Zwick / Roell Intelligent Testing

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Micromechanics of thin films

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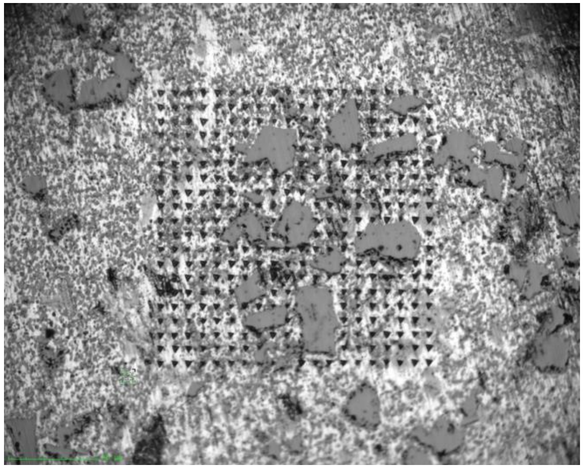
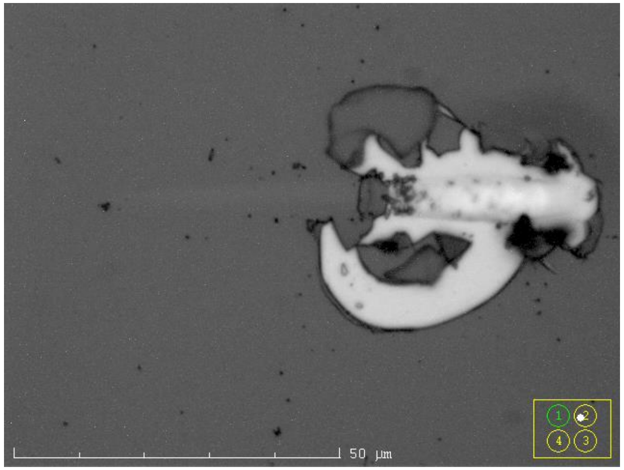
ASMEC Advanced Surface Mechanics GmbH





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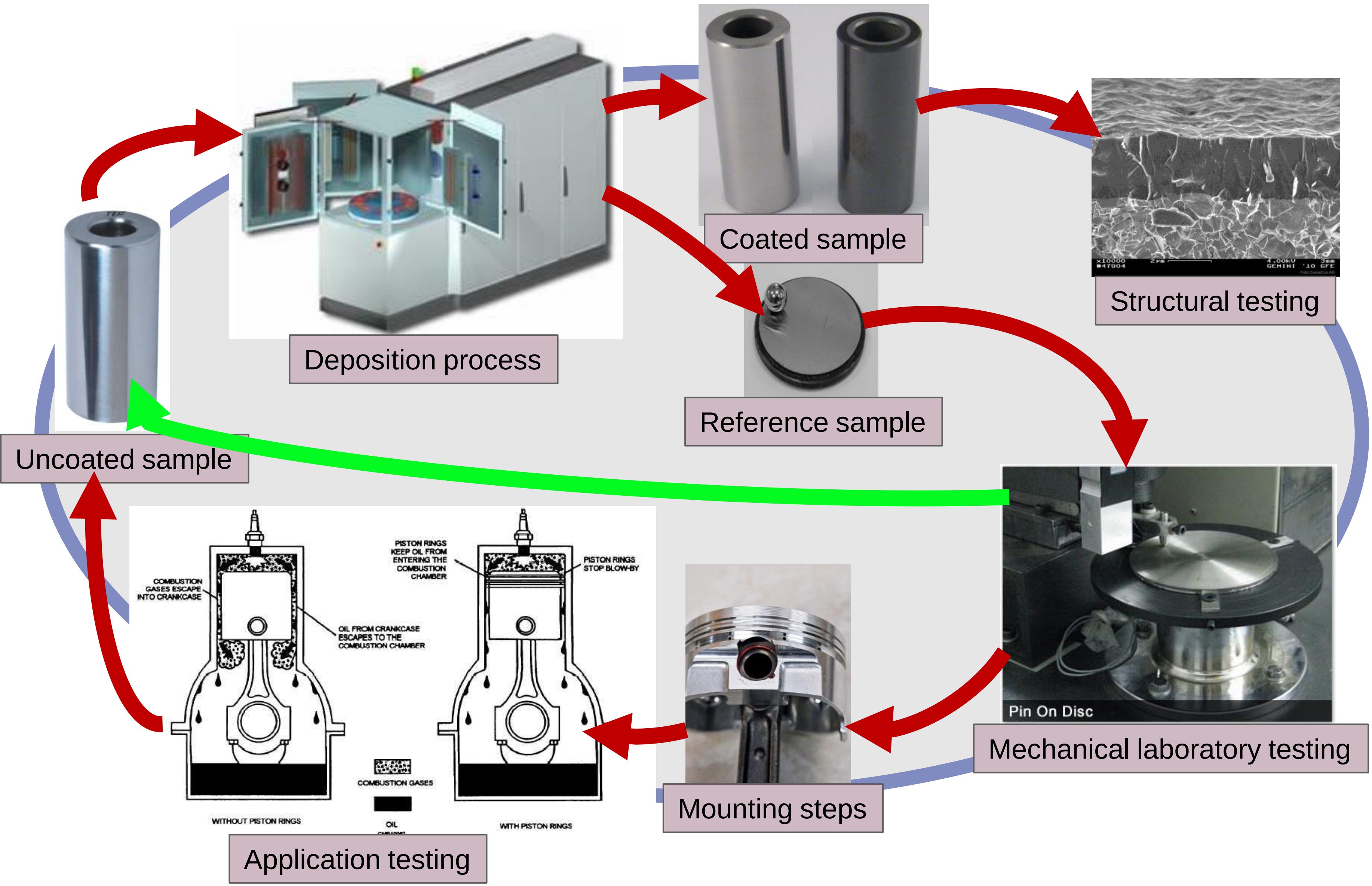
1) Motivation for mechanical testing

Iterative development process



Trial and error development process in a multi parameter space

Example: Double layer coating

Testing of:

- 10 materials for each single layer (10 x 10)
- 10 process parameters per material
- 10 film thickness combinations

= 10 x 10 x 10 x 10 combinations

= **10000** samples

Problems of surface mechanics for coated systems

The search for the optimum coating material is
time consuming and expensive.

Mechanical parameters of thin coatings and small structures are
difficult to measure.

Laboratory measurements and simulations often
differ from the conditions in a real application.

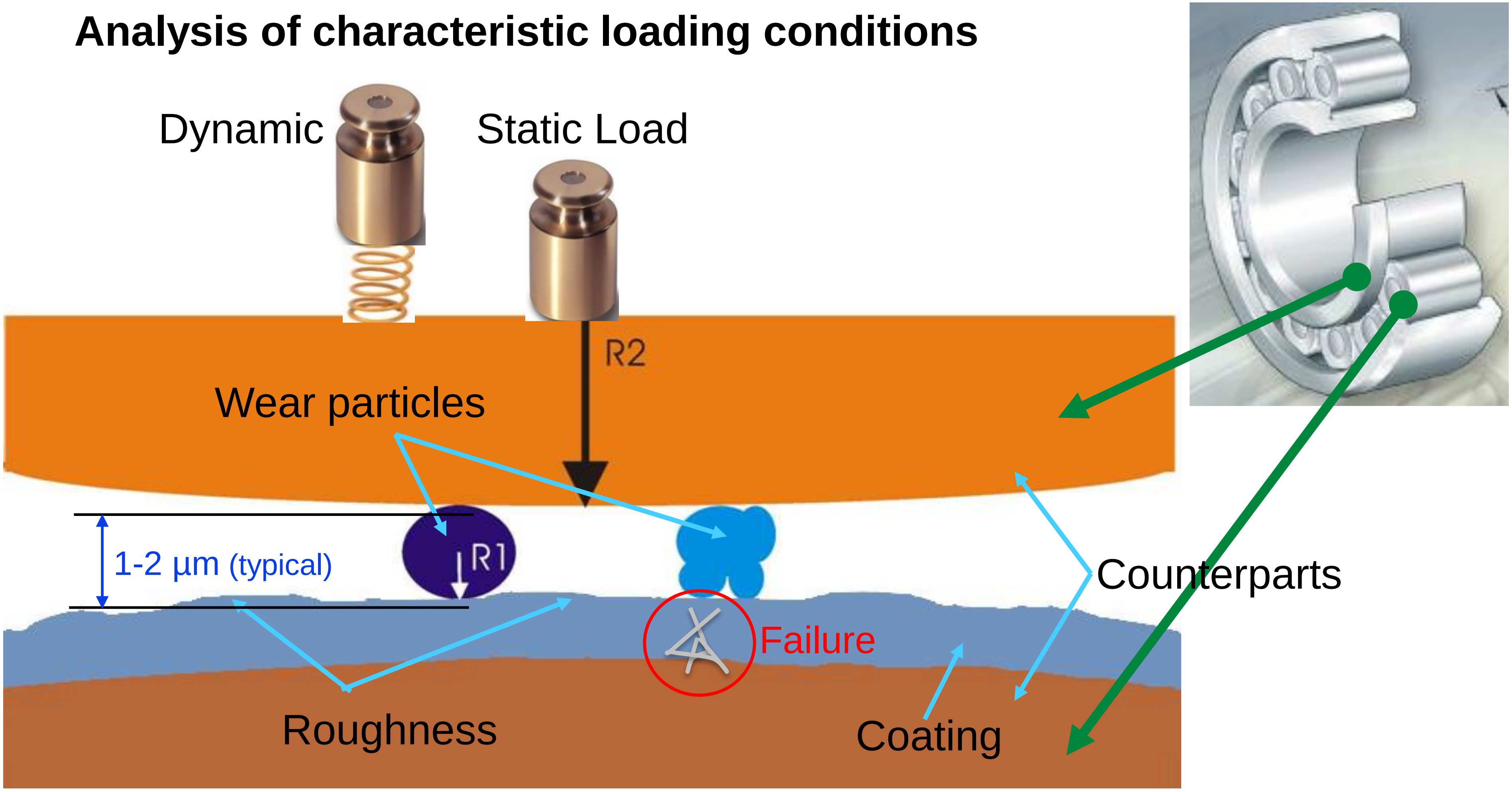
Steps for a reduction of the development effort

1) Identify the most critical conditions in the application, which cause failures

2) Identify the most critical mechanical (physical) material parameters

3) Combine tests with a higher level of modelling and calculations

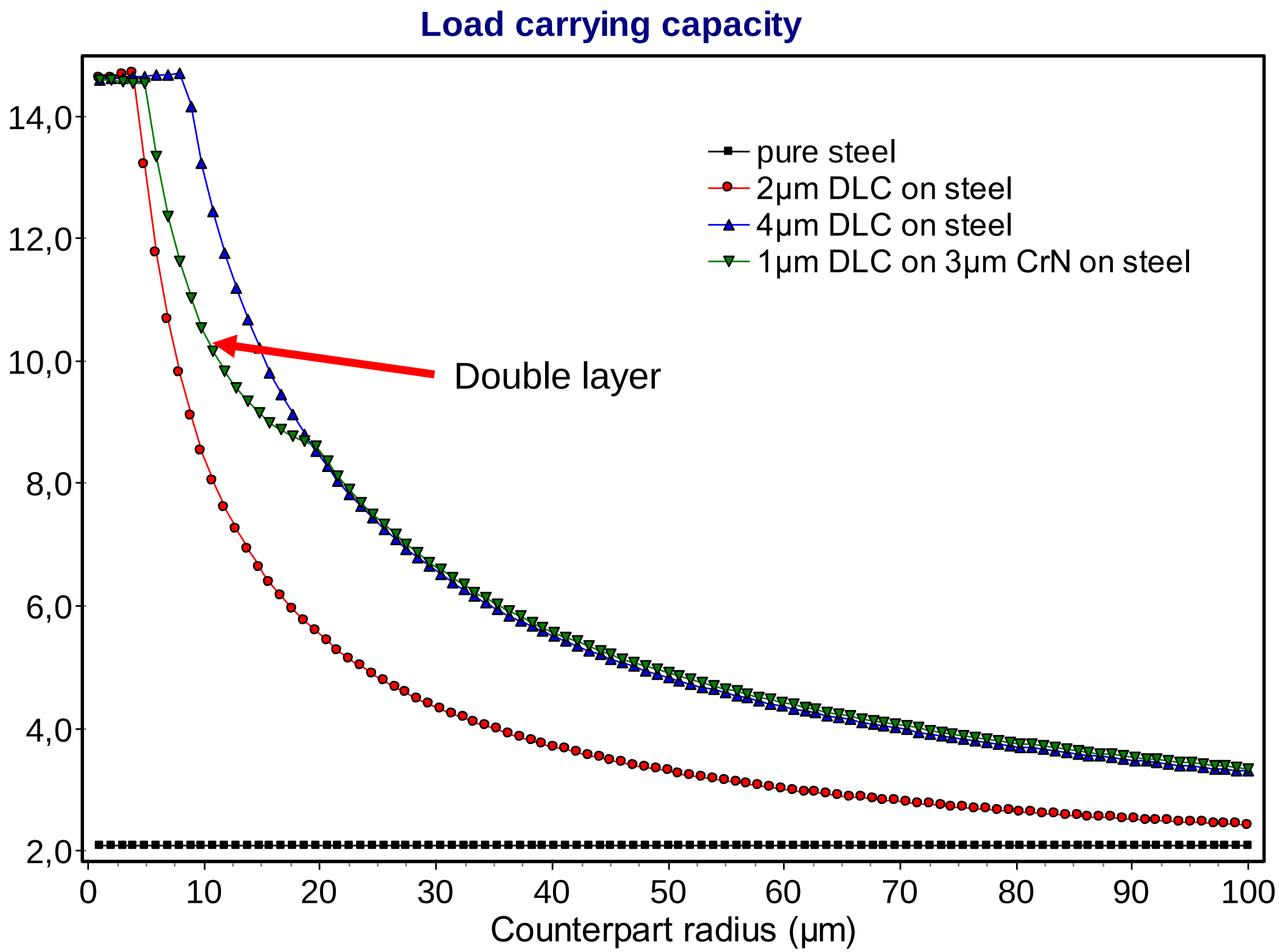
Analysis of characteristic loading conditions



To understand the failure reasons one has to go down to the dimension of roughness and wear particles → This requires high resolution.

Example for modelling

Load carrying capacity of different coating systems on steel



Input parameter required:
E – Young's modulus
v – Poisson's ratio
Y – Yield strength
(T – Tensile strength)

	Steel	DLC	CrN	
E	200	270	300	GPa
v	0.3	0.2	0.25	
Y	2	15	8	GPa

2) Mechanical material parameters

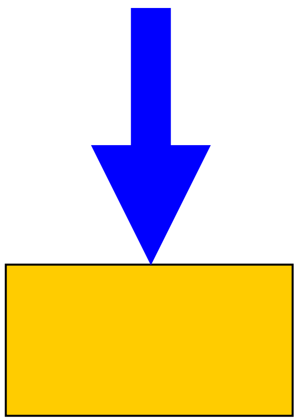
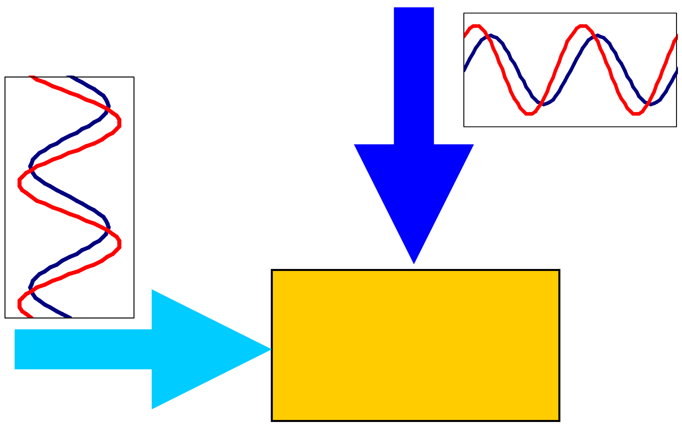
Mechanical behavior	Parameter	Test methods for coatings
Elastic behavior	Young's modulus, Poisson's ratio	Nanoindentation+LFU, ultrasonic surface waves, AFAM, impulse excitation technique
Plastic behavior	Hardness, Yield strength, Stress-strain-curve, hardening exponent	Nanoindentation+LFU
Brittle behavior	Fracture toughness, tensile strength	Nanoindentation+LFU, 4-point bending
Time dependent behavior	Creep, fatigue resistance, strain rate dependence	creep test, impact test, cavitation test, fatigue test (cyclic contact loading)
Frictional and wear behavior	Friction coefficient (not intrinsic) Wear coefficient (not intrinsic) Stribeck curve (not intrinsic)	Friction test, wear test, nanoindentation+LFU
Adhesion	Adhesive strength Scratch resistance (not intrinsic)	Scratch test (LFU), peel test, centrifuge test, cavitation test, Rockwell test

For all parameter it also has to be considered:

- Internal stress
- Dependency on temperature
- Dependency on sample homogeneity: failure density, gradients, thickness constancy, roughness

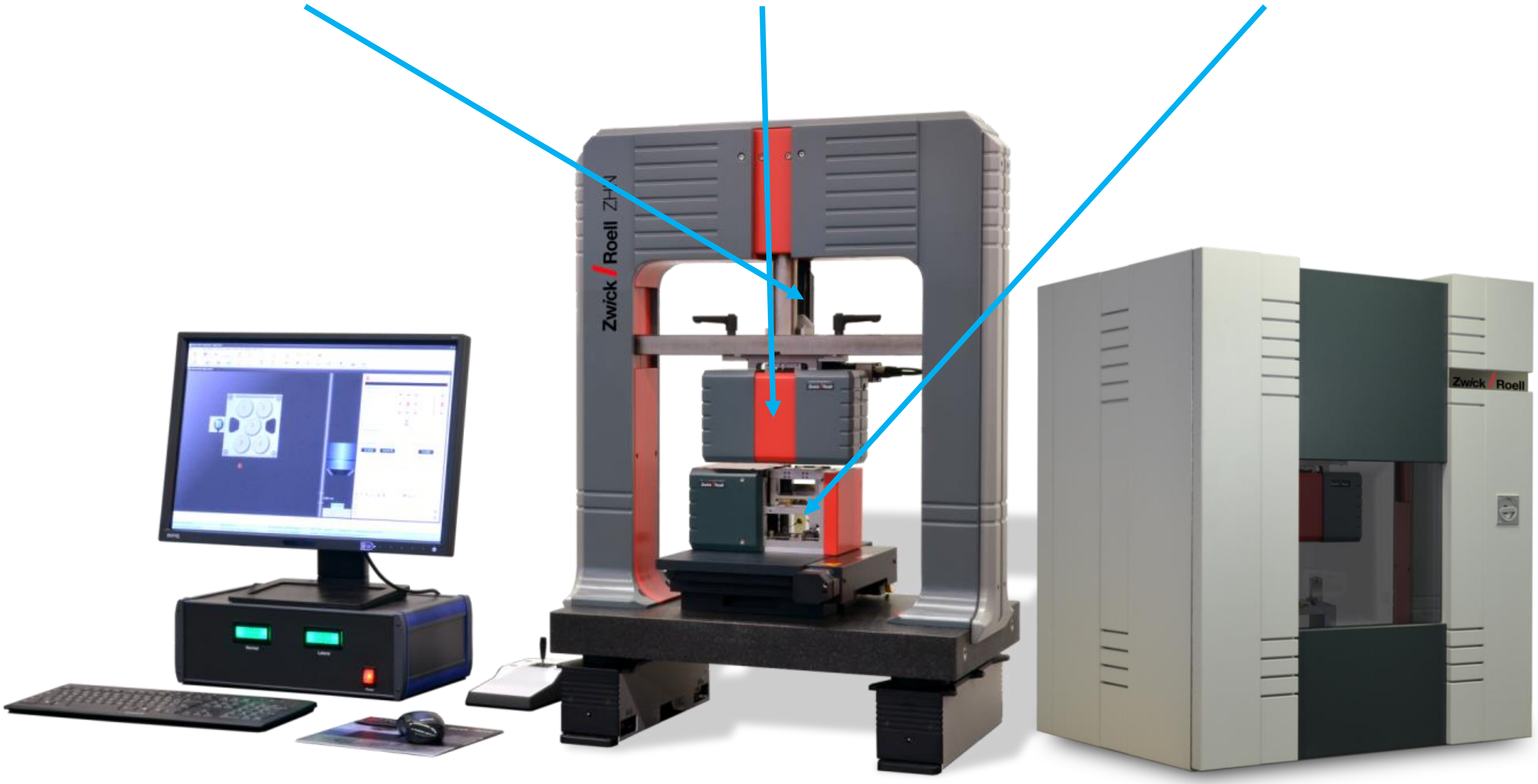
LFU = Lateral Force Unit

Closer to application conditions

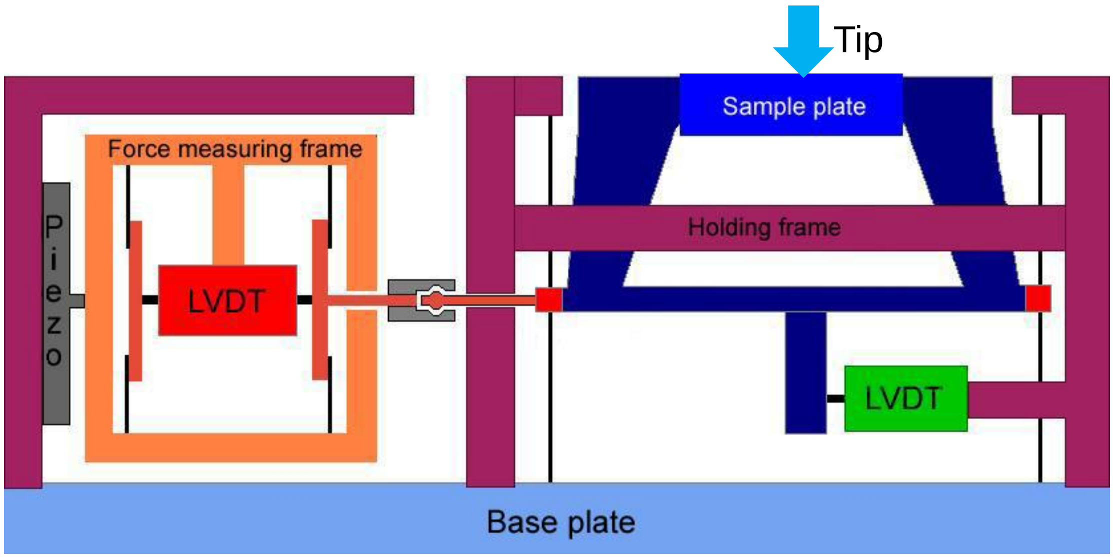
Default Nanoindenter	Universal nanomechanical tester
	
