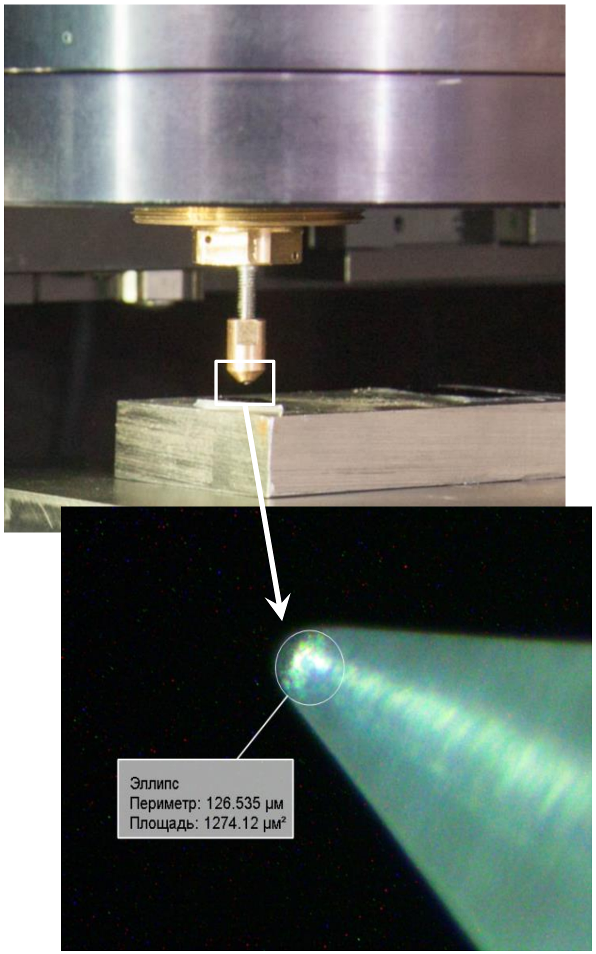
$$V = L\left(R^2 \arcsin\left[\frac{w}{2R}\right] - \frac{w}{2}\sqrt{R^2 - \frac{w^2}{4}}\right)$$

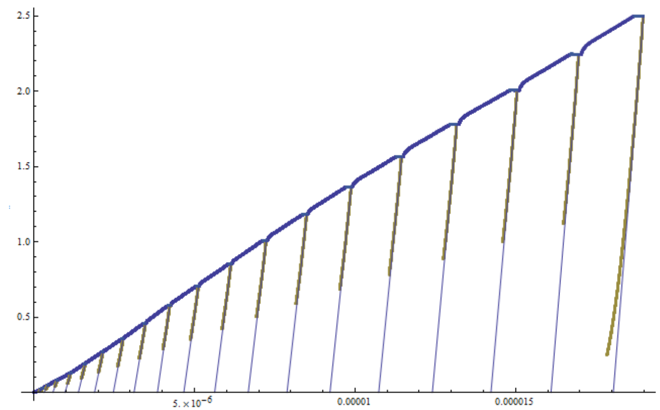
R=17 um — effective stylus radius;
w — width of the residual groove;
L=100 um — groove length;