1 degree of freedom: - Normal load-displacement-curve	4 degrees of freedom: - Normal load-displacement-curve - Lateral load-displacement-curve - Oscillation normal (dynamic mode) - Oscillation lateral (dynamic mode)

ZHN – Zwick Universal Nanomechanical Tester

Optics behind the head Normal force unit NFU Lateral force unit LFU

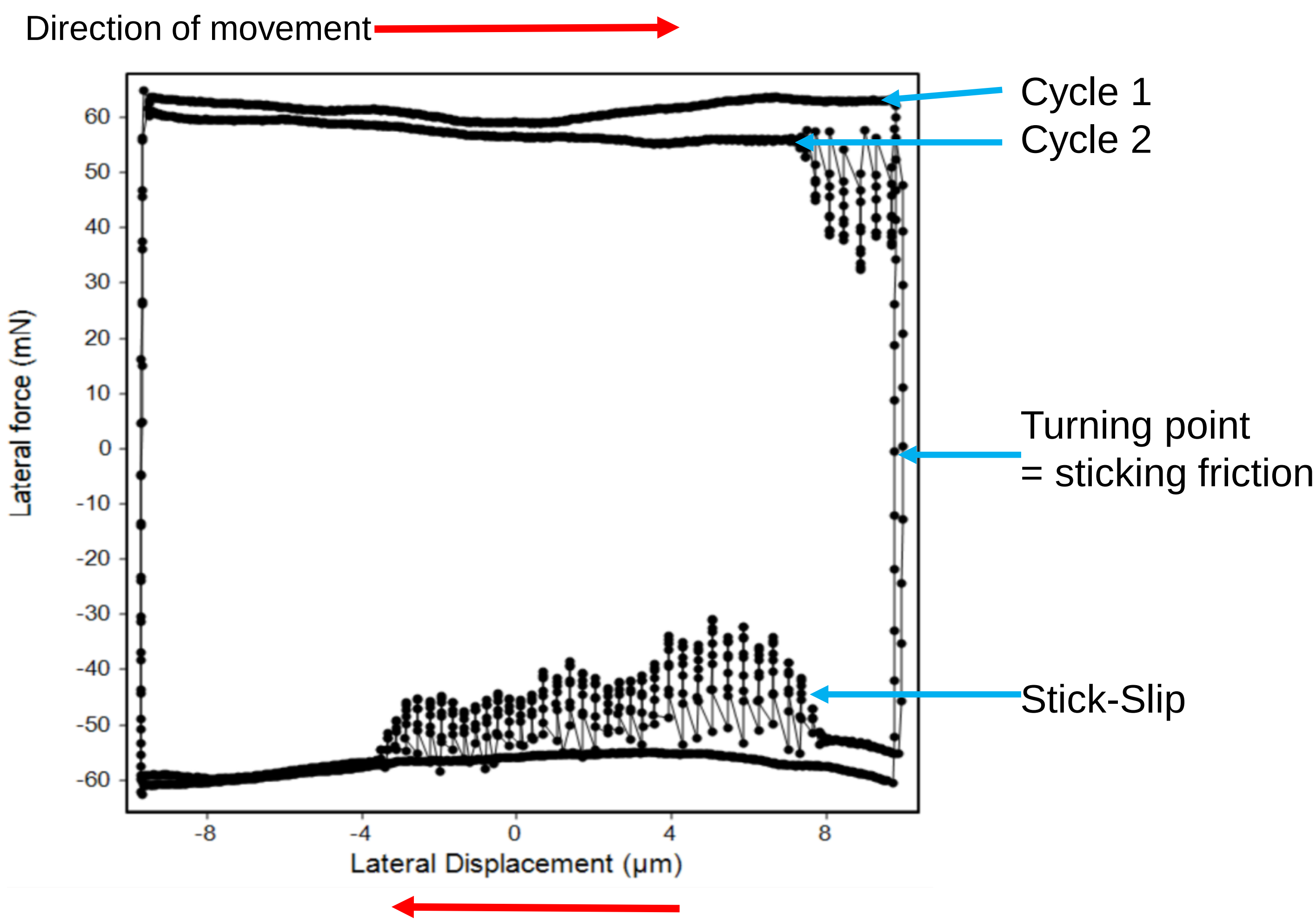


Lateral force unit – the additional component



- Nanometer resolution like in normal direction
- The sample is moved laterally (not the tip)
- High stiffness in normal direction
- No height change during lateral movement
- Force generation independent on movement

- A force can be applied and measured without any movement of tip or sample
- No rolling motion of the tip due to bending of the indenter shaft
- Transition sticking- sliding friction highly resolved



Example: Diamond on diamond in water

Hardness and modulus measurements by nanoindentation

Depth sensing indentation = instrumented indentation = IIT

Standardized since 2002: DIN EN ISO 14577 Part 1-3,
2007: Part 4: Coatings

Metallic materials - Instrumented indentation test for hardness and materials parameters

Macro-range:	Force 2 N < F < 30.000 N
Micro-range:	Force F < 2 N
Nano-range:	Depth h < 200 nm

Indenters:	Pyramids	Vickers	(4 edges)
		Berkovich	(3 edges)

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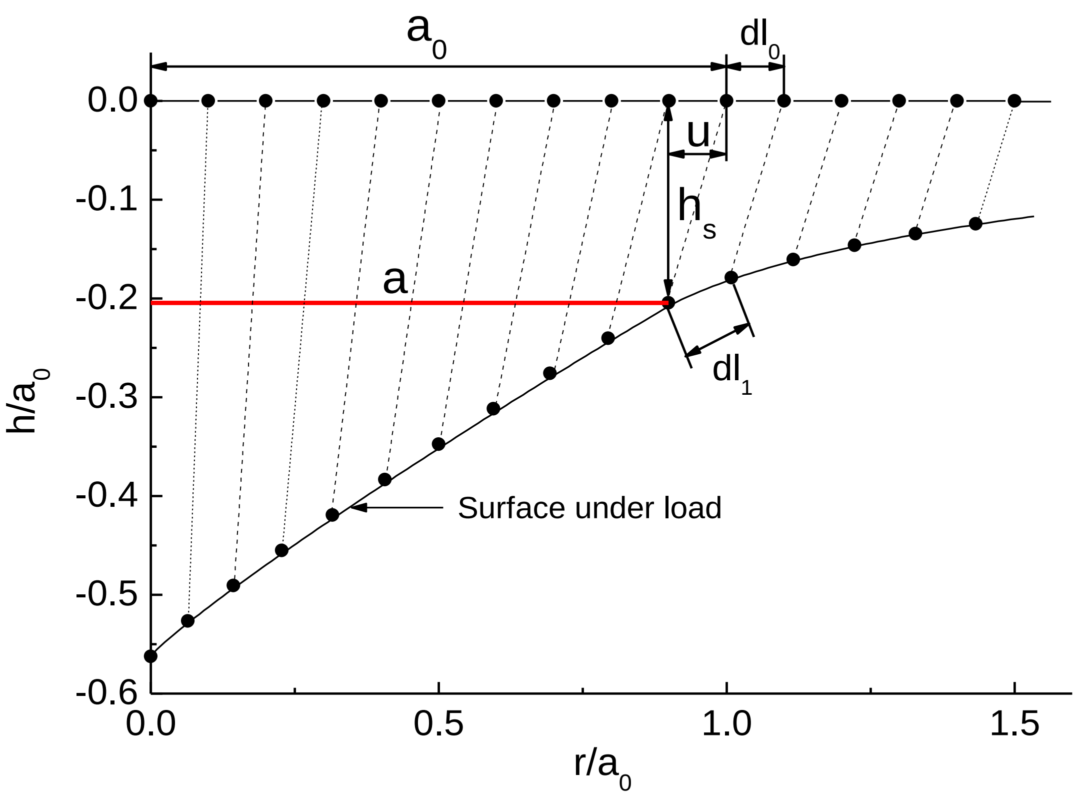
Additional correction: radial displacement correction

Included in the new revision of the standard ISO 14577



Example for influence:

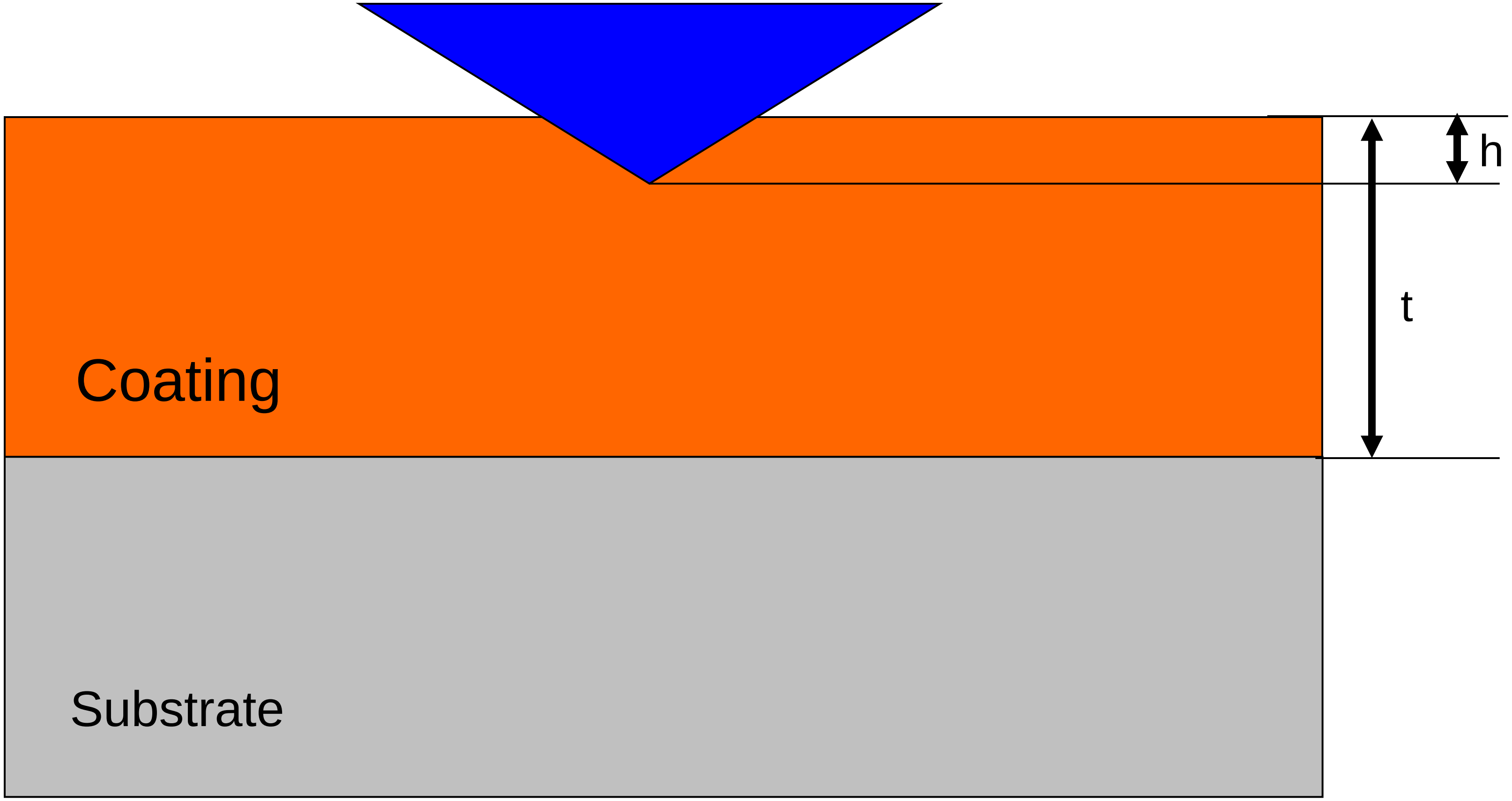
	with	without correction
H:	30 GPa	→ 31,5 GPa
E:	400 GPa	→ 413 GPa

$$u_r = \frac{(1-2\nu)(1+\nu)}{2}\frac{F}{E\cdot a^2}\cos(\arctan(\frac{h_0}{a}))$$

T. Chudoba, N. M. Jennett, Higher accuracy analysis of instrumented indentation data obtained with pointed indenters, J. Phys. D: Appl. Phys. 41 (2008)

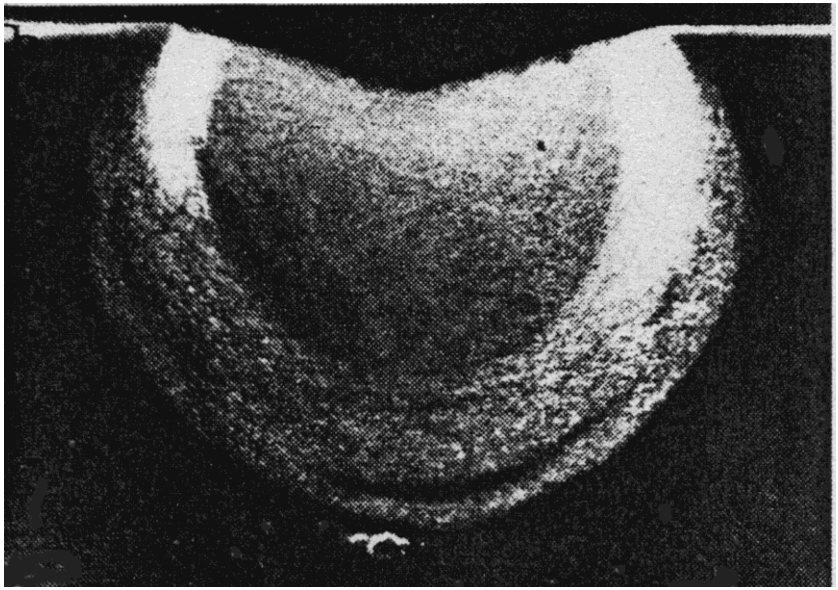
Upper depth limit for the hardness of coatings

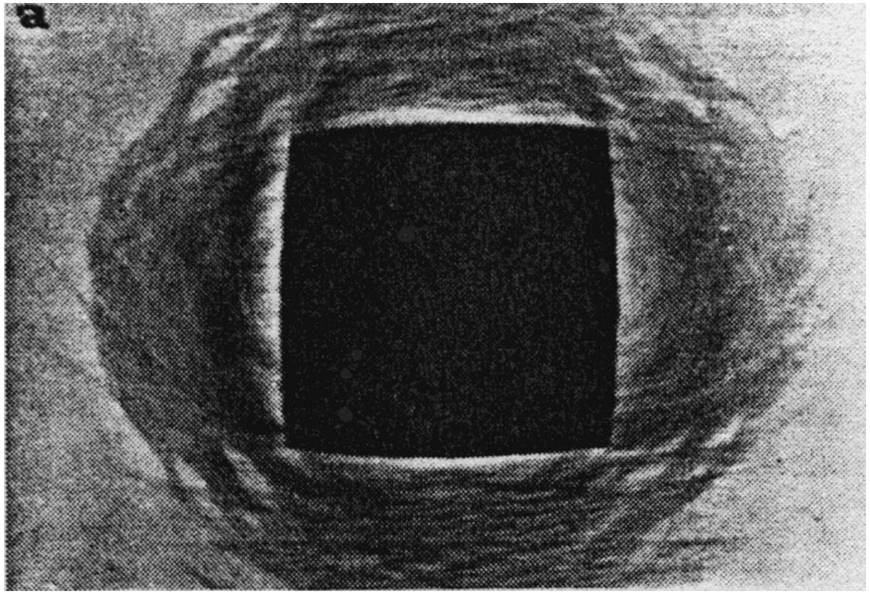
One tenth rule $h < \frac{t}{10}$



Reason for depth limit

The plastic zone is much larger and deeper than the indent.
Therefore the information in the load-displacement curve comes from a depth of up to10 times the indentation depth.



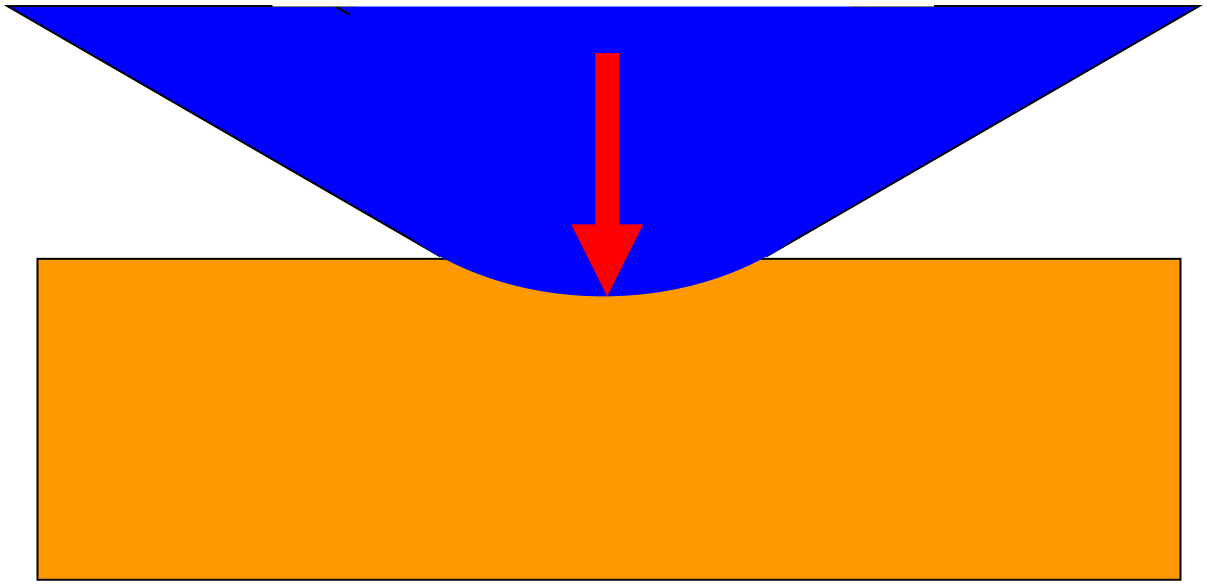


Presentation of the plastic zone in steel using a special etching technique

Lower depth limit for hardness tests due to tip rounding

Minimum indentation depth for comparable hardness results:
20% of tip radius

A Berkovich tip has a typical tip radius between 100 – 300 nm



Minimum depth for hardness tests:

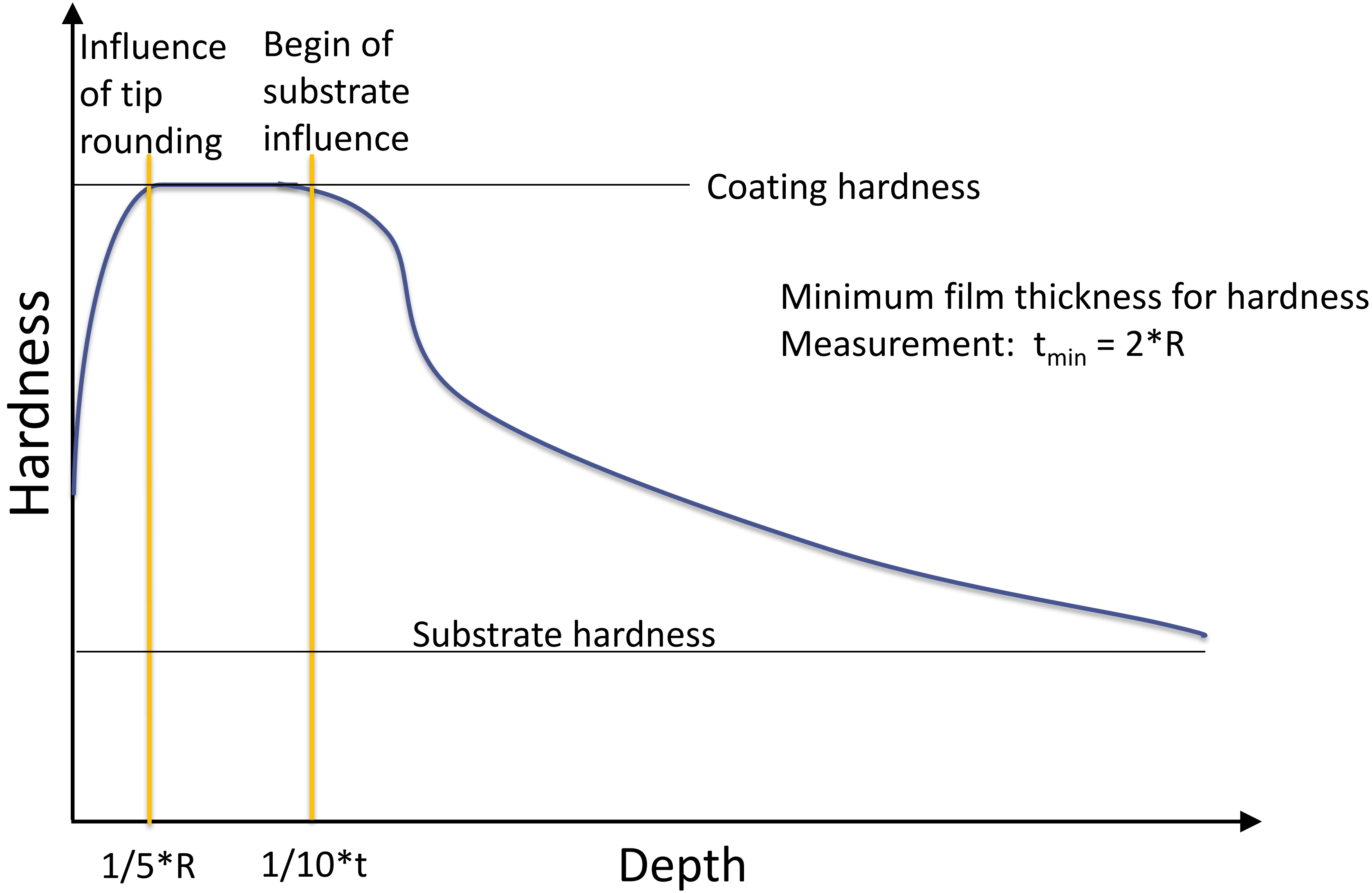
$h_{min} = 0.2*R$

Minimum film thickness for **hardness** measurements in dependence on tip radius (not for modulus)

Tip radius (nm)	Minimum film thickness (nm)
50	100
75	150
100	200
150	300
200	400
250	500
300	600

typical

Substrate influence on hardness



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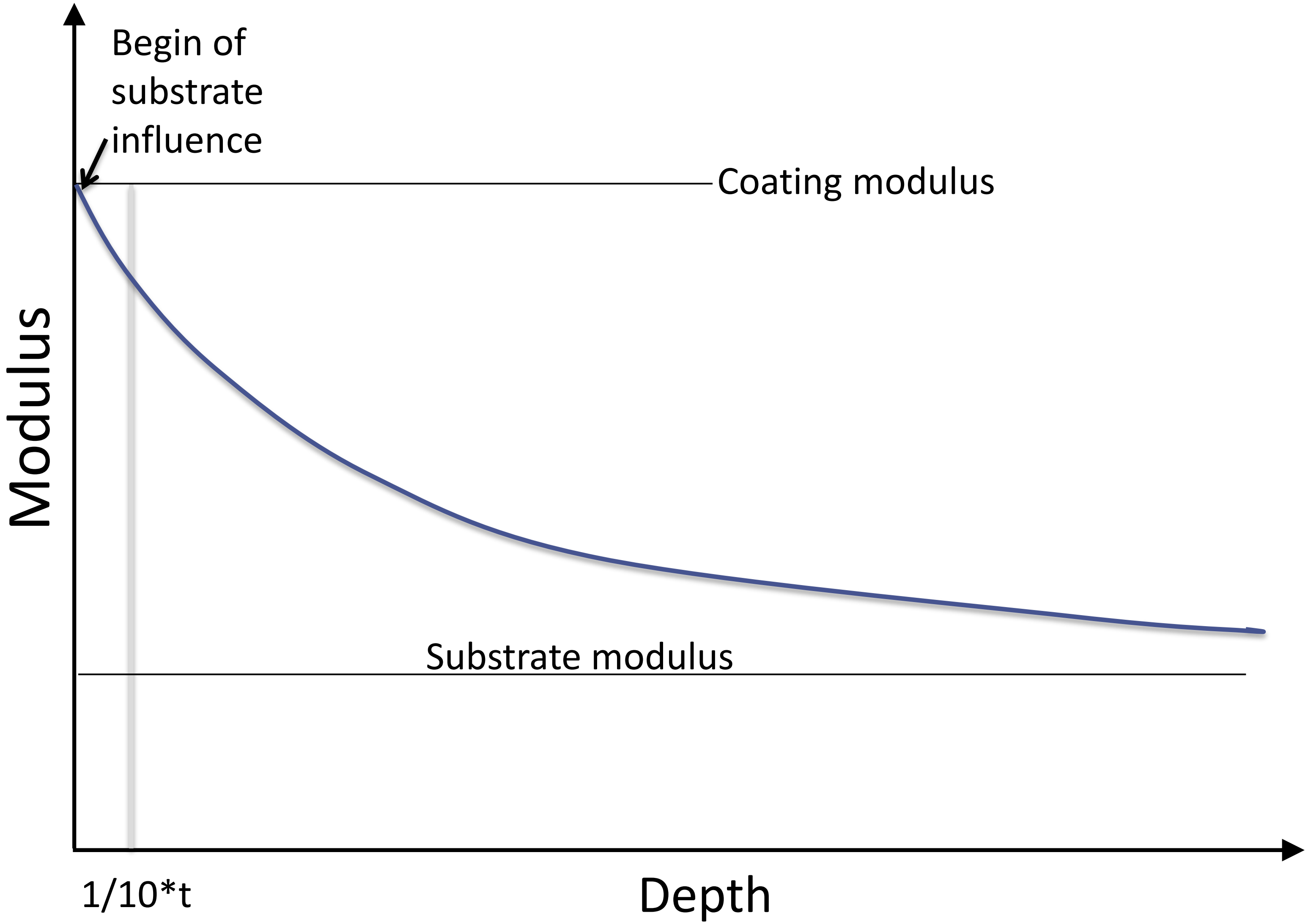
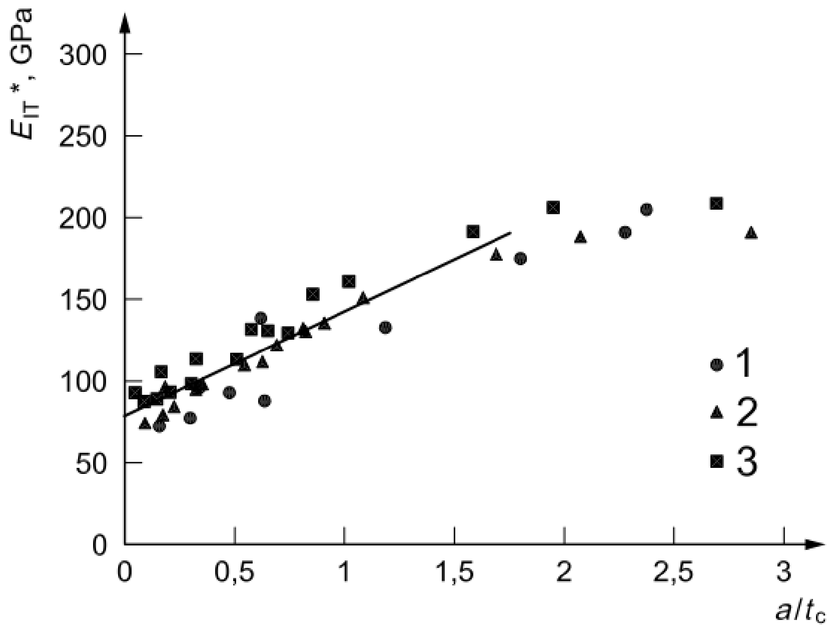
Substrate influence on modulus



ISO 14577 Part 4

In the case of soft/ductile coatings, indentation force or displacement and indenter geometry shall be chosen such that data shall be obtained in the region where $a/t_c < 1,5$. The plane strain indentation modulus of the coating E_c* is obtained by taking a series of measurements at different indentation depths and extrapolating a linear fit to plane strain indentation modulus vs. a/t_c to zero, see Figure 4.



Key

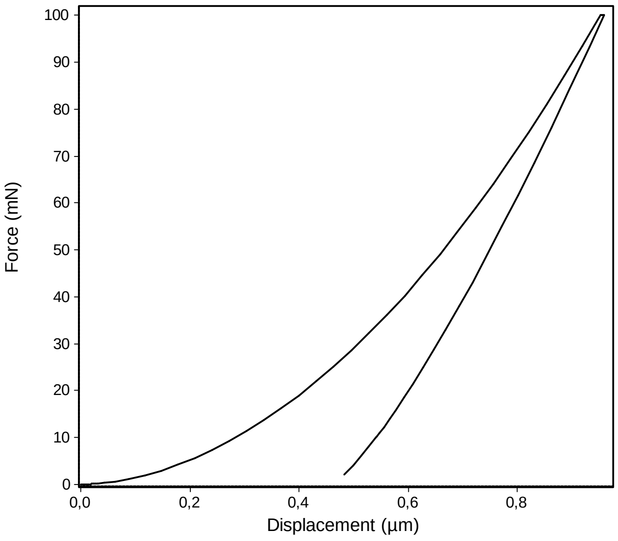
1 spherical indenter

2 Berkovich indenter

3 Vickers indenter

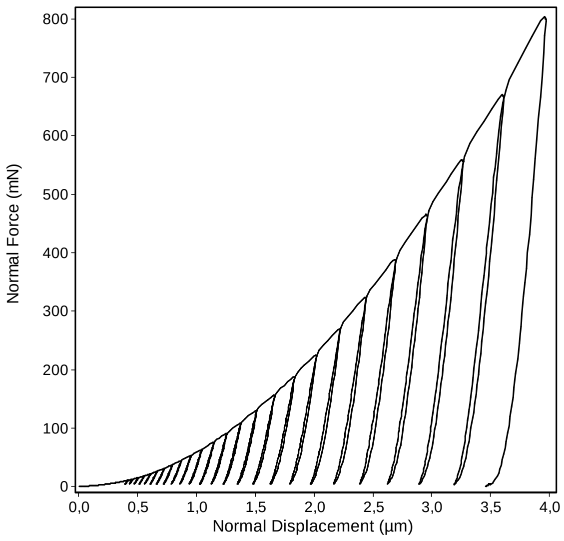
Test methods for hardness and modulus

Single test



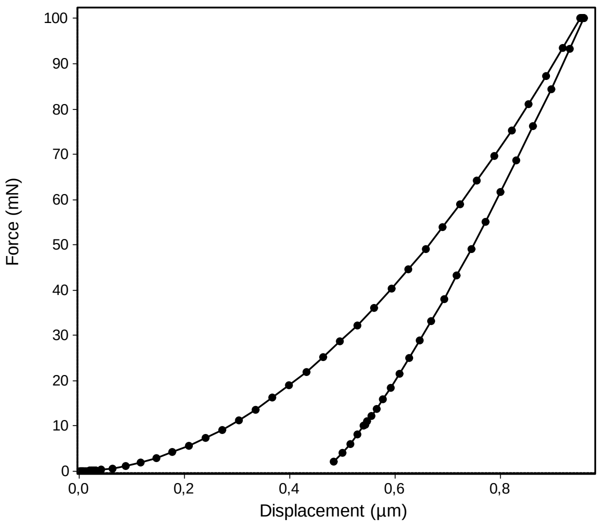
1 Result for maximum depth
Typical test time 20s
Medium accuracy
Results after creep

Cyclic test



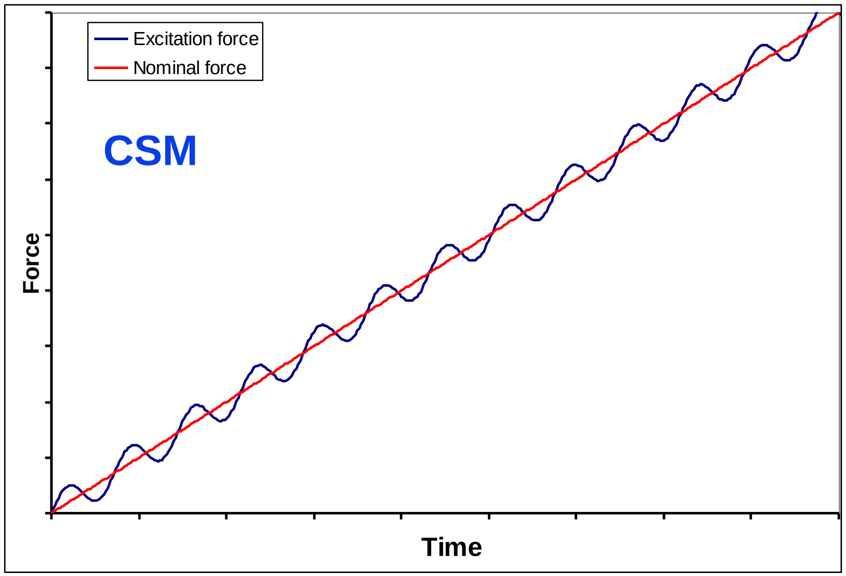
About 20 results over depth
Typical Test time 250s
Lowest accuracy
Results after creep

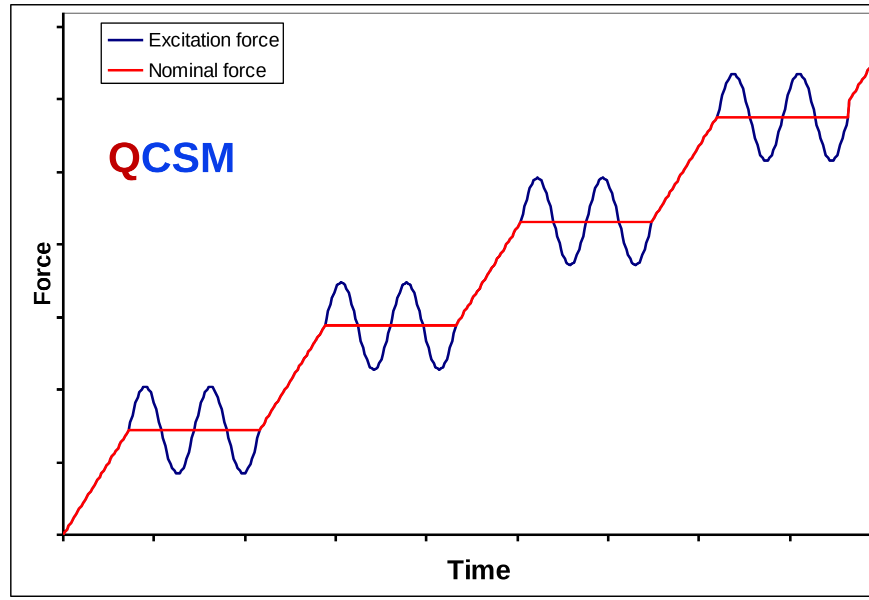
Dynamic test (QCSM)



About 35 results over depth
Typical test time 125s
Highest accuracy
Results during creep

Continuous and quasi continuous stiffness measurement



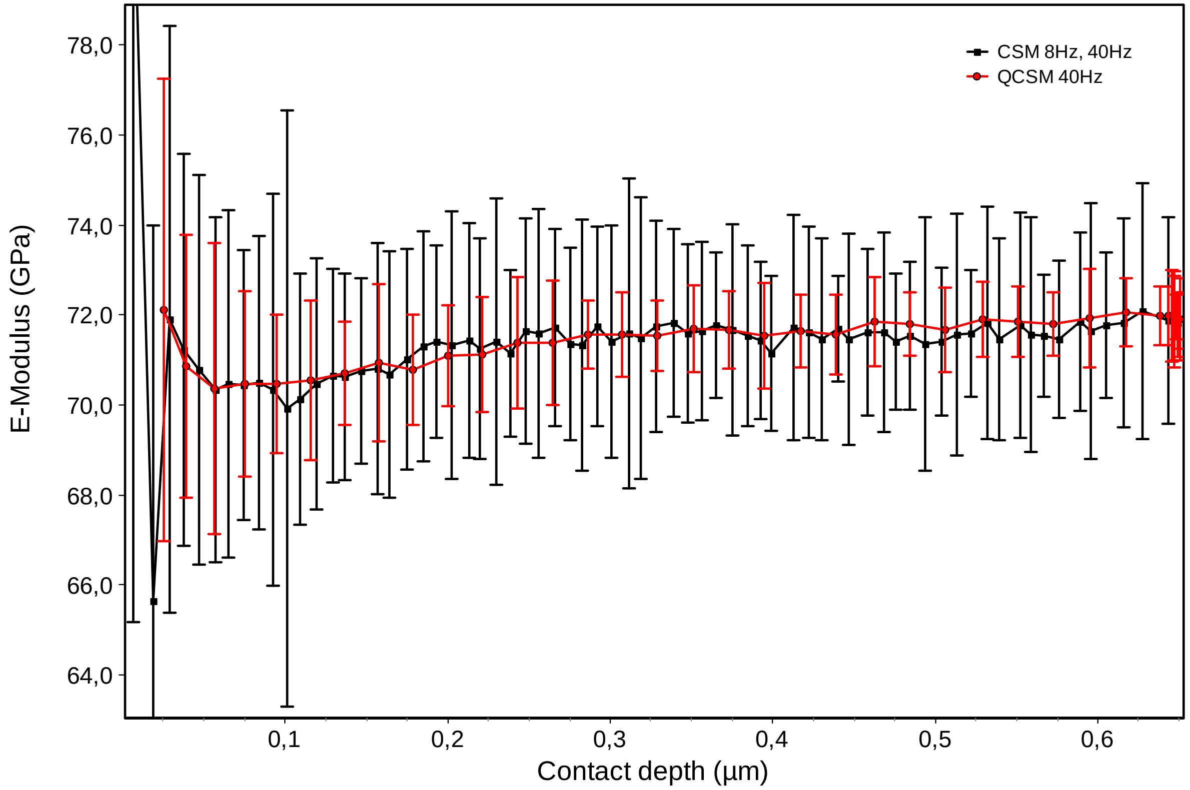


QCSM: sinusoidal oscillation only on (0,5 – 3s) when static force is constant.
First 20% of data per force step are neglected to reduce creep influence.

Advantages:

- Averaging of several oscillations at same conditions possible without problems → more accurate.
- Creep influence is reduced.
- Static forces are well defined for every oscillation result.

Comparison of accuracy between CSM and QCSM



Error bars give statistical error for average of 6 measurements
Error of QCSM tests < 50% of error of CSM tests.