Direct volume estimation over the SPM image

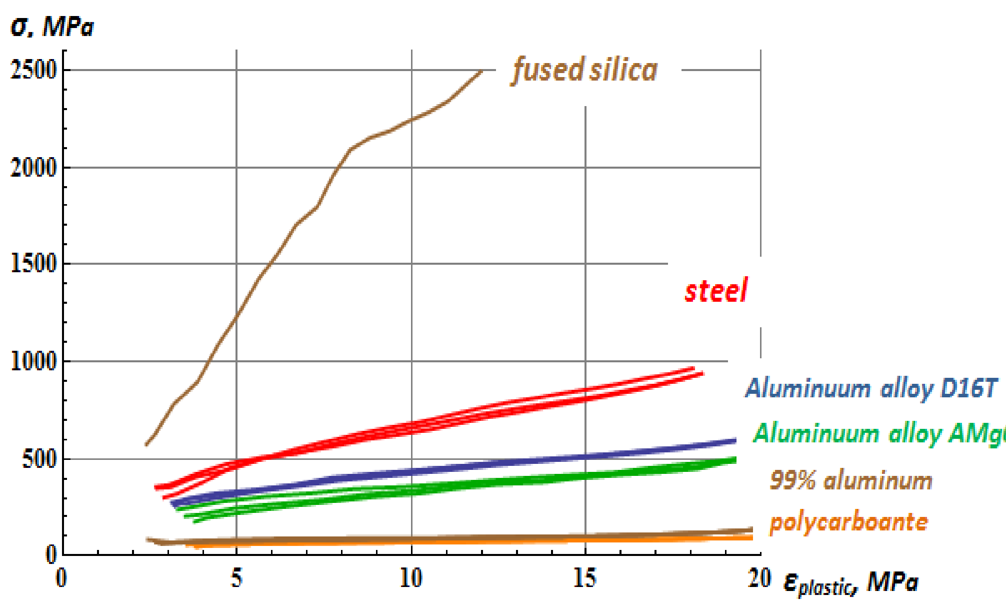


ABI (automated ball indentation)





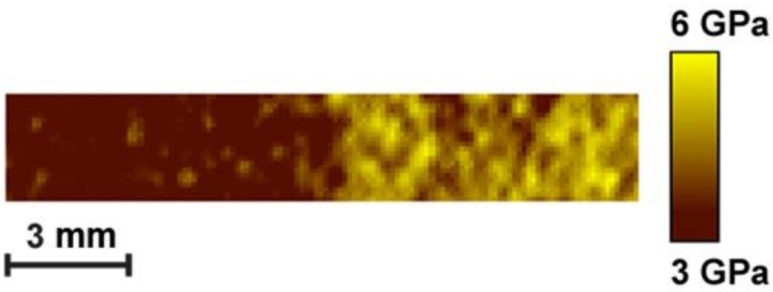
Experimental multicycle loading-unloading curves



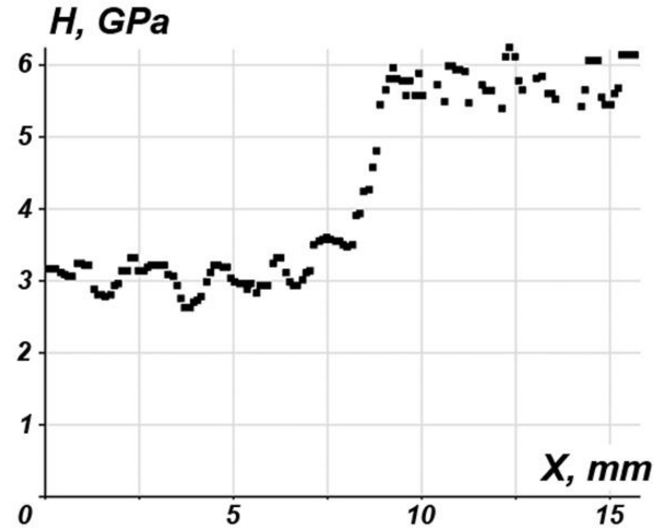
2D: H=f(x, y), static loading

Sample: gradient magnetic alloy of the following composition:

Chemical element	Cr	Ni	Si	Mn	C	V	Fe
Contents, %	16,5	7,5	0,48	1,0	0,08	0,04	remains