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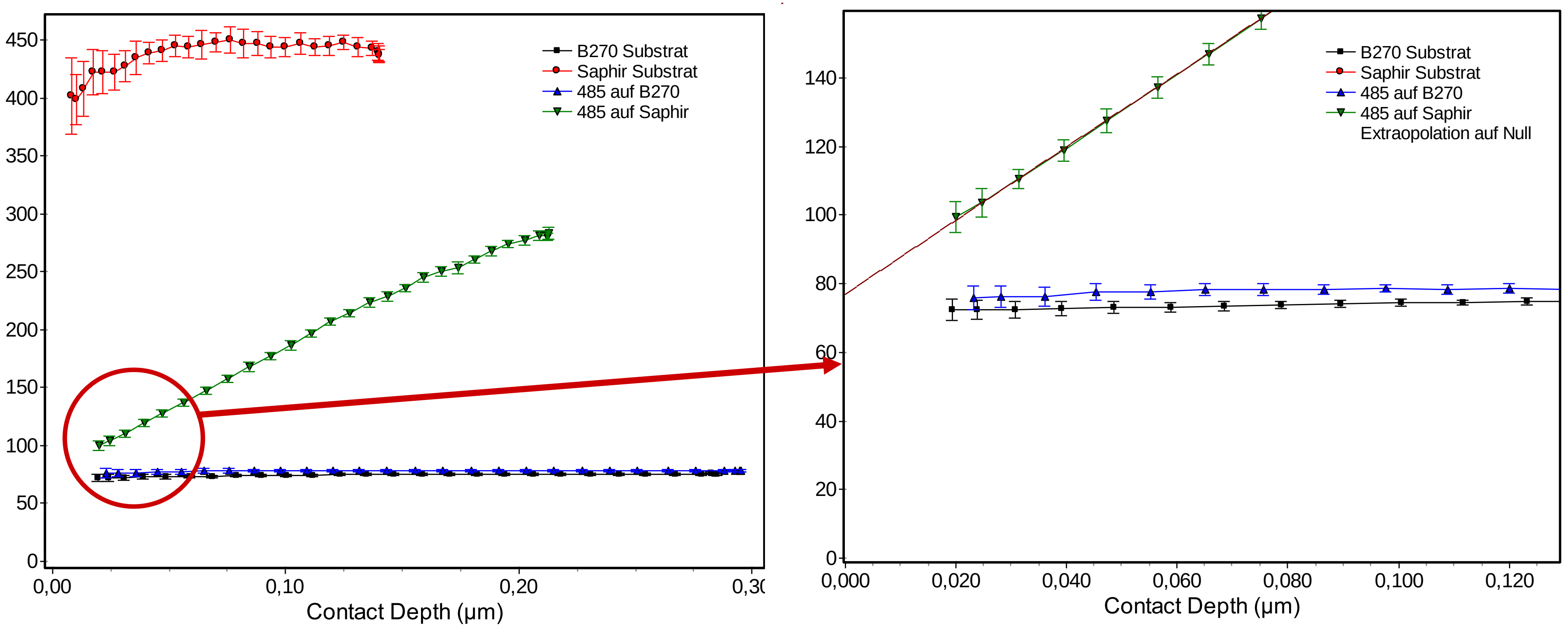
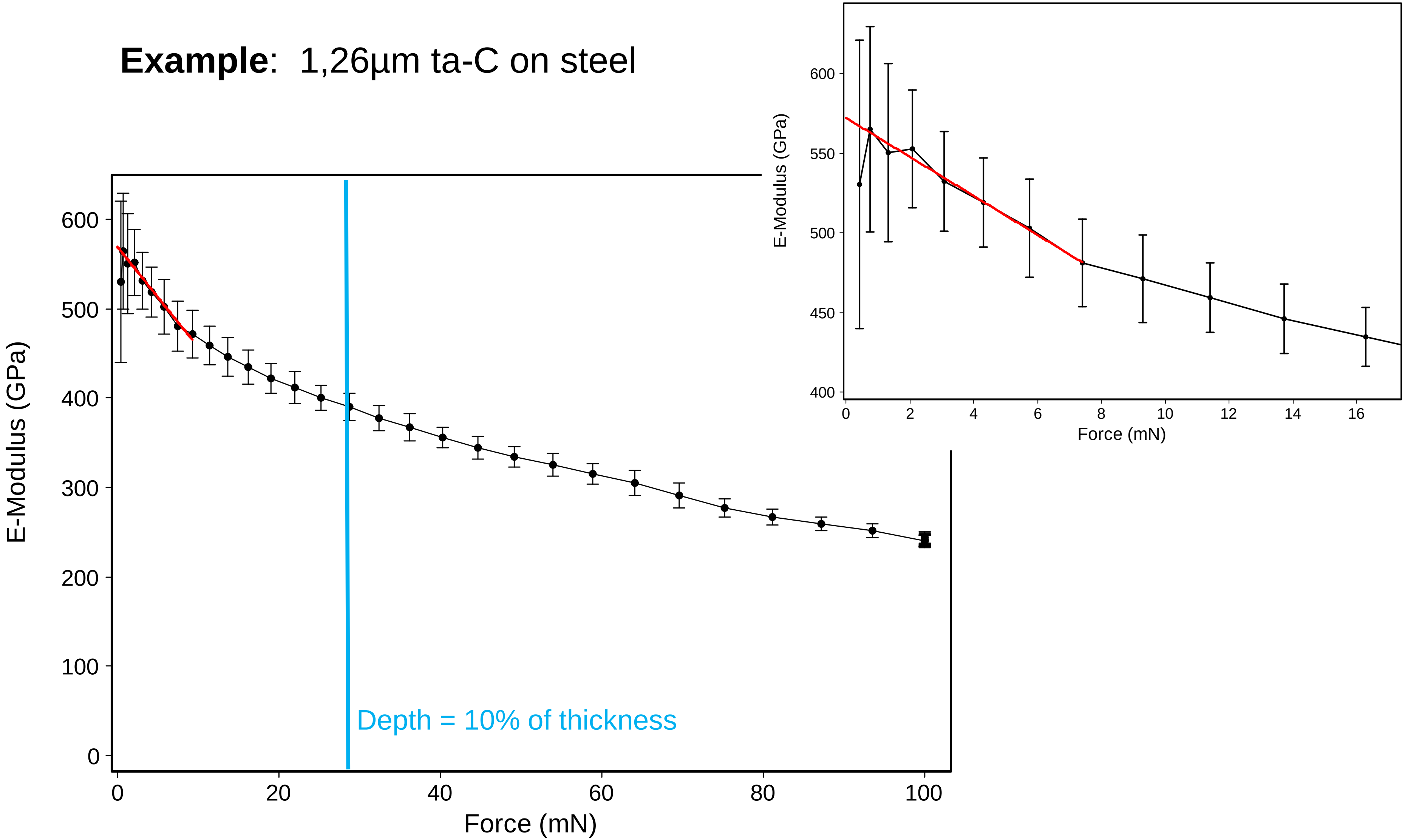
Example: equal 260nm SiO_2 coatings on glass and sapphire

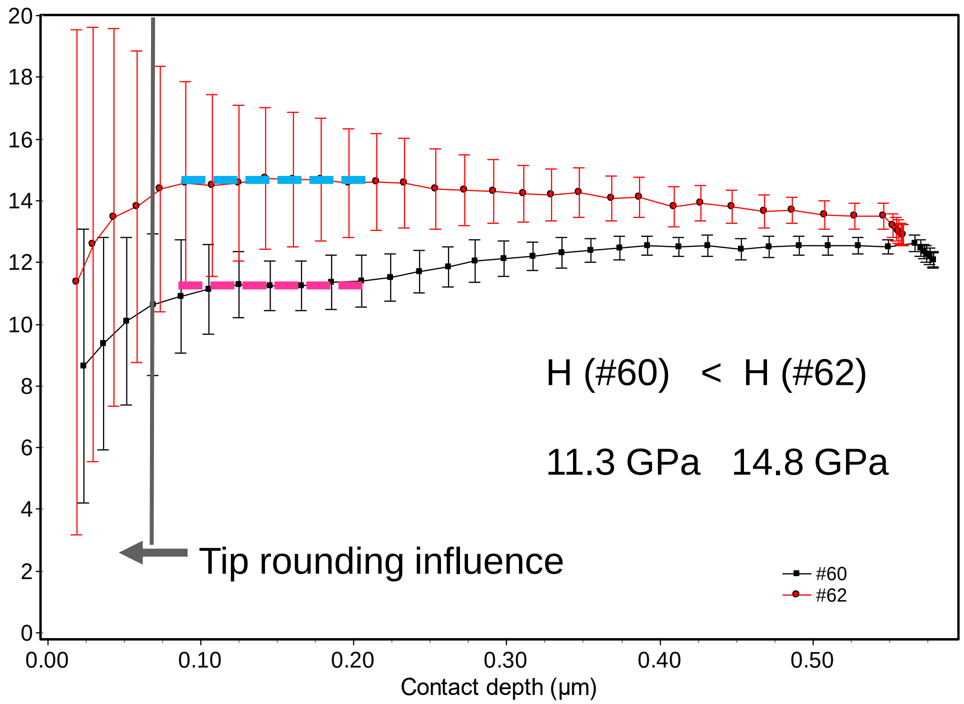


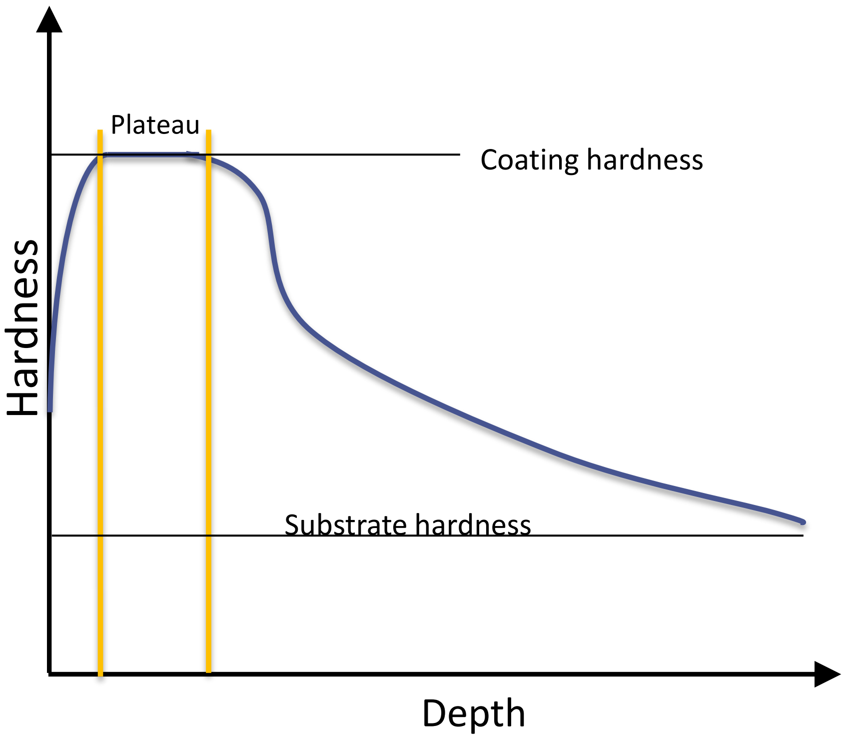
Maximum force 18mN; first point at (20 nm; 0,24 mN).

Example: 1,26µm ta-C on steel

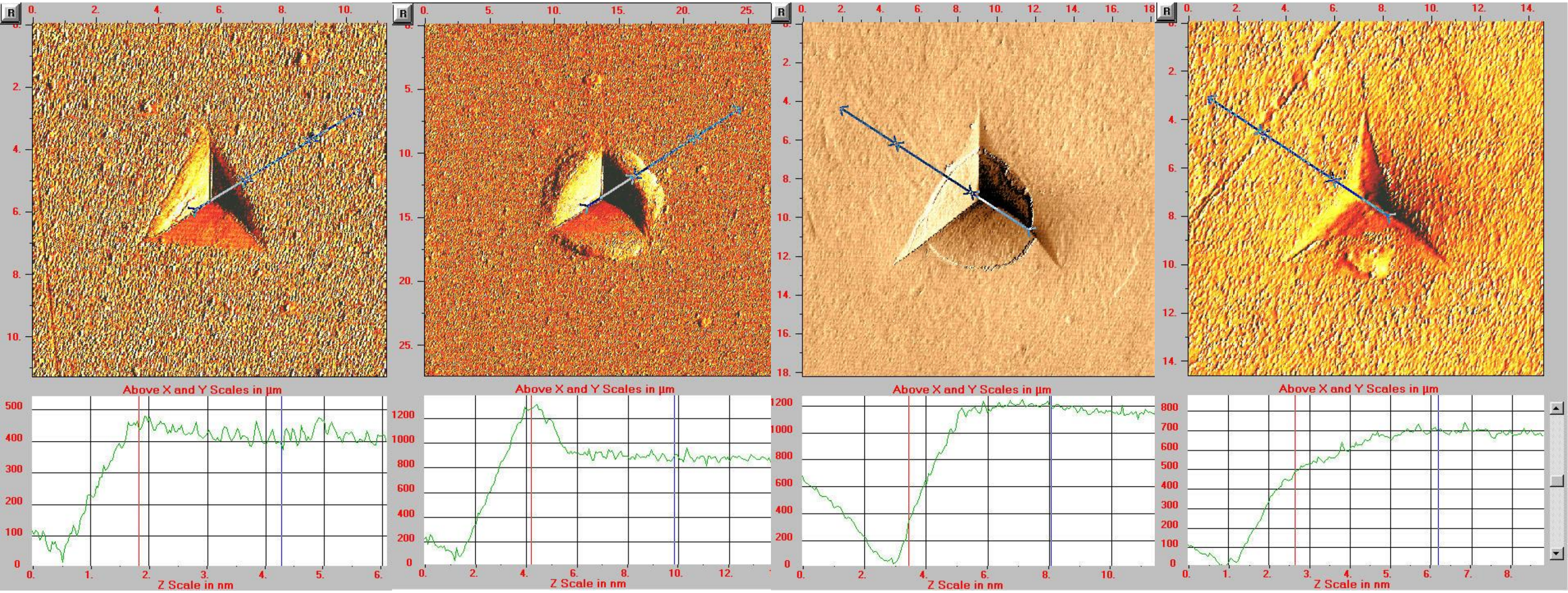


Hardness: finding a plateau without substrate influence





Indentation hardness of two different **a:C:H:W** coatings (DLC) as function of depth, measured with dynamic QCSM method



normal	pile-up	cracks	sink-in
Al on BK7 glass	Al on BK7 glass	Al_2O_3 on Nickel	Al_2O_3 on Nickel
0,4 µm / 1,1 µm	0,9 µm / 1,1 µm	1,2 µm / 0,9 µm	0,7 µm / 2 µm
Load: 10 mN	50 mN	100 mN	100 mN

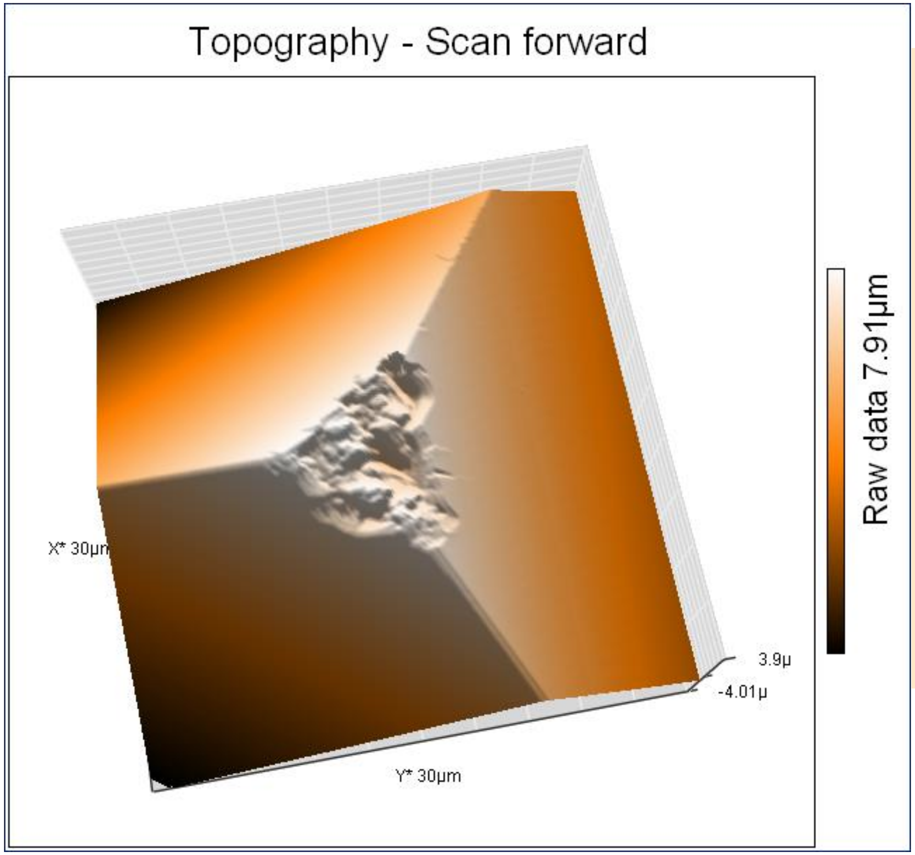
green = depth / film thickness

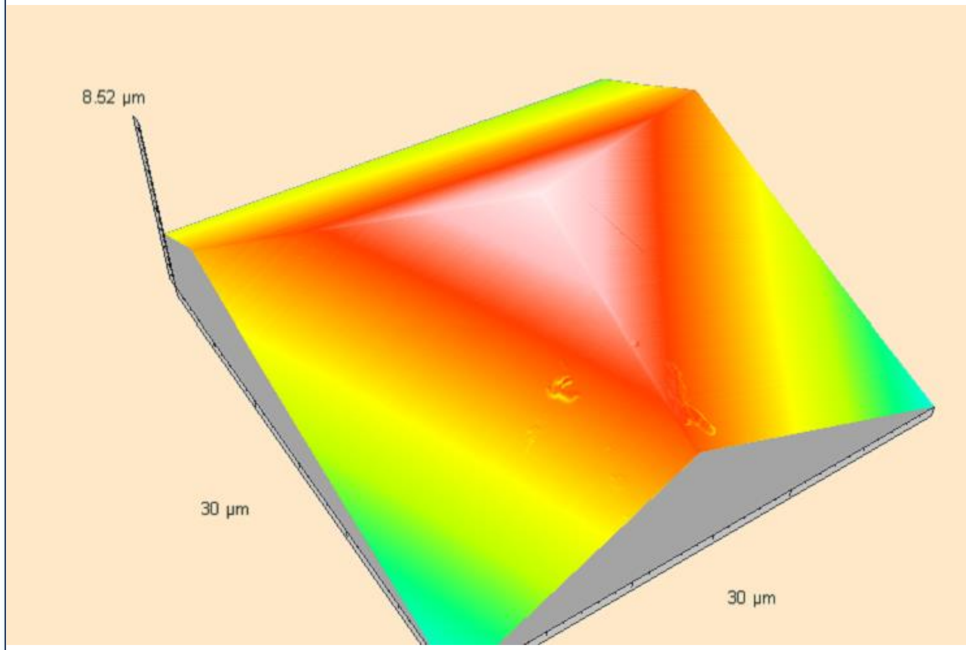
Determination of area function

In the low depth range (<6µm) an accurate determination of the real tip shape (area function) is the key for correct hardness and modulus measurements.

Determination of area function

Direct method: AFM scan of the tips



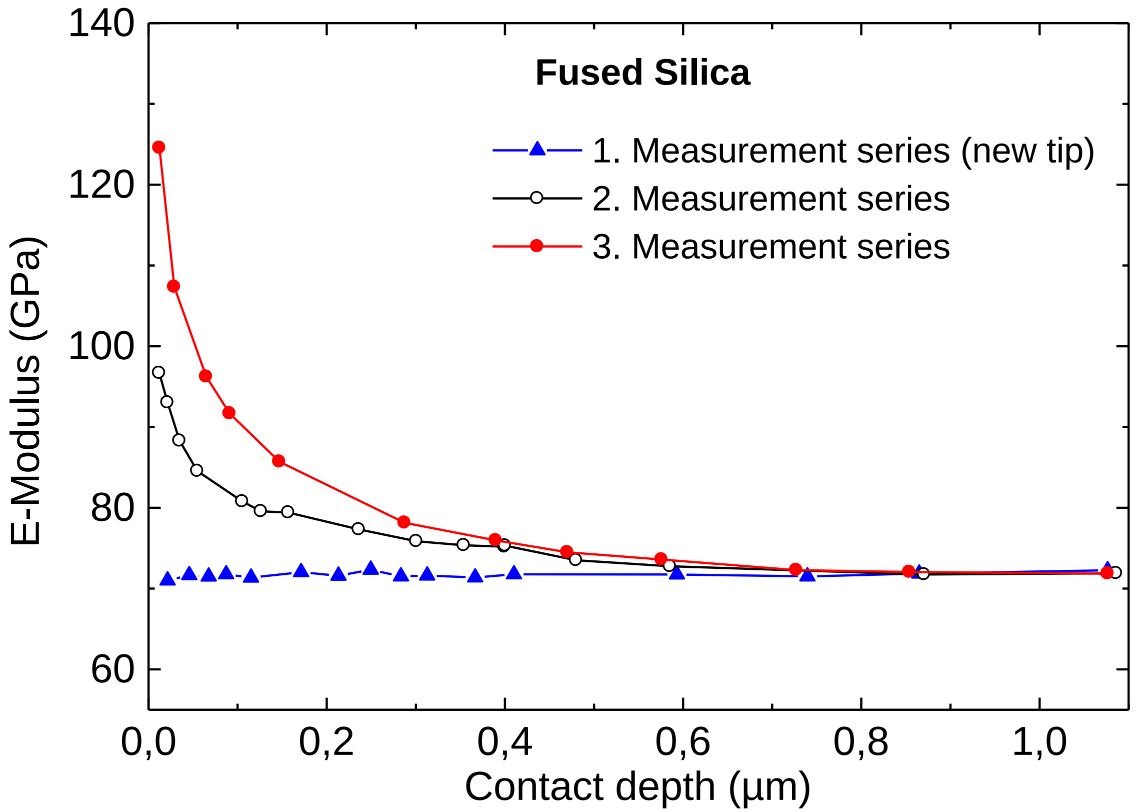


Left: broken diamond tip

Right: new tip without defects

Error due to incorrect area function in dependence on depth

Example: fused silica

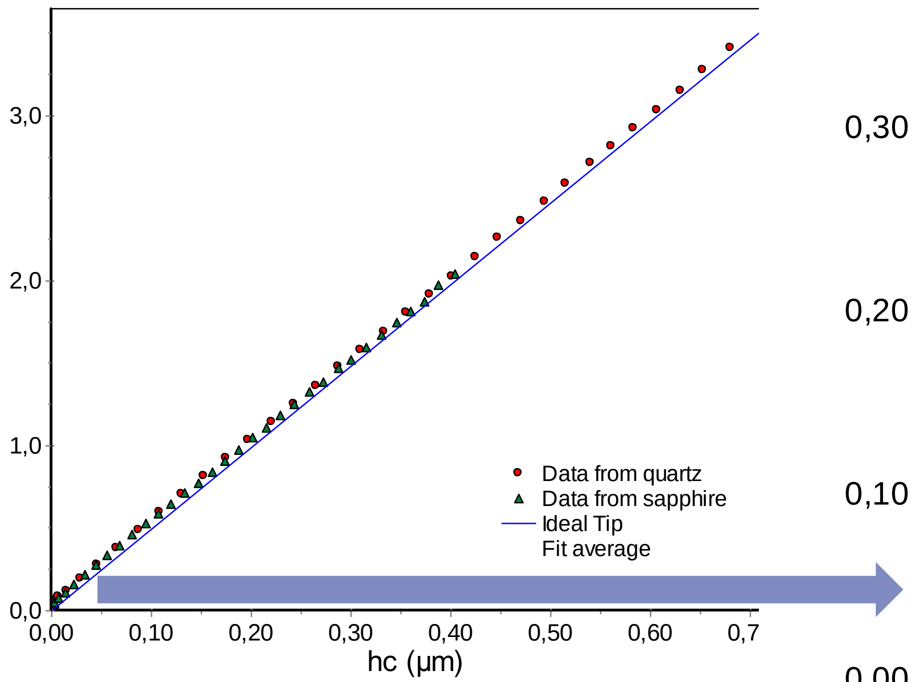


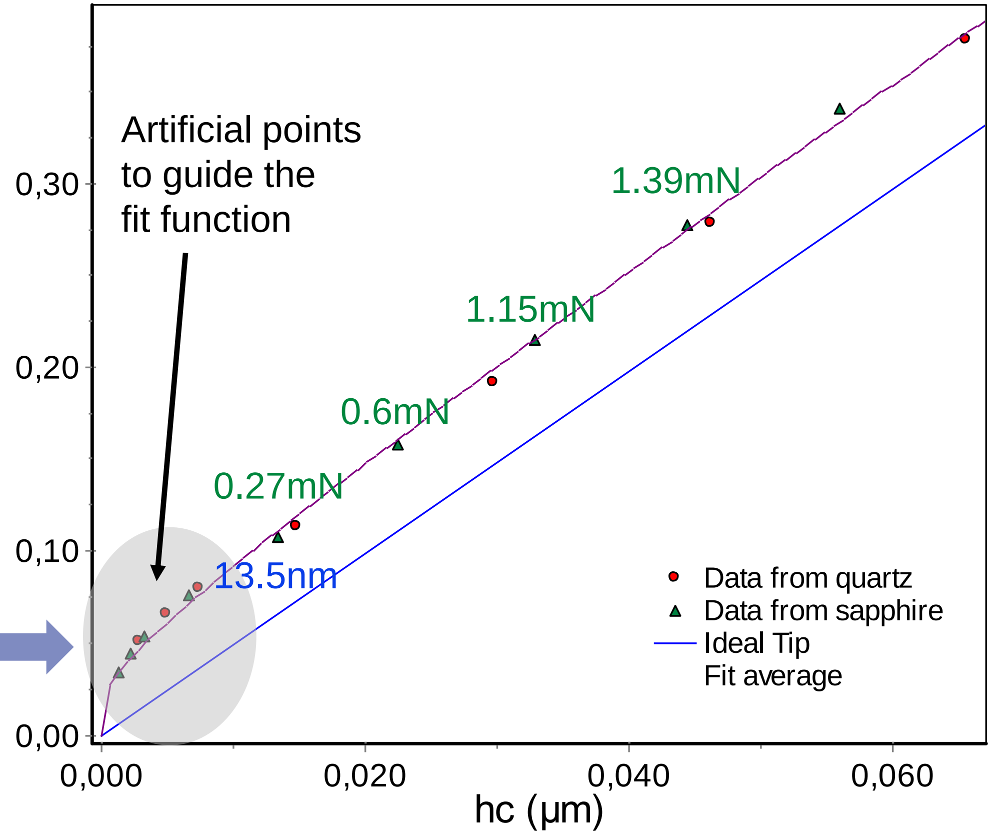
Tip radius:
1. ≈ 150 nm (calibrated fresh tip)
2. ≈ 350nm (no new calibration)
3. ≈ 700 nm (no new calibration)