Hardness map
of the magnetic zone boundary

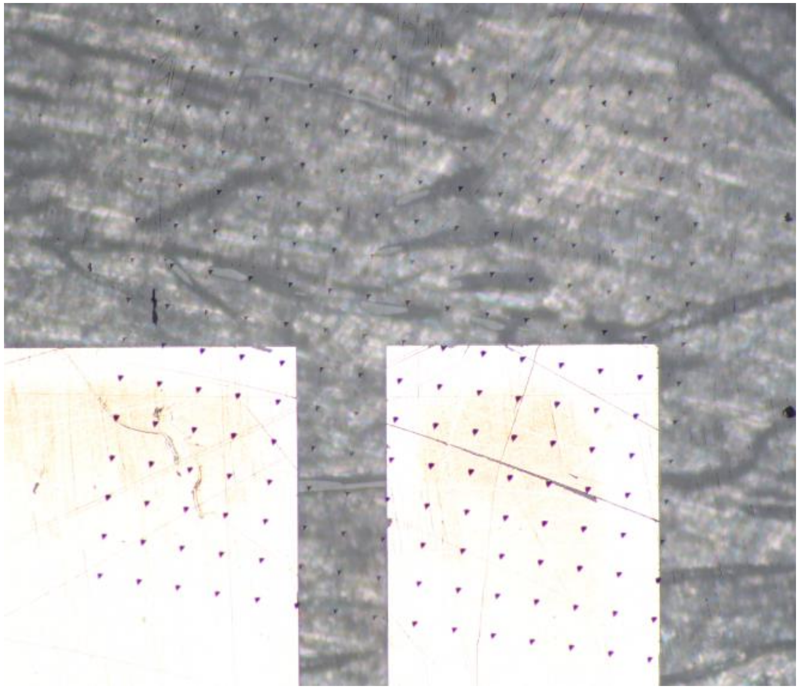


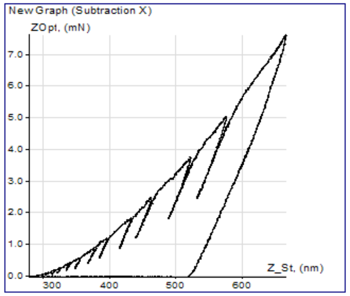
Hardness profile across the
magnetic zone boundary