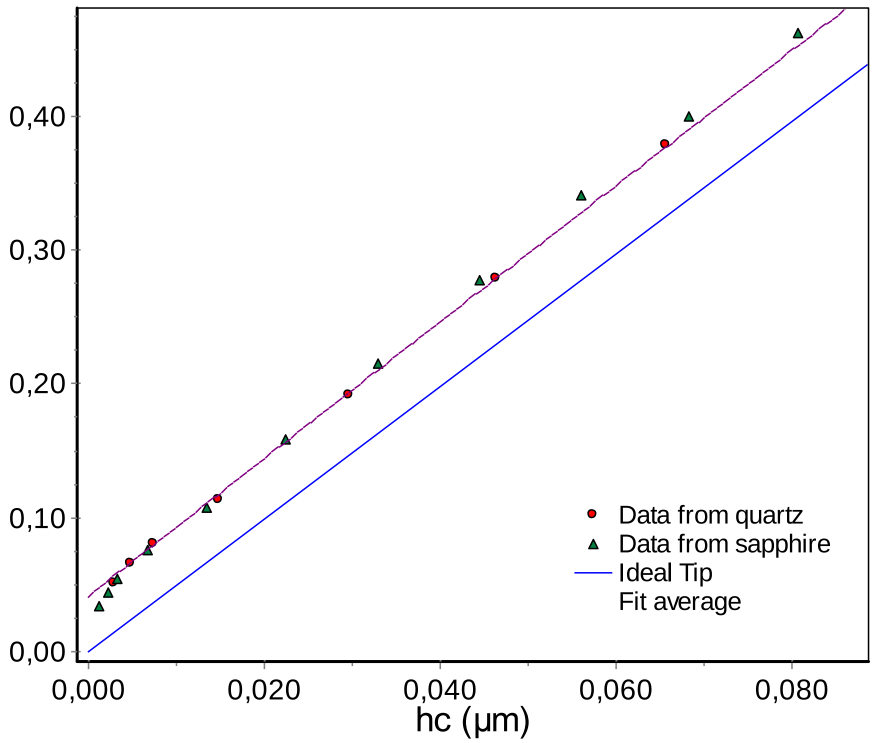
Determination of area function

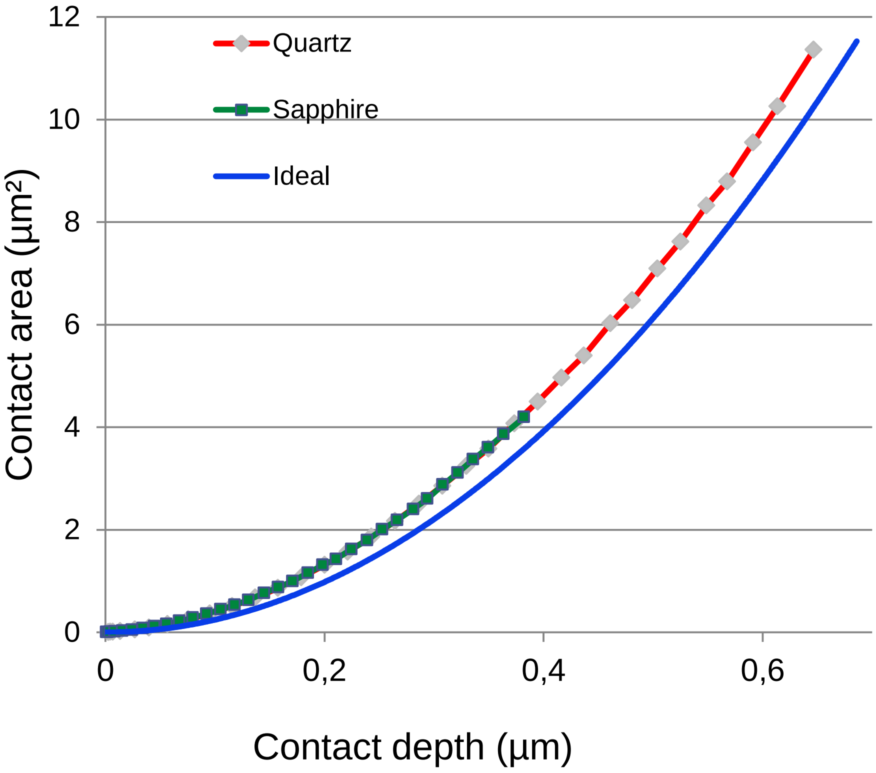
Direct method: Measurement with metrological AFM

Indirect method: Indentation measurements on homogeneous reference materials









Inadequate fit function with insufficient term number

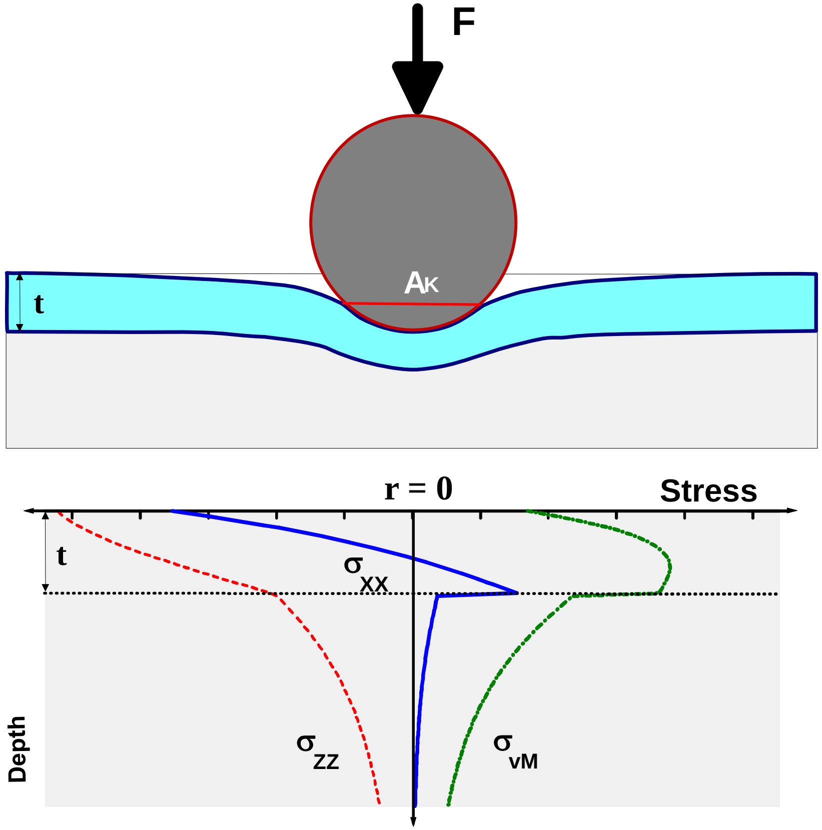
A quadratic presentation (area over depth) allows only a bad estimation of the tip quality

Alternative modulus test methods

especially for ultra thin coatings

Elastic indentation with spherical indenter

and compensation of substrate influence



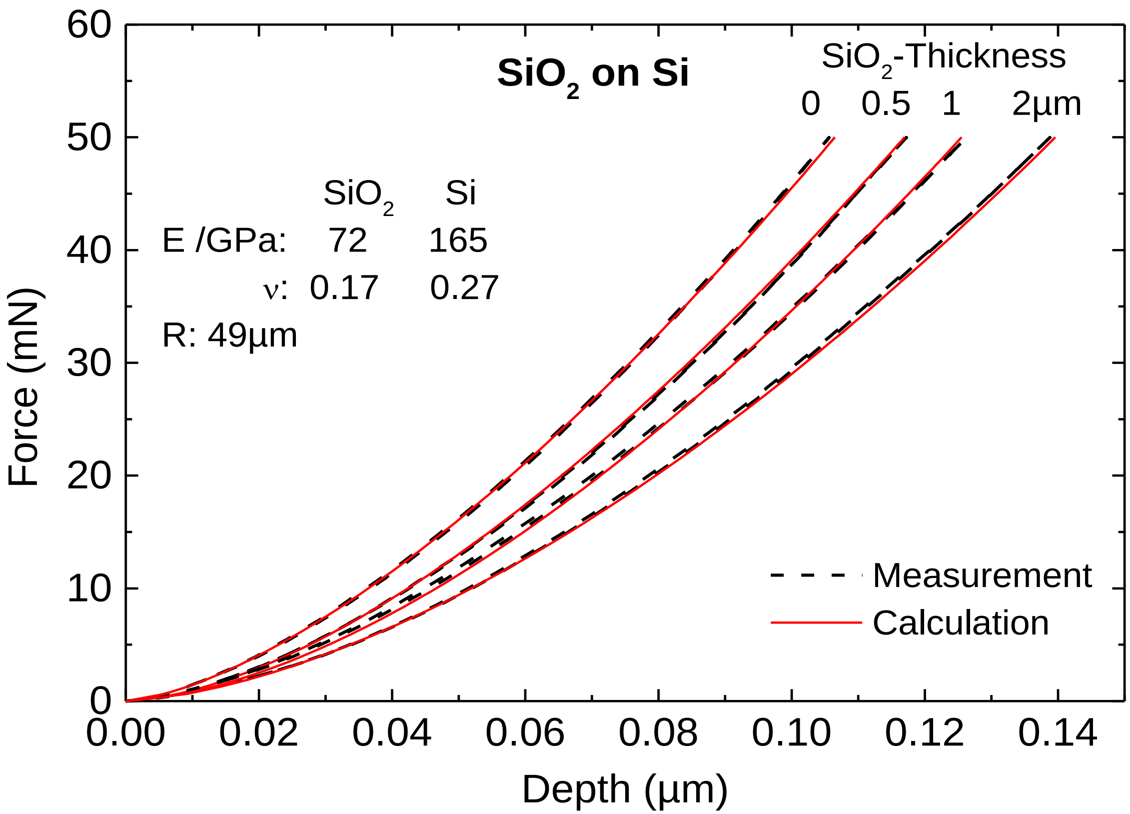
Requirements

Wholly elastic indents

High accurate measurements with resolution < 1 nm

Accurate knowledge of tip radius and frame compliance

Combination with elastic modelling



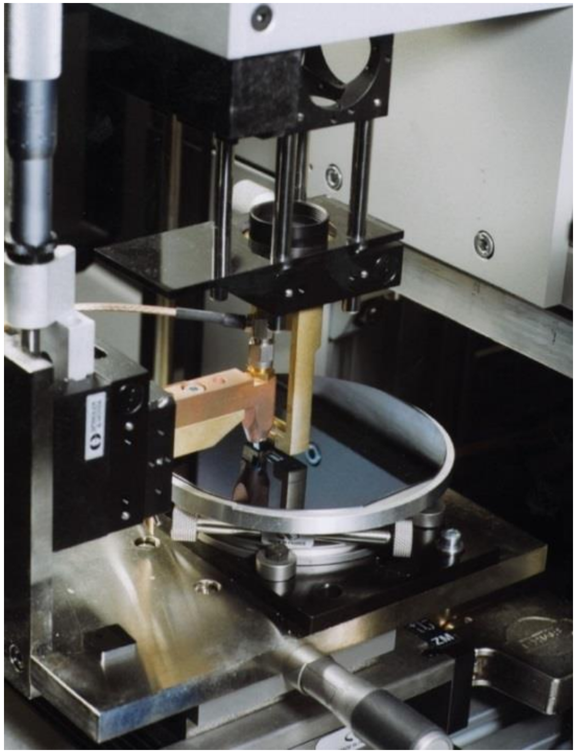
T. Chudoba, N. Schwarzer, F. Richter
Surf. Coat. Tech. 127 (2002) 9-17

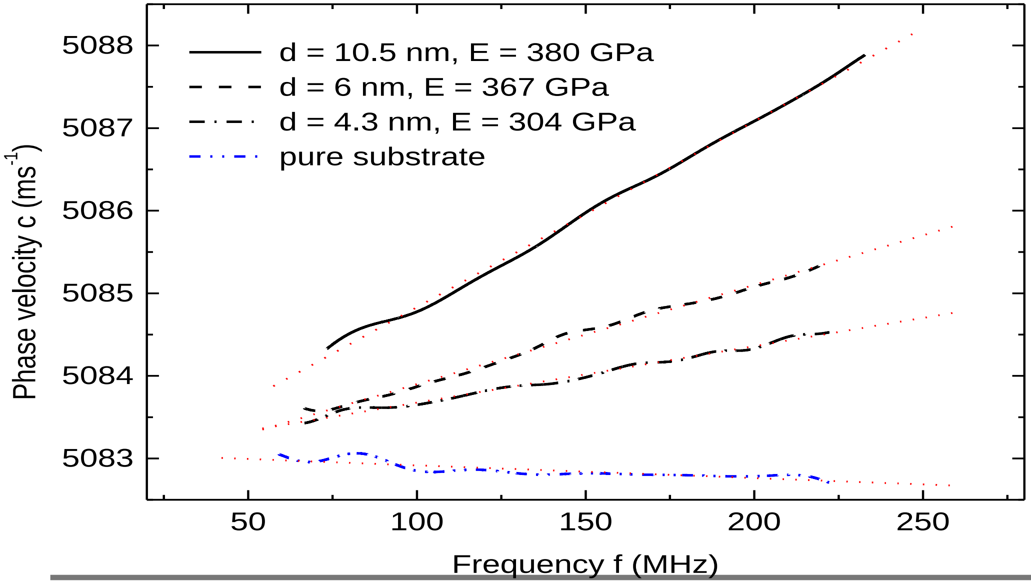
Fit of the measurement data with a theoretical load-displacement curve.

Known substrate properties; fit parameter: film modulus

Useable software ELASTICA, FilmDoctor

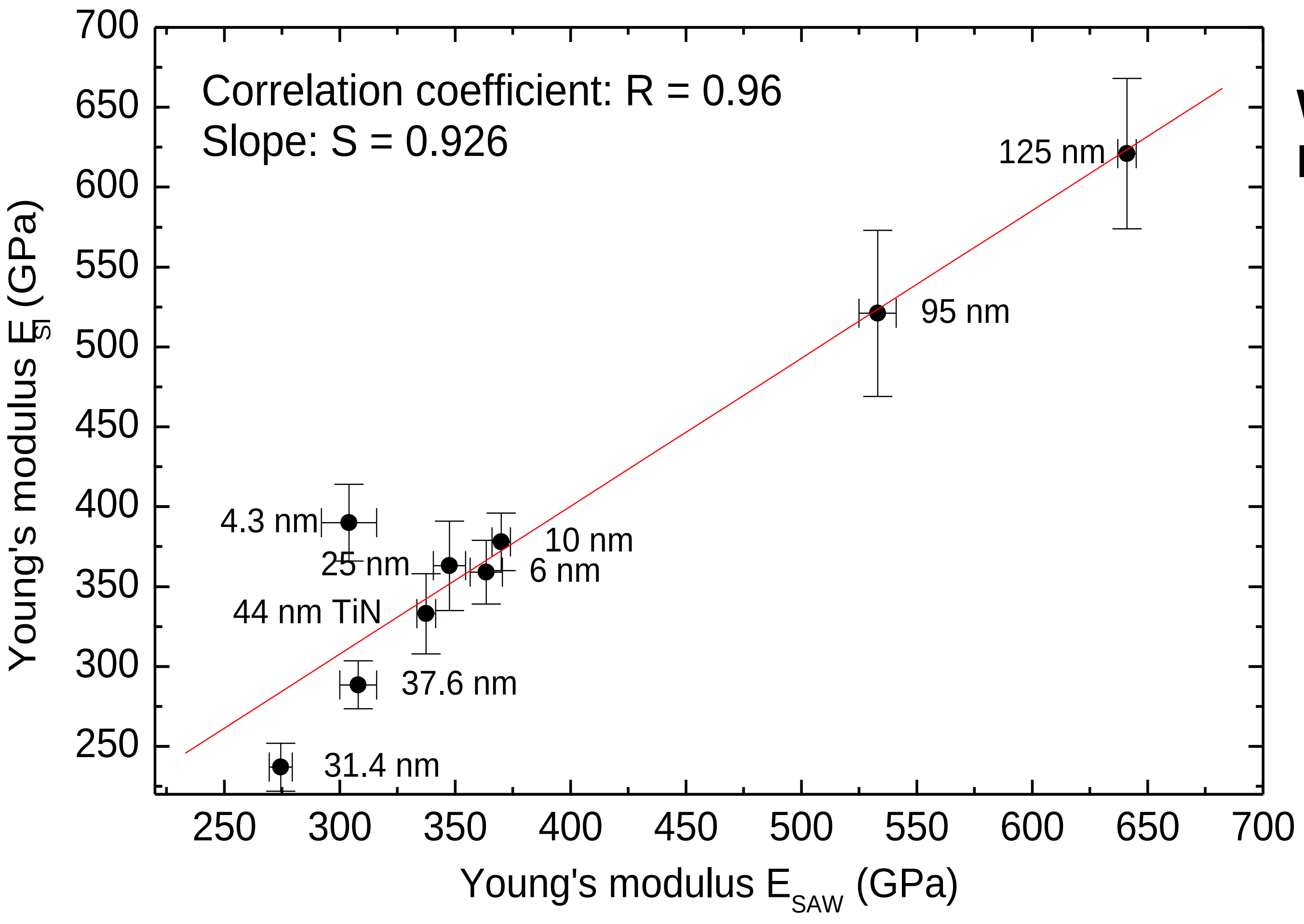
Acoustic surface waves
DIN 50992-1





LAwave

Comparison SAW - Nanoindentation



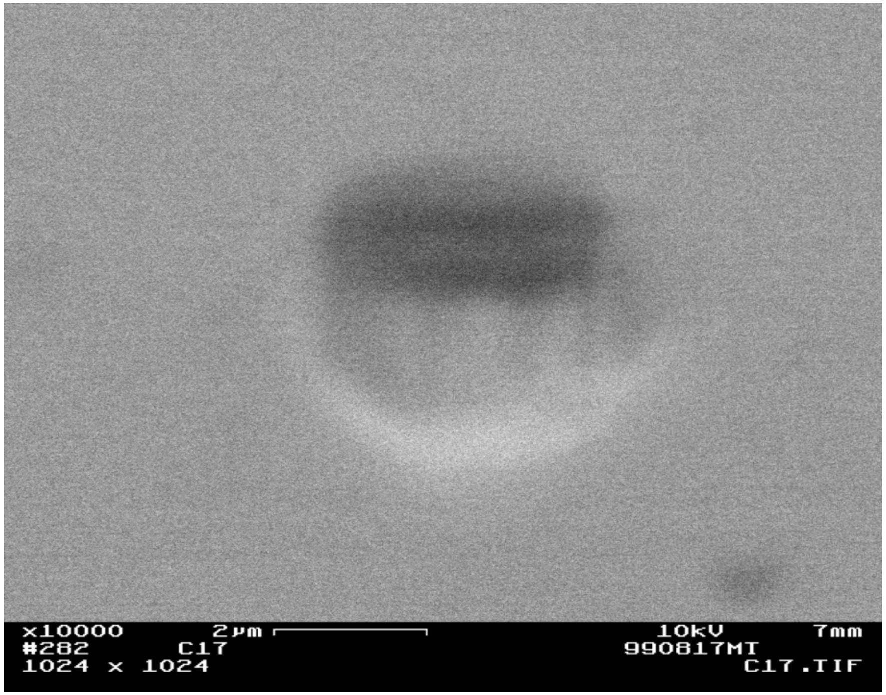
Without 4.3 nm:
R = 99.1; S = 0.993

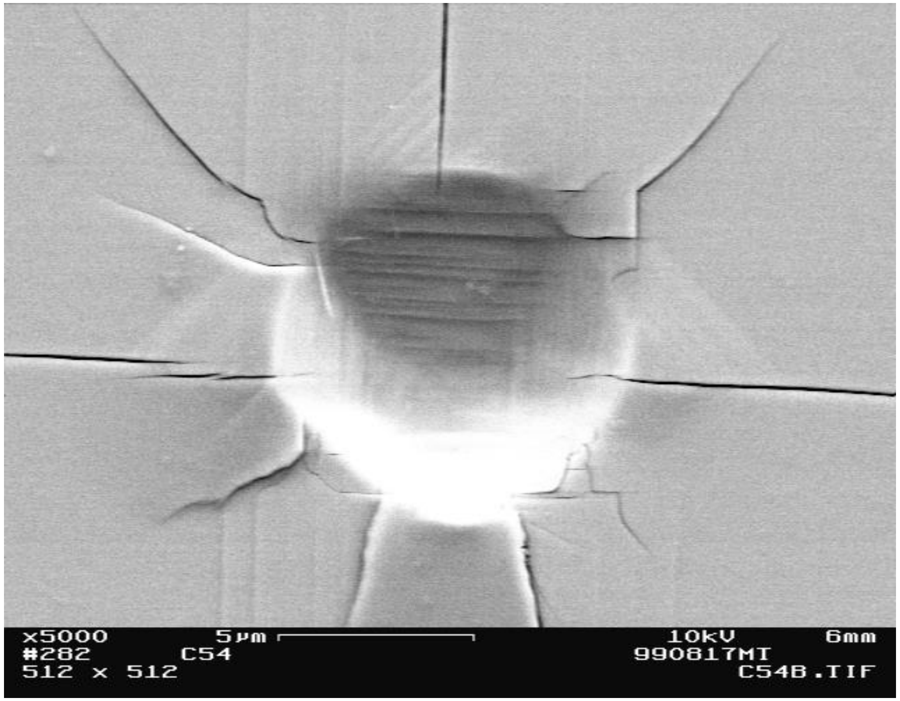
Yield strength measurements

Yield strength

Can we measure the yield strength of hard and brittle materials?

Yes, it is a question of dimension!



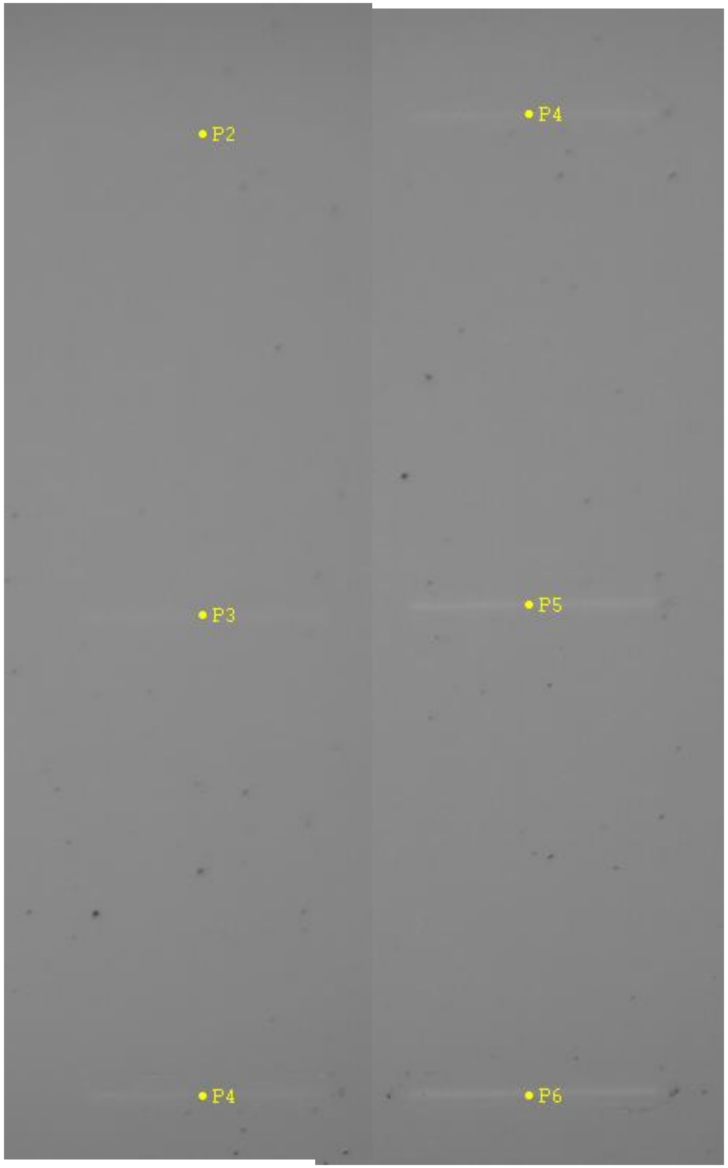


small load

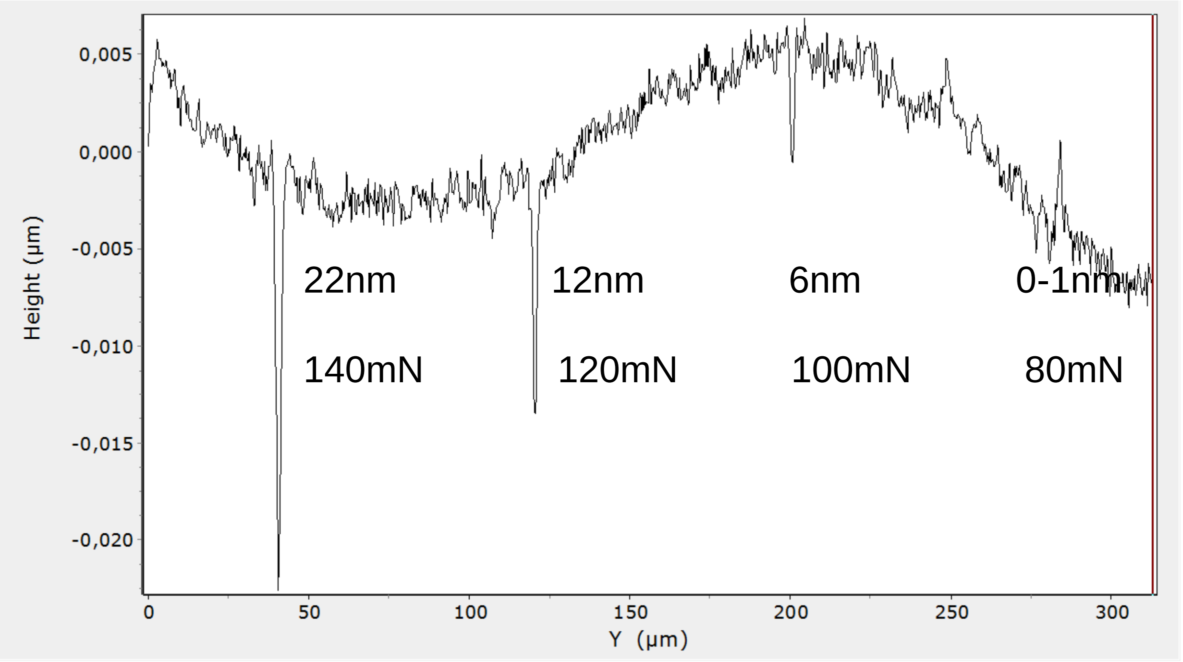
higher load

4µm radius indenter in GaAs

Wear test on fused silica (100 cycles)



Wear depth (obtained by surface scan)

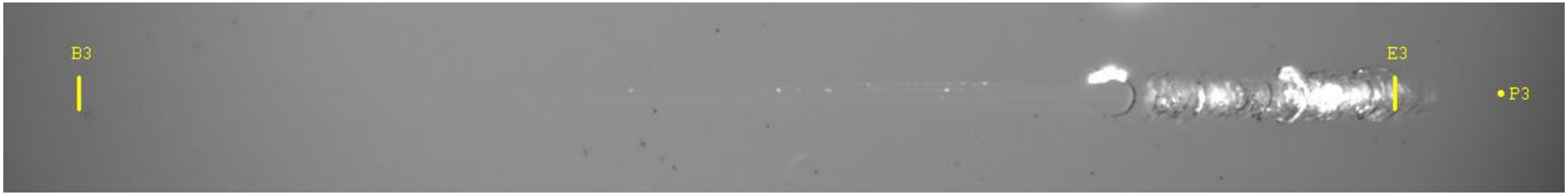


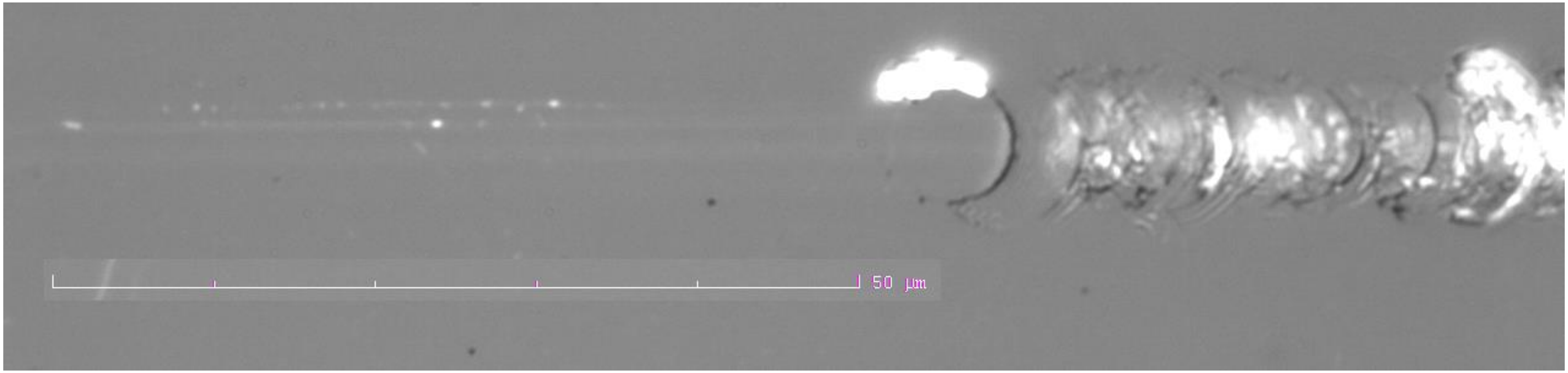
5 tests in a distance of 80 µm;
Time per cycle: 5s
Forces: 50, 80, 100, 120, 140 mN

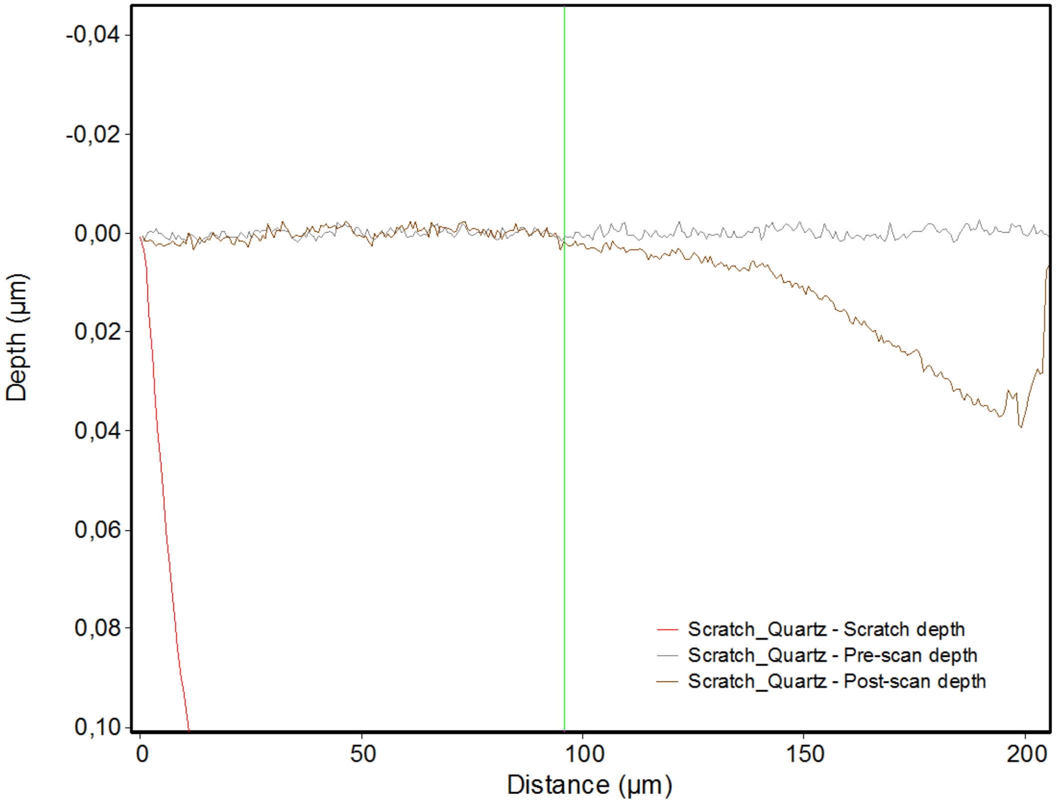
Distance: ± 20 µm = 40 µm
Speed: 16 µm/s

Scratch test on fused silica

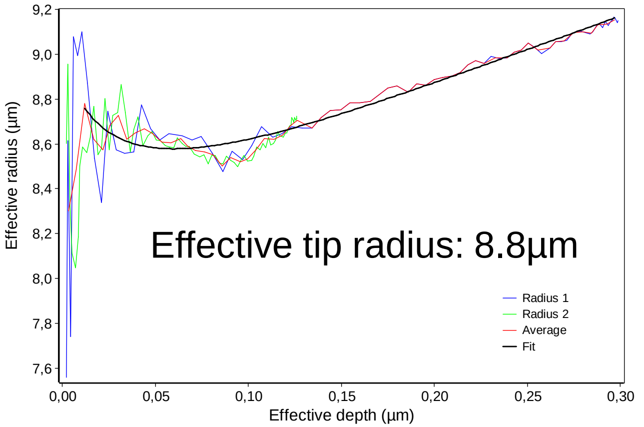
Maximum force:	300mN (increasing)
Distance:	250µm
Speed:	10µm/s

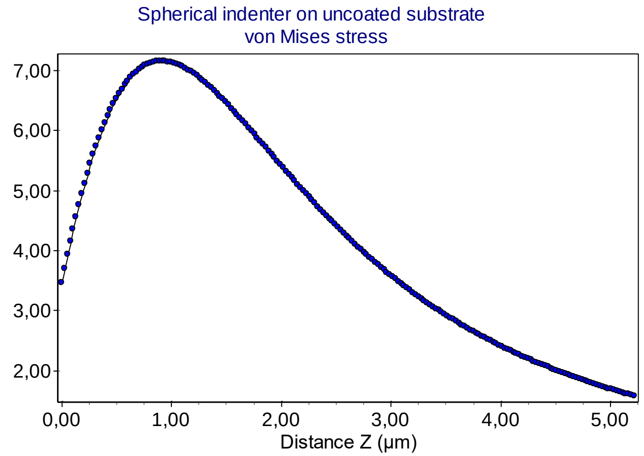






Critical force for plastic deformation: 95.7mN



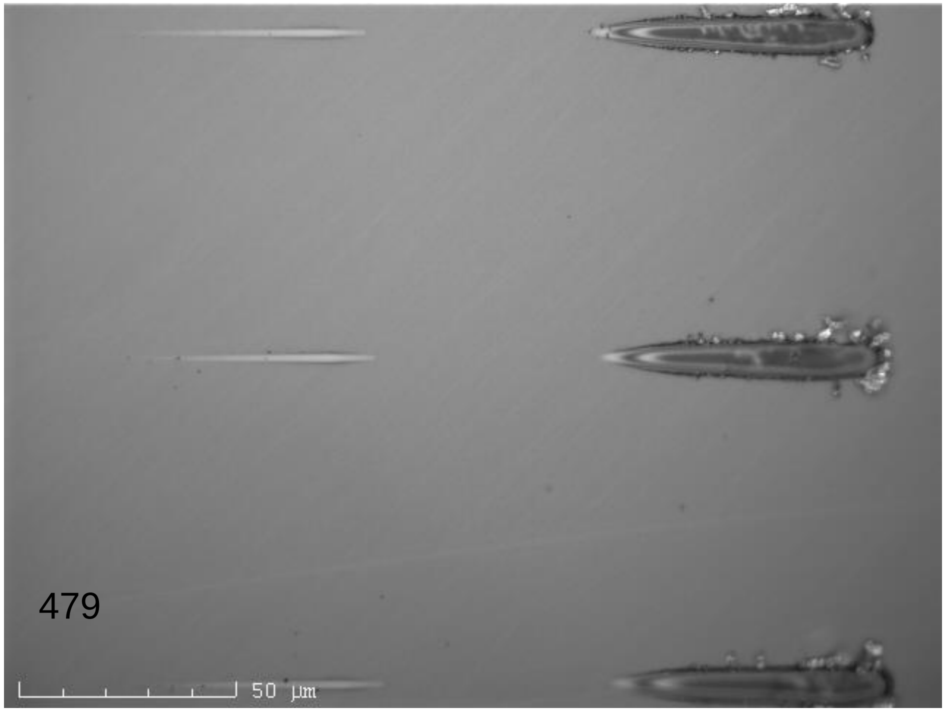


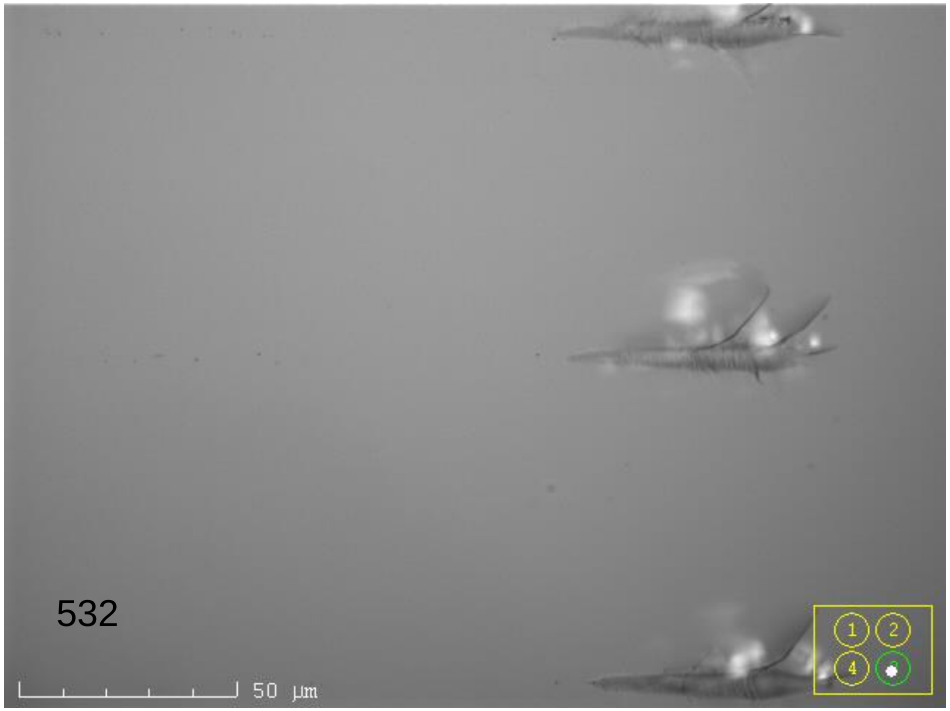
Max. von Mises stress: 7.15 GPa

= yield strength

240 nm thick optical coatings on sapphire

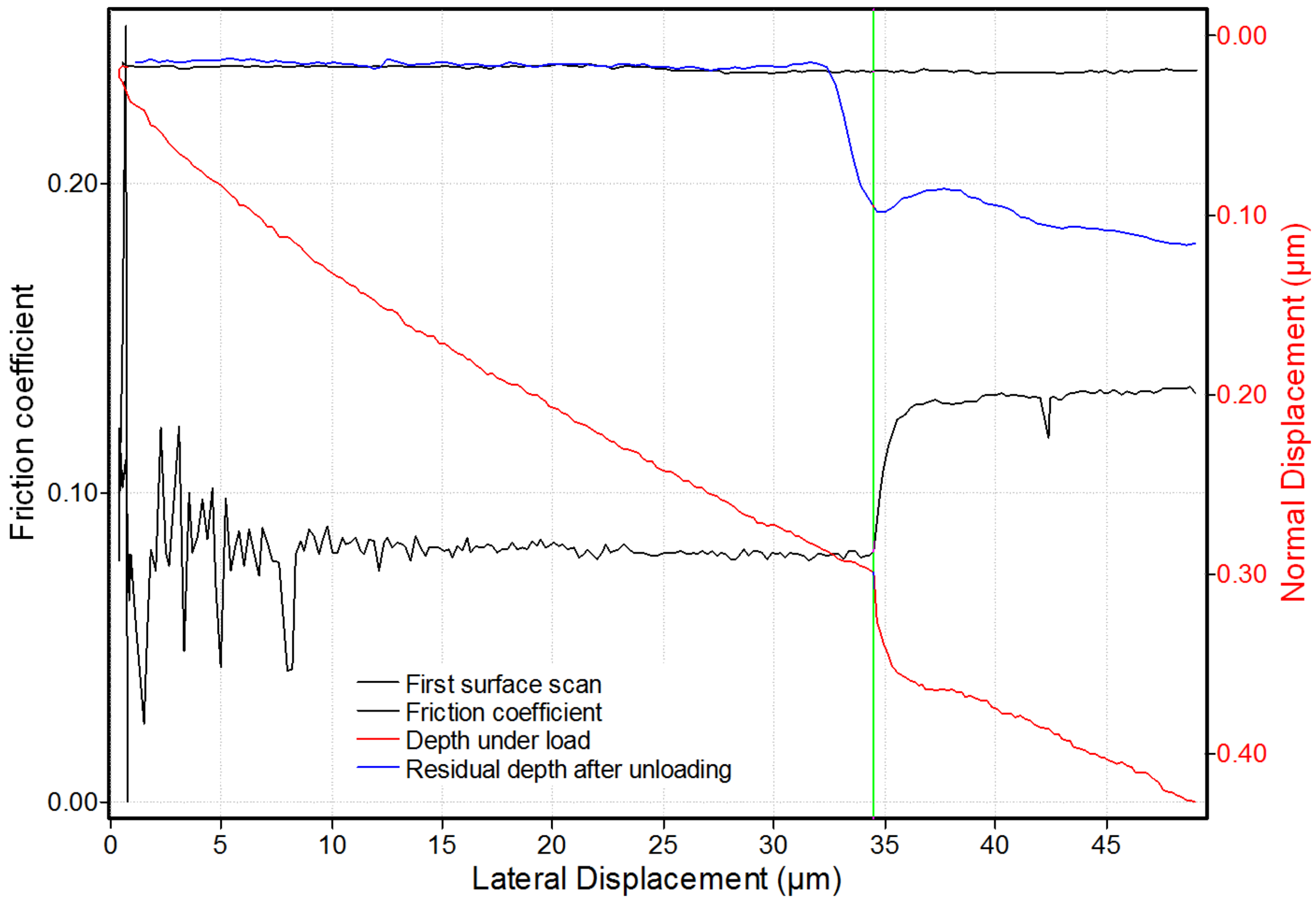
Three 50mN und 700mN micro scratch tests over each other