Mapping 3D: H=f(x, y, z), hardness tomography

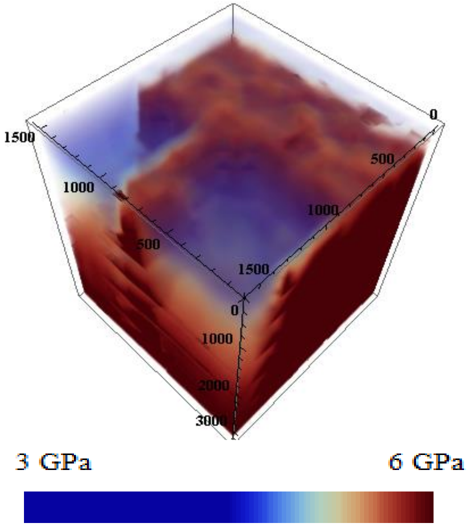
Sample: thin Ag film on glass substrate

Optical micro-photo of the array of PUL indentations



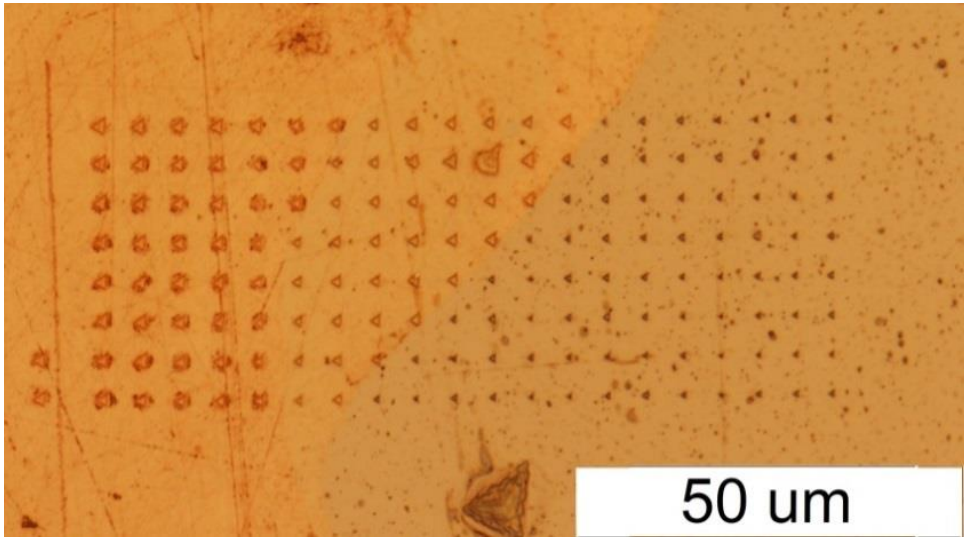


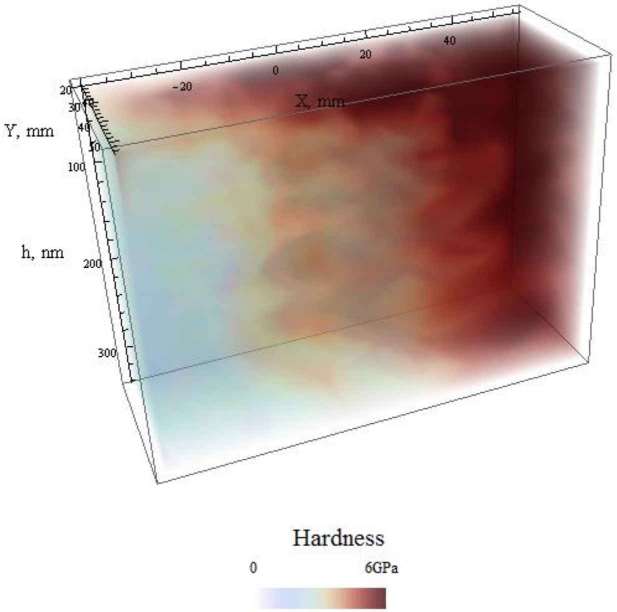
Volumetric quantitative map (tomogram) of hardness



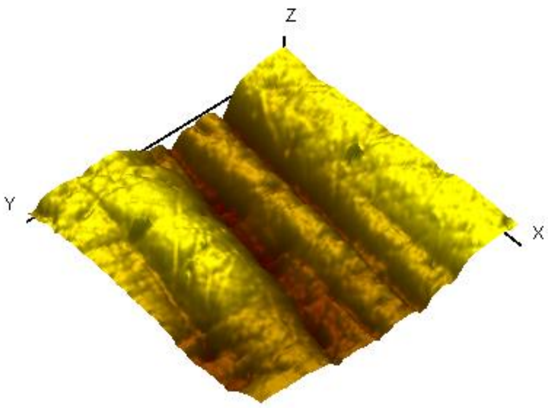
Mapping 3D: hardness tomography

Sample: separation border between copper and Fe-Cr-Al alloy





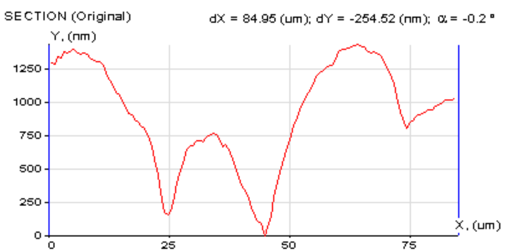
Sample: Polymer material for tooth prosthetics



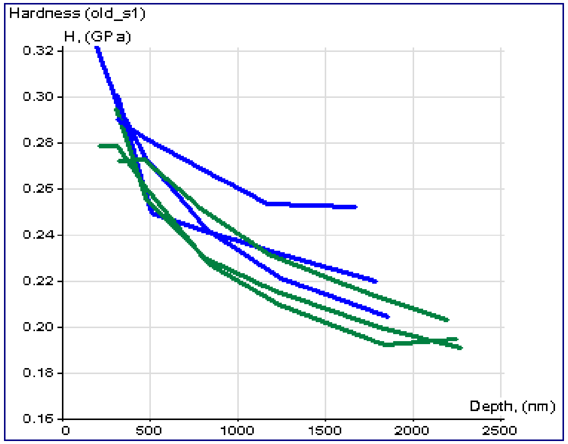
It has been shown that chemical treatment reduces hardness and elastic modulus for 10-15%