The two coatings show different failure modes

The difference between pre-scan and post-scan of the surface allows detection the elastic-plastic transition



Failure of the coating at: X: 34.48 µm Fn: 174.94 mN µ: 0.081

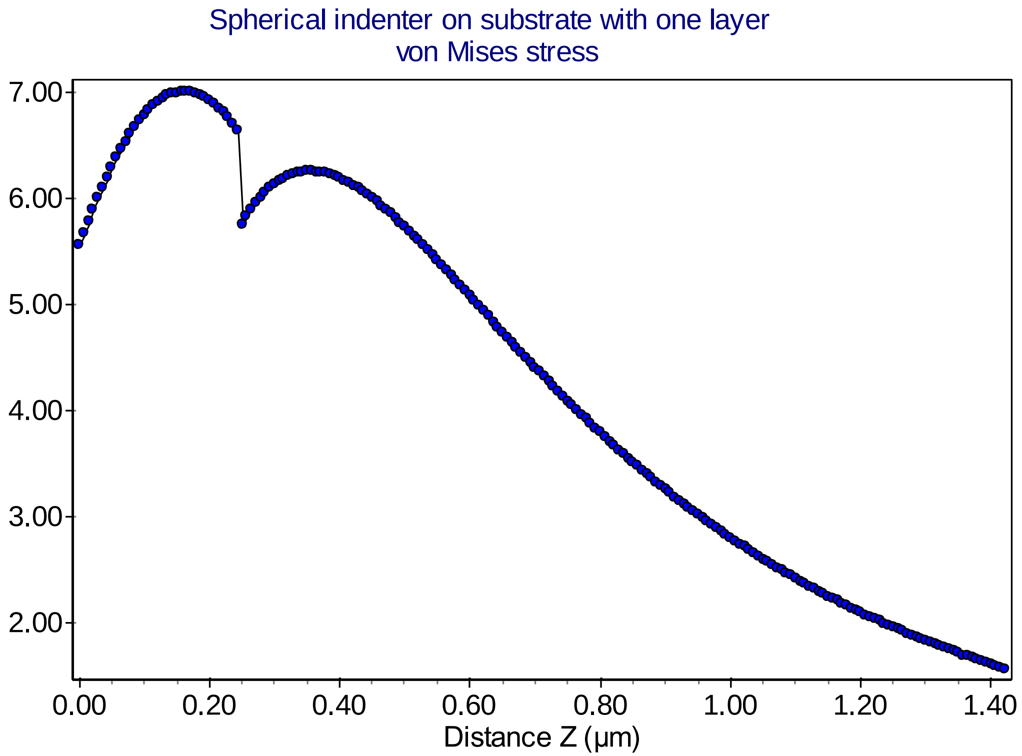
Yielding starts in the coating since the substrate was hard enough for these samples

Yield strength results

Sample number	Fcrit mN	µ	Yield strength GPa
479	**6.89**	**0.061**	**6.97**
486	6.60	0.067	6.5
488	8.30	0.078	7.4
489	8.49	0.088	7.64
531	12.75	0.110	8.79
532	**131.79**	**0.069**	**20.8**
485/Sapphire	8.93	0.065	6.31
485 /Glass	31.92	0.057	6.25
Saphir	171.500	0.084	**27.7**

on sapphire

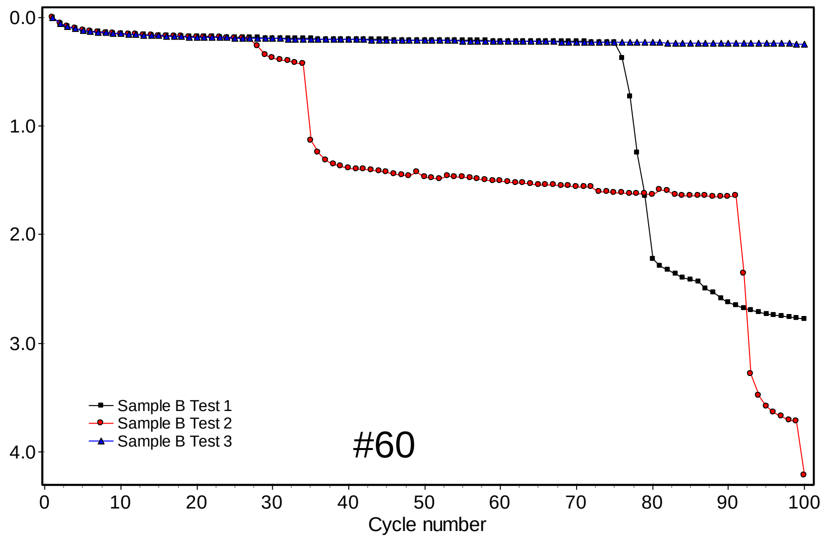
on glass

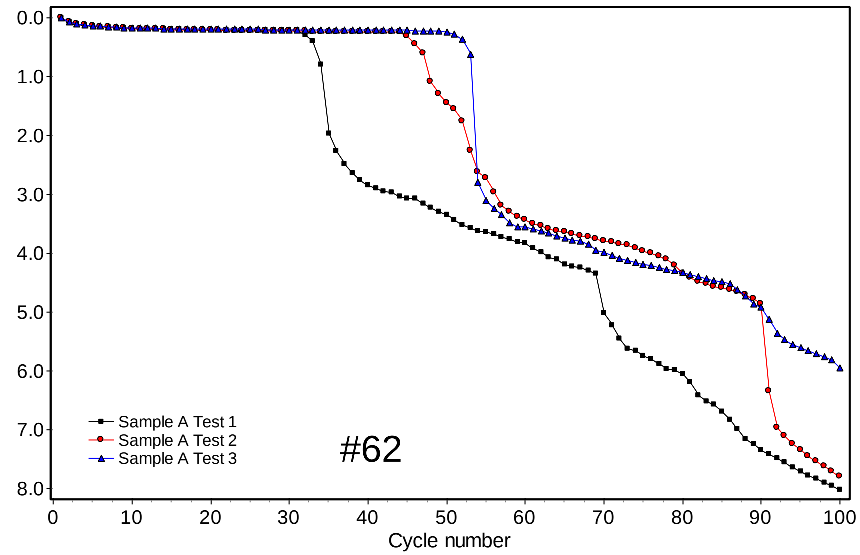


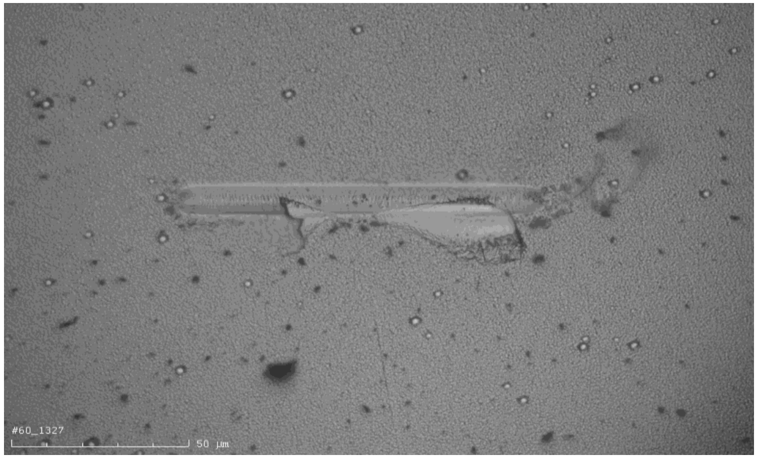
Von Mises stress profile for sample 479

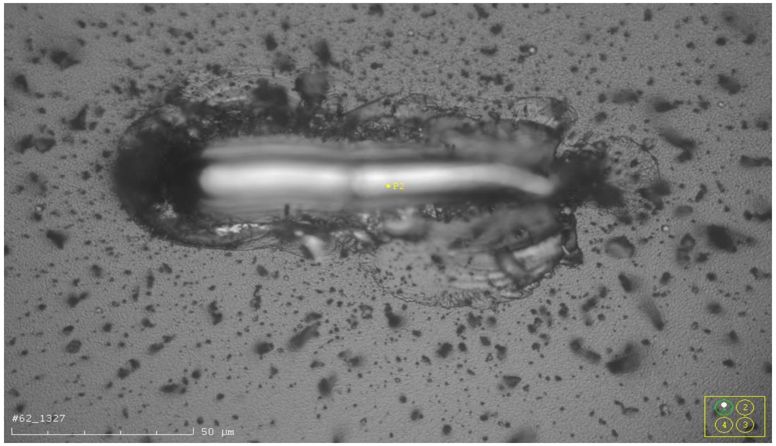
Low cycle fatigue

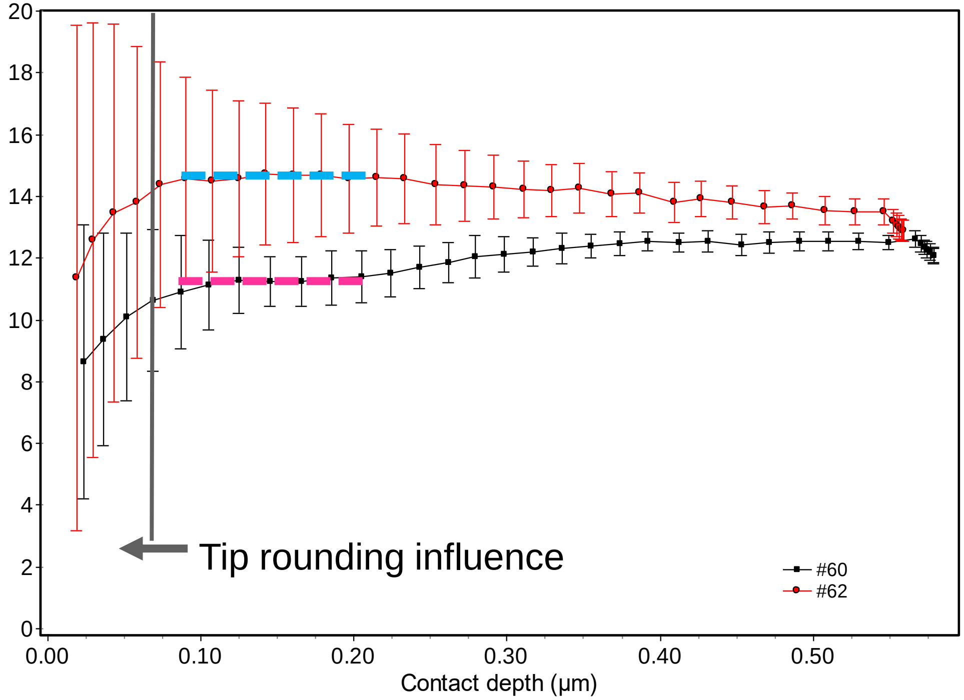
Wear behavior of sample #60 is better than for sample #62 although the hardness is lower
Test conditions: 500mN, 9.3µm radius indenter











(#60) versus (#62)

Hardness
11.3 GPa < 14.8 GPa

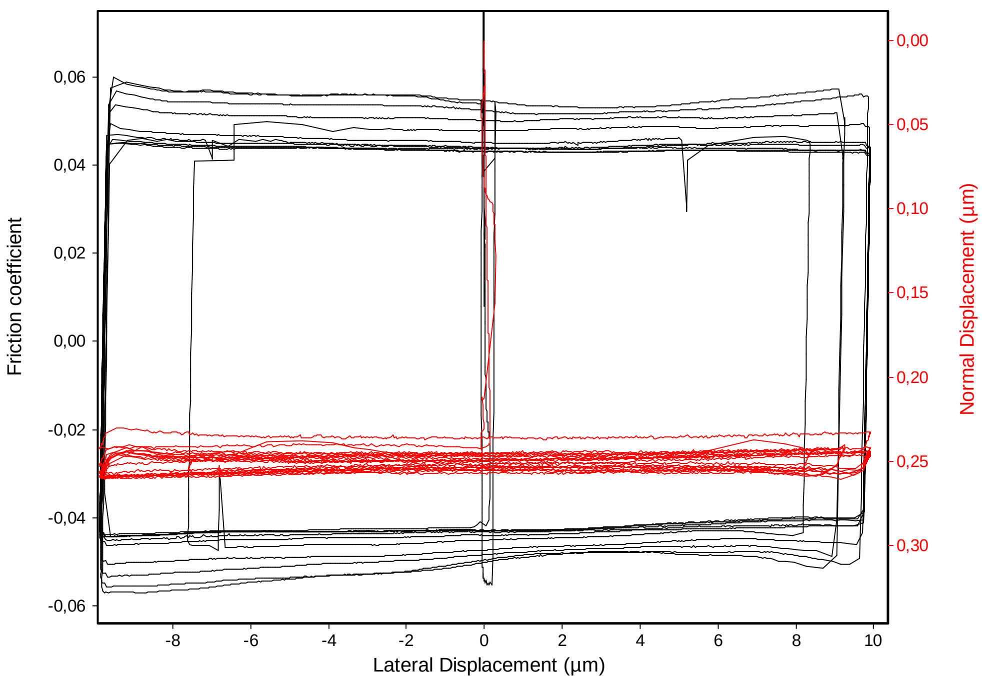
Av. failure cycle
71 > 43

Hardness is not the only criteria for the durability of a coating.

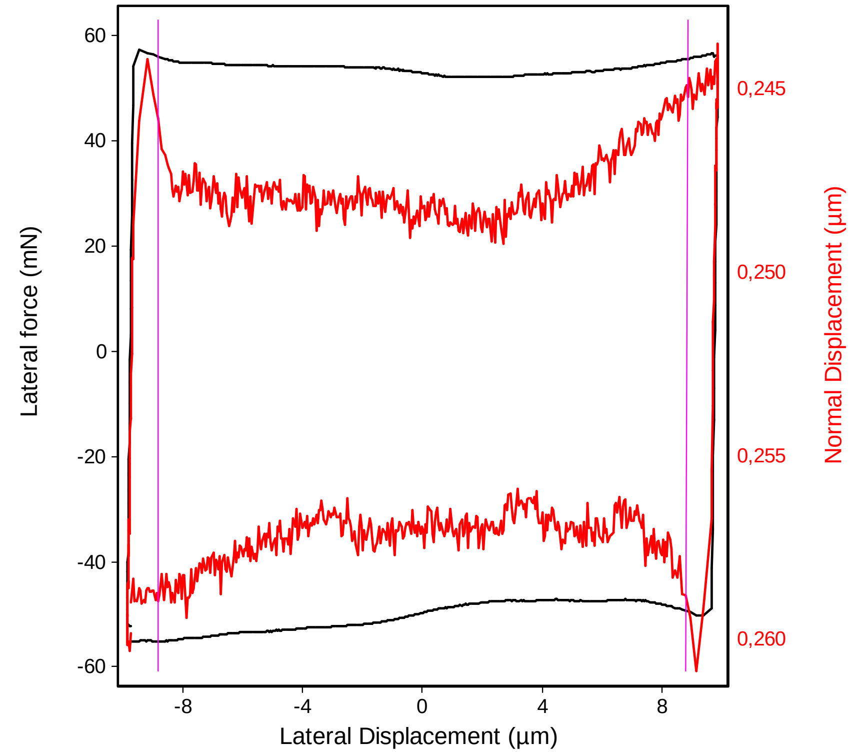
Micro wear tests

Example: **Dry** friction of diamond (Rockwell indenter) against diamond layer

Minimum friction: 0.04 Normal force: 1N

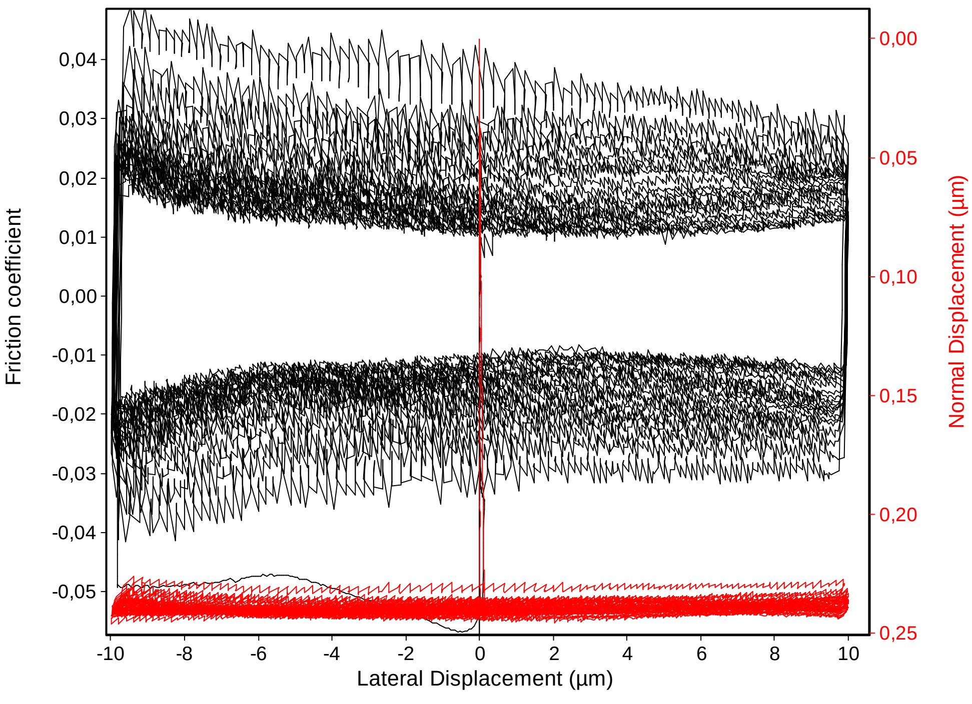


10 cycles in one graph

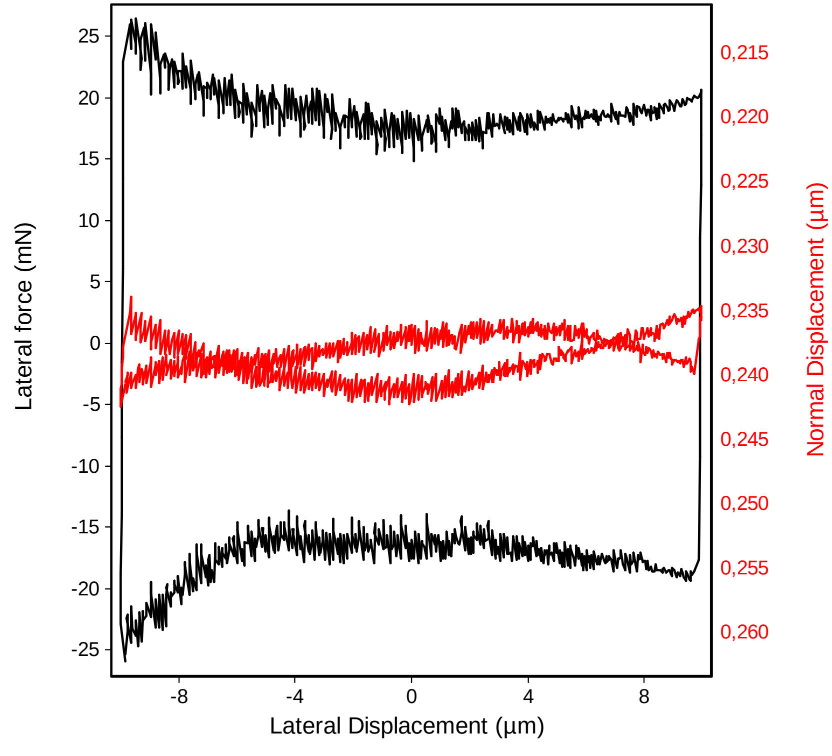


Cycle 9

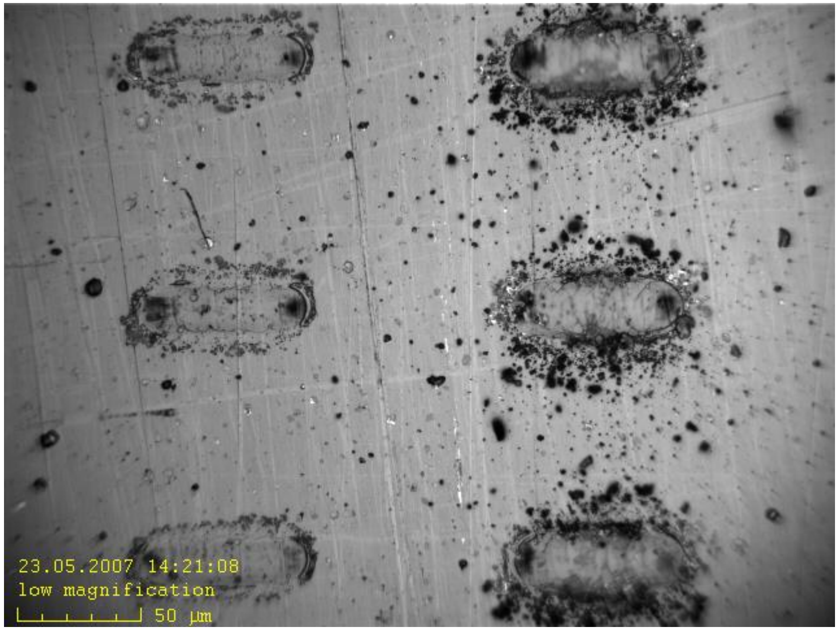
Example: **Wet** friction of diamond (Rockwell indenter) against diamond layer
Minimum friction: 0.01 Normal force: 1N