Blue lines – before treatment

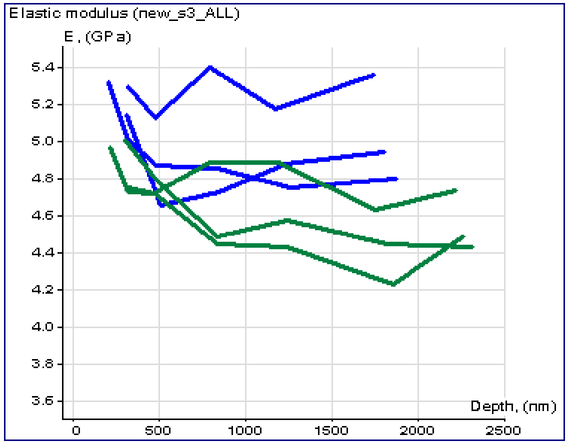
Green lines – after treatment



Surface topography and cross-section profile

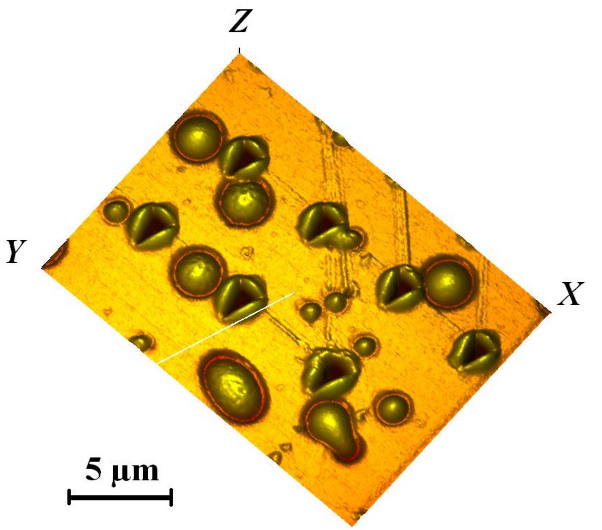


Hardness vs. depth dependency for 3 polymer samples



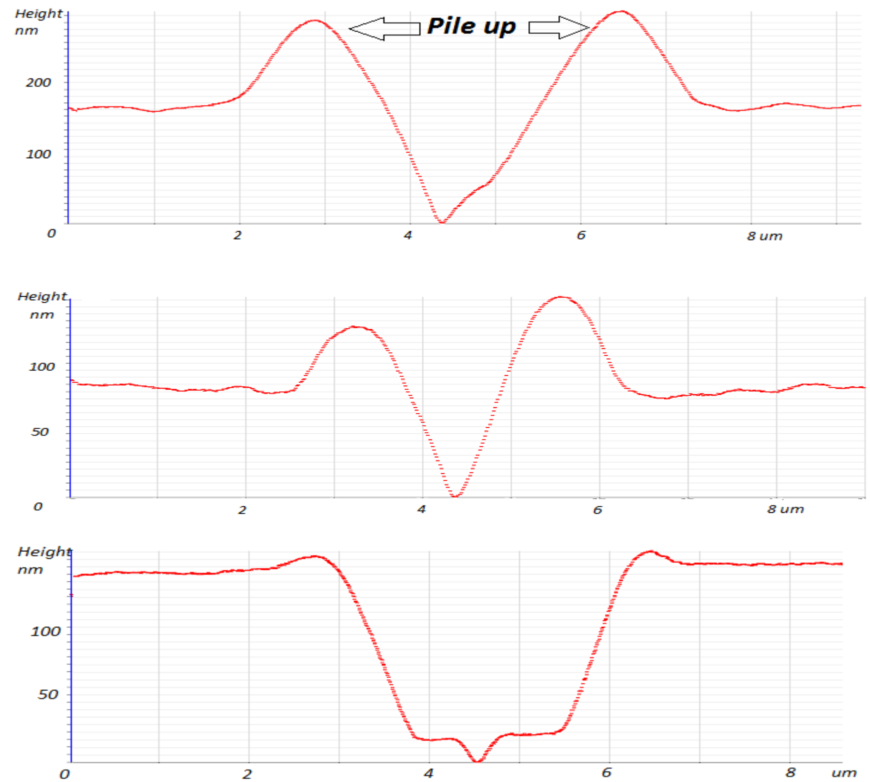
Elastic modulus vs. depth dependency for 3 polymer samples

Sample: Thin film of poly-n-isopropylacrylamide



This polymer exhibits the properties of superabsorbent and is used in pharmaceutical industry during remedies extraction.

Thickness, nm	Hardness, GPa
100	0.5±0.1
5000	1.1±0.3



Height profile of the pNIPAm film. Pile up effect depends on applied forces (F) and film thickness (*h*): a) F=5mN, *h*=5µm; b) F=1mN, *h*=100nm; c) F=2mN, *h*=100nm.

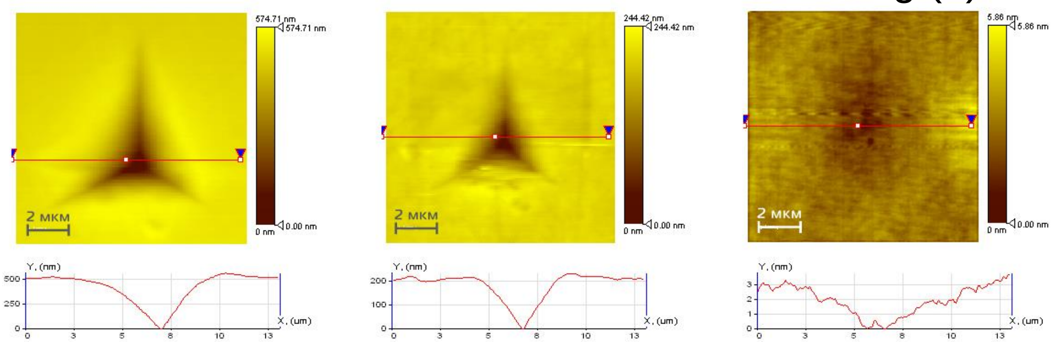
Example: protective polymer coatings

Samples: polymer materials with thermosetting siloxane protective coating

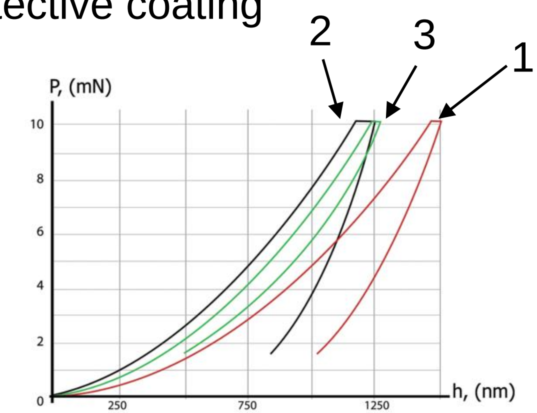
Profile, hardness, elastic properties testing

Polycarbonate (1)

Polycarbonate + coating (3)

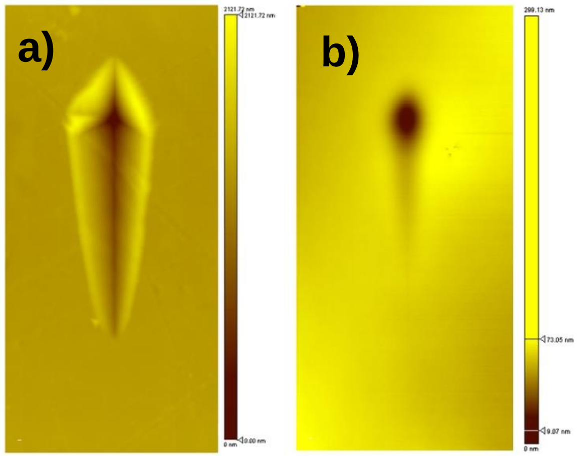


Polymethylmethacrylate (2)



Instrumented indentation loading-unloading curves

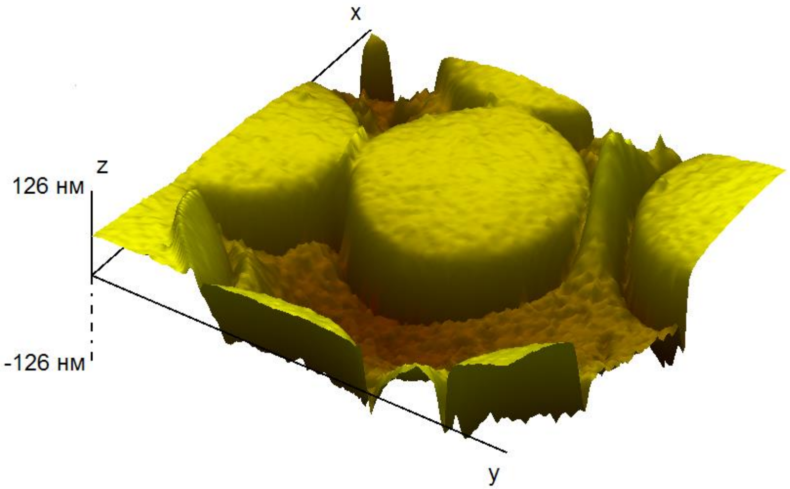
Sample	Roughness, nm	Elastic (Young) modulus, GPa	Hardness, GPa	Elastic recovery rate, %
PC	11,8	2,2	0,27	72
PMMA	4,4	4,5	0,33	60
PC+coating	0,5	1,4	0,68	99



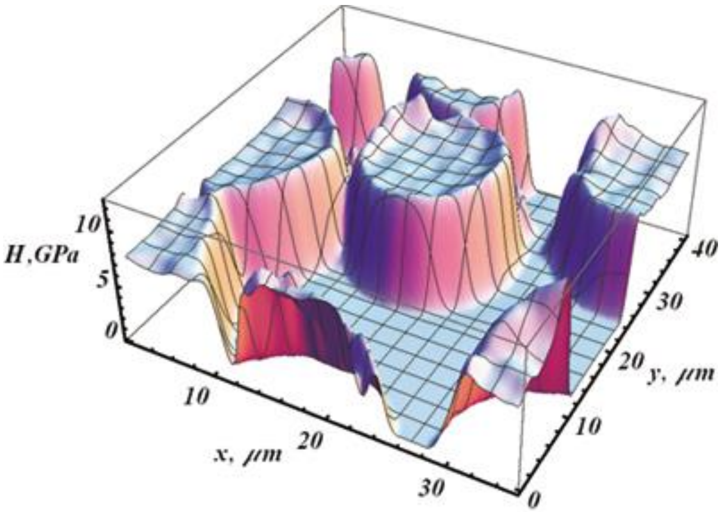
Topography image after progressive scratch test (0 to 30 mN): PC (a) and PC+coating (b)

Mapping 2D: H=f(x, y), dynamic loading

Sample: glass fibers in the polymer matrix



Surface topography



Hardness map

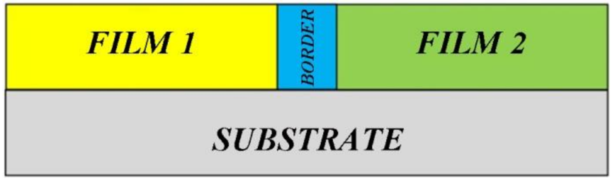
$$\frac{H}{E^2} = \frac{F}{\pi}\left(\frac{f_0}{\Delta f}\ \frac{1}{k}\right)^2$$

F - load force,
Δf - shift of the resonance frequency,
f_0 - the resonance frequency of free probe
k - the dynamic stiffness.

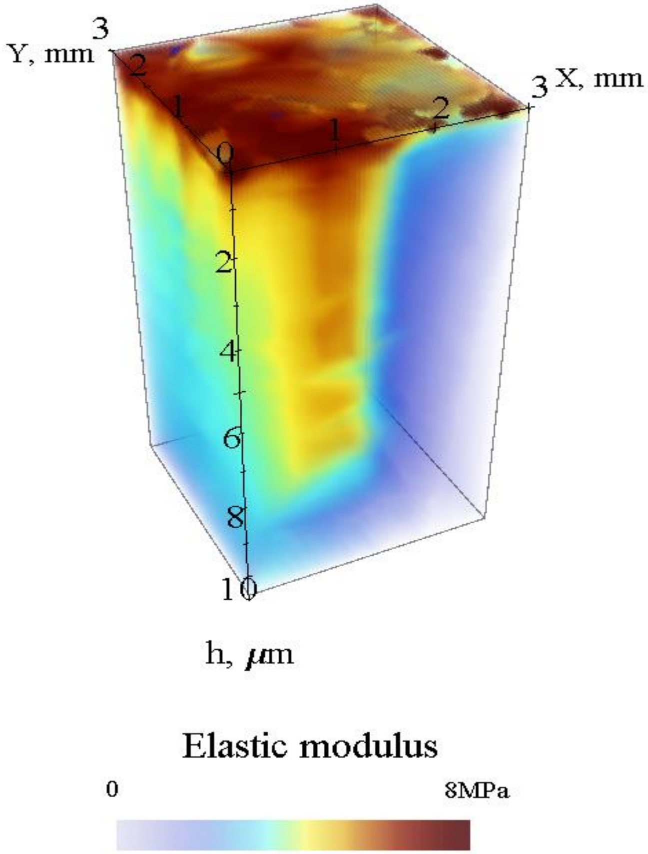
Using this formula allows us to calculate H/E^2 (and accordingly, *H* or *E* if at least one of these quantities is known) as a function of depth or surface coordinates.

Mapping 3D: hardness tomography

Sample: transition area between 2 polymer coatings on polydimethilsiloxane (PDMS) substrate

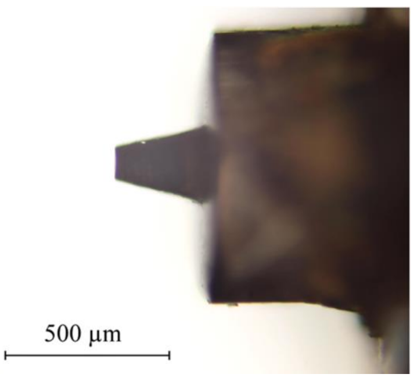


Hardness tomography technique is based on combination of PUL or DMA method with precise indentor positioning over the regular XY grid on the sample surface

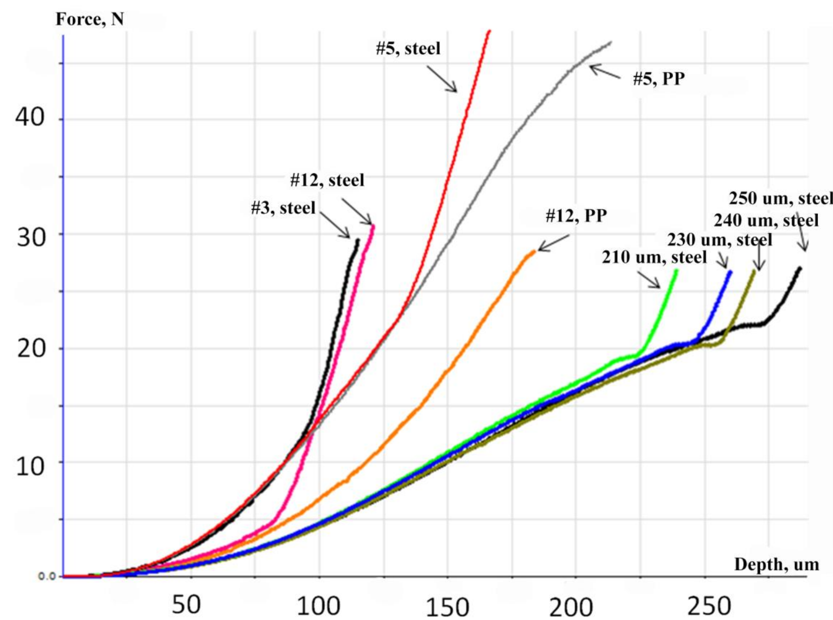


Samples: polyethylene multilayer film with thickness 50-300 um

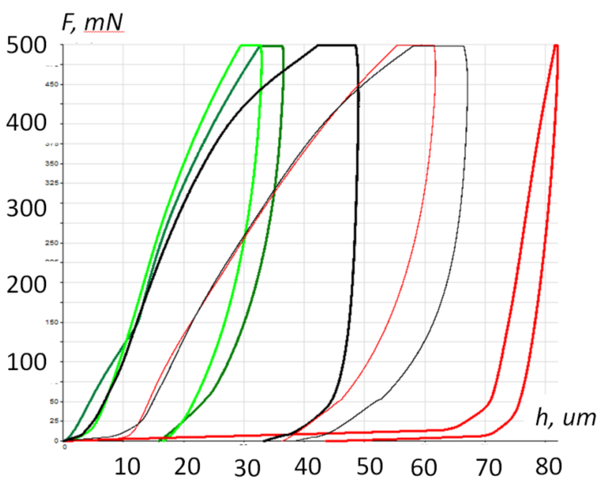
Task: testing the mechanical strength and resistance to puncture of films on the substrates of polypropylene and steel



Diamond flat punch indentor



Loading curves during puncture test with flat punch indentor showing comparison of mechanical strength of polymer films



Loading-unloading curves obtained with Berkovich diamond indentor

future's
in the making

Thank you!