20 cycles in one graph



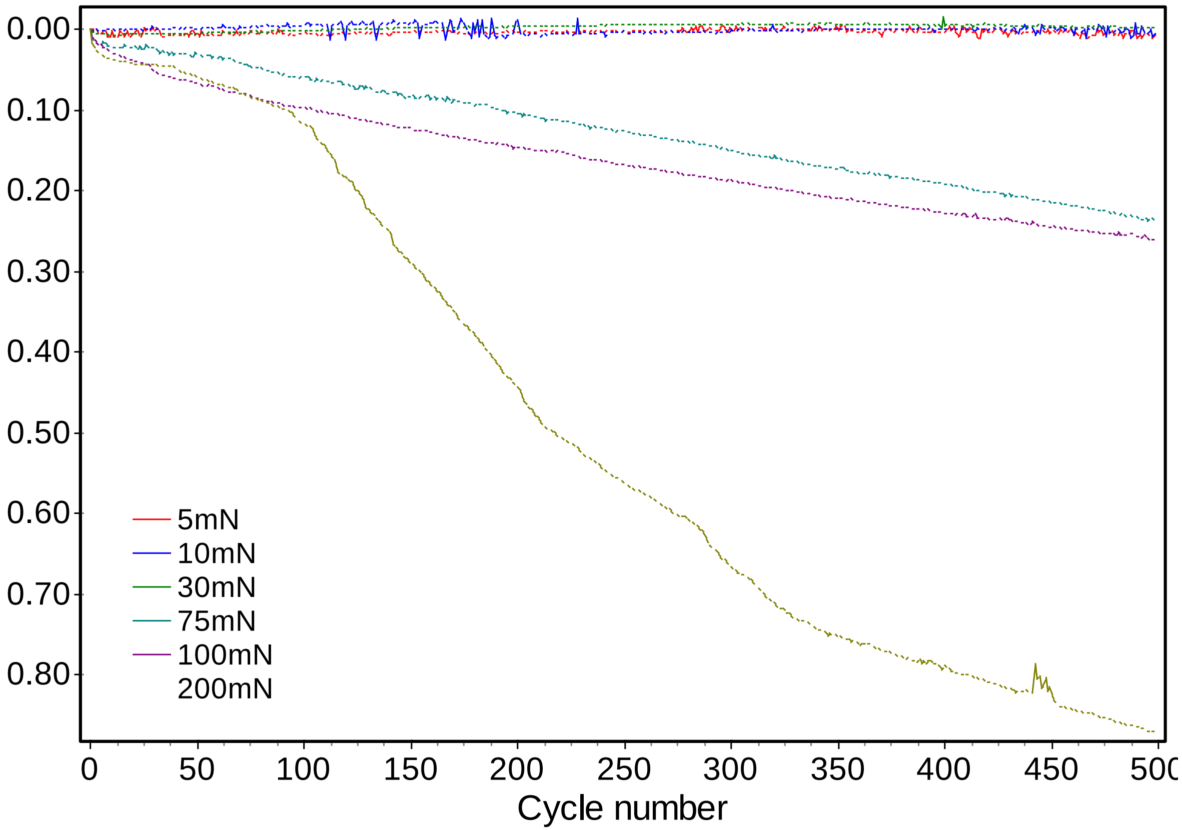
Cycle 9



2.5µm DLC on steel, 55µm tip radius
Amplitude 50 µm, Measuring time 1800 s

CVD DLC on steel

5,7µm film thickness, Film modulus: 100 GPa, Hardness: 12 GPa
Indenter: diamond sphere, 6 µm radius
Displacement amplitude 80µm measurement time 3144s



No wear until 50mN normal force
Wear rate at 75mN and 100mN: 0,4nm/cycle

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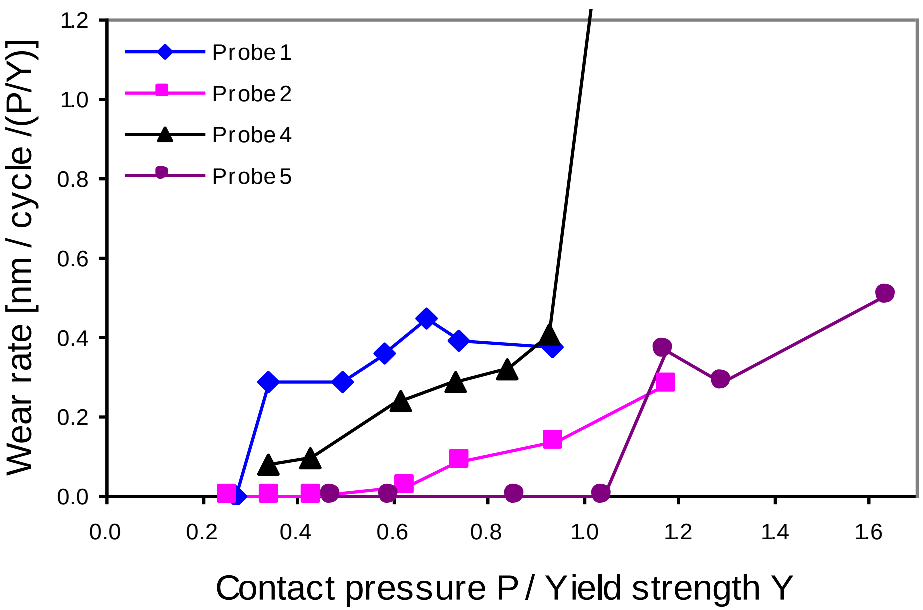
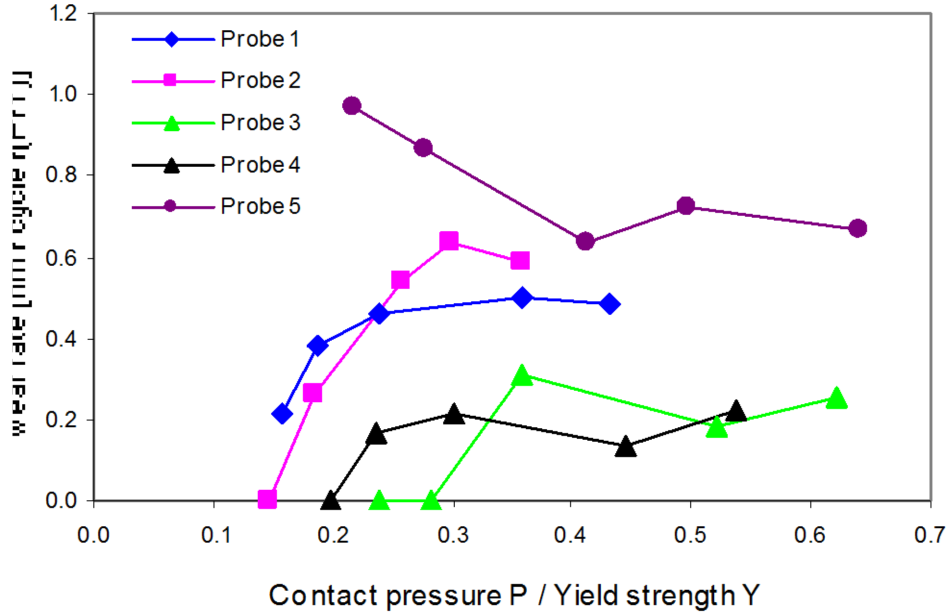
Wear tests on different DLC coatings (a-C:H and t-aC)

Tests with diamond indenter of 6 µm radius

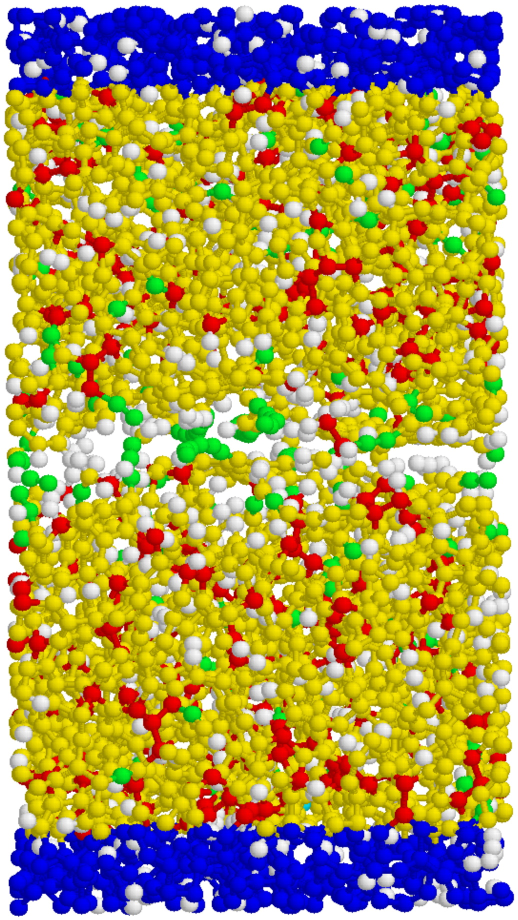


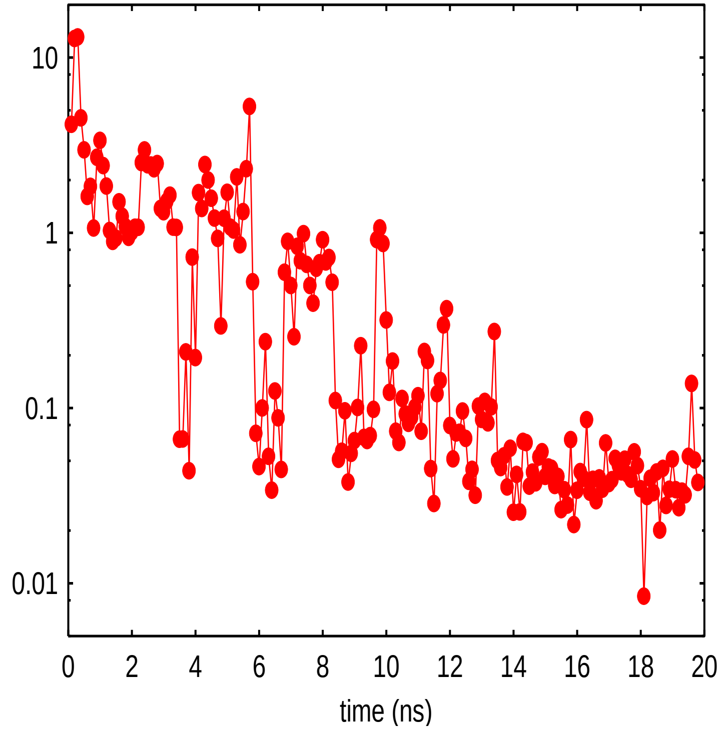
67 µm radius

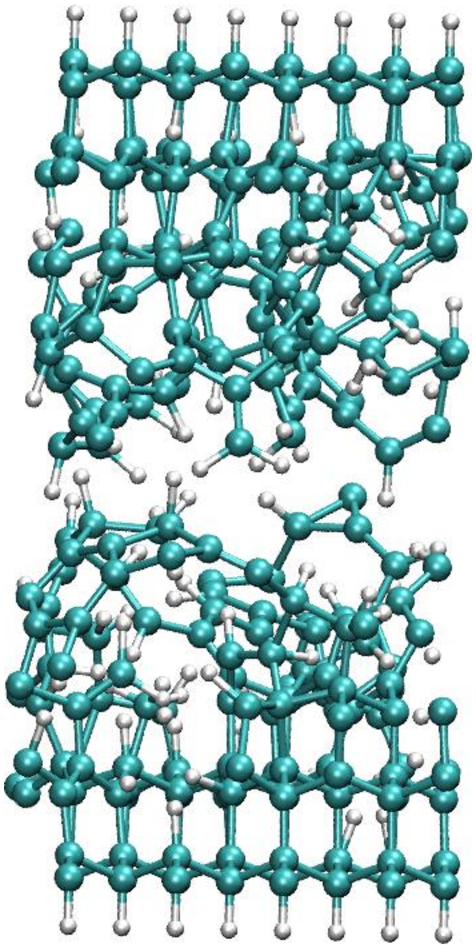


Normalization with yield strength Y (X-axis)
and P/Y- ratio (Y-axis)

Moleculardynamic simulation of running in behavior of DLC → graphitization observed

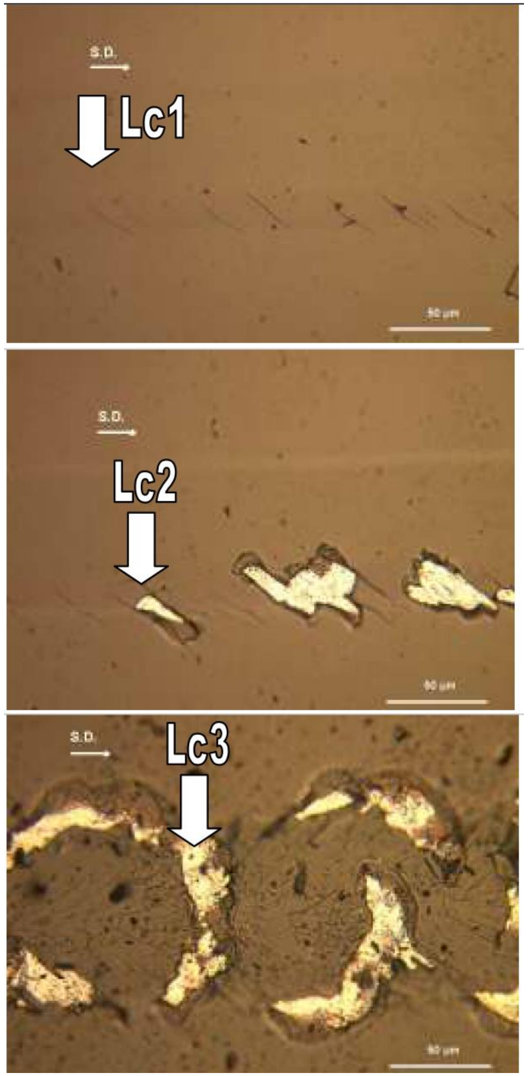






Source: Moseler, Fh IWM, Freiburg, Germany

Micro scratch test as adhesion test



Scratch analysis via definition of critical forces

L_{C1} - first cracks in the scratch track

L_{C2} – local interfacial spallation

L_{C3} – coating removal and visibility of substrate

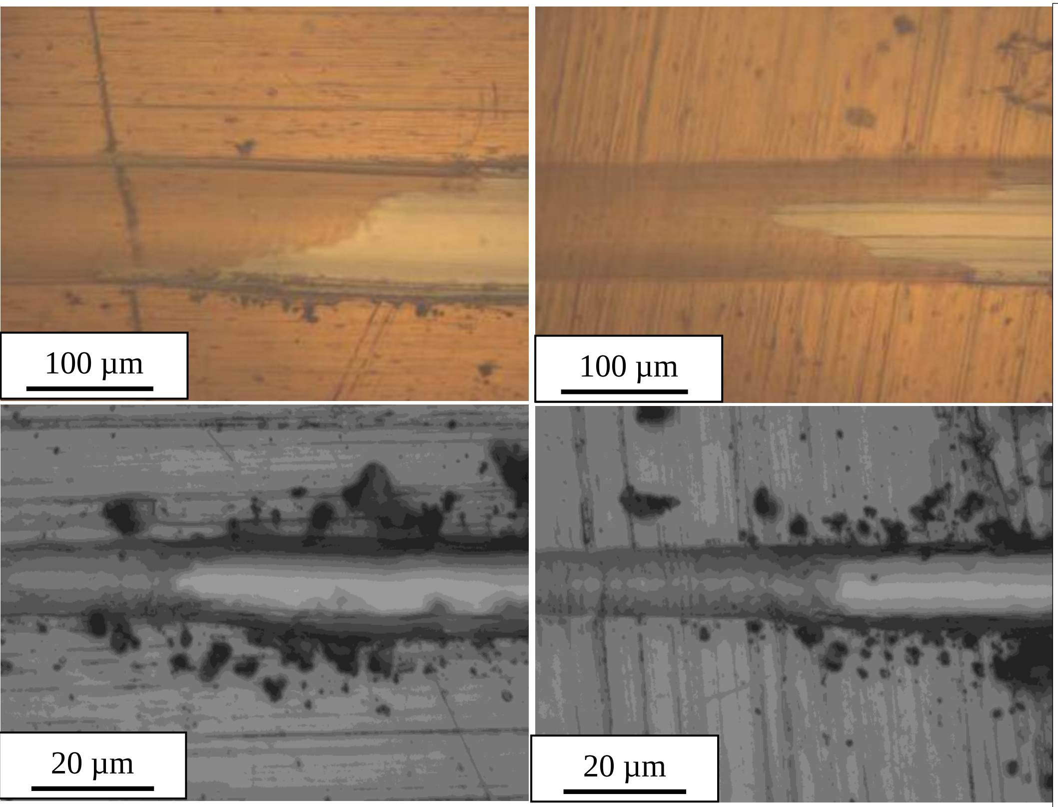
The indication C1 .. C3 depends on the occurrence of the failure mode

Images from: The certification of critical coating failure loads: a reference material for scratch testing according to ENV 1071-3: 1994 IRMM (European Institute for Reference Materials)

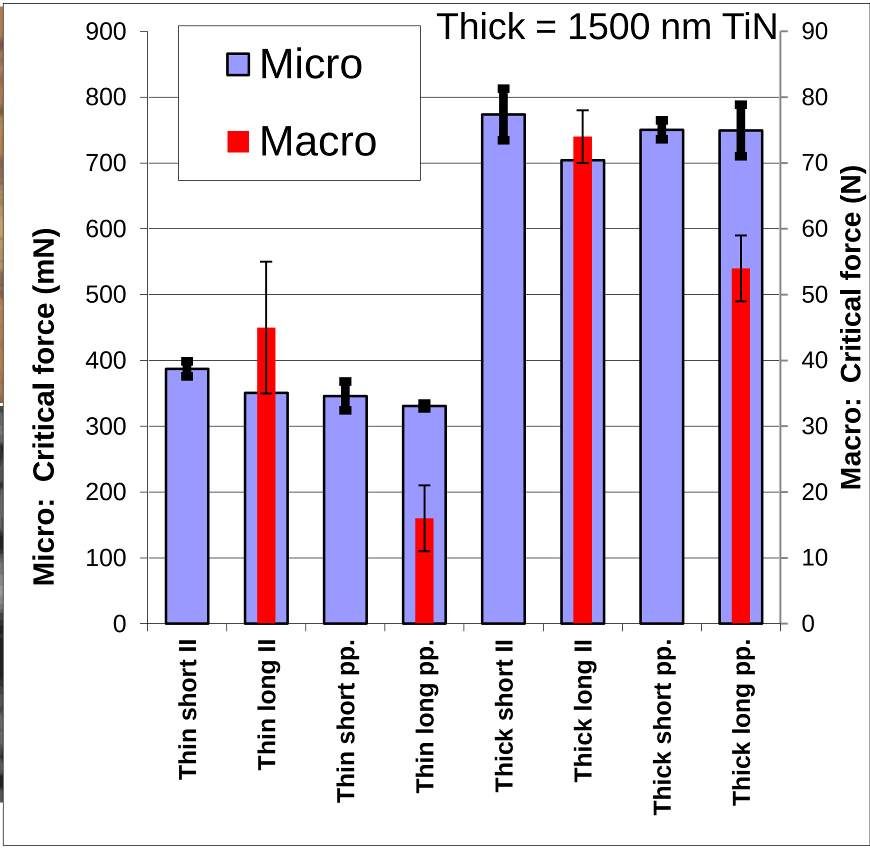
Macro scratch test versus micro scratch test

(from EU project Nanoindent)

550 nm TiN on steel



Thin = 550 nm TiN
Thick = 1500 nm TiN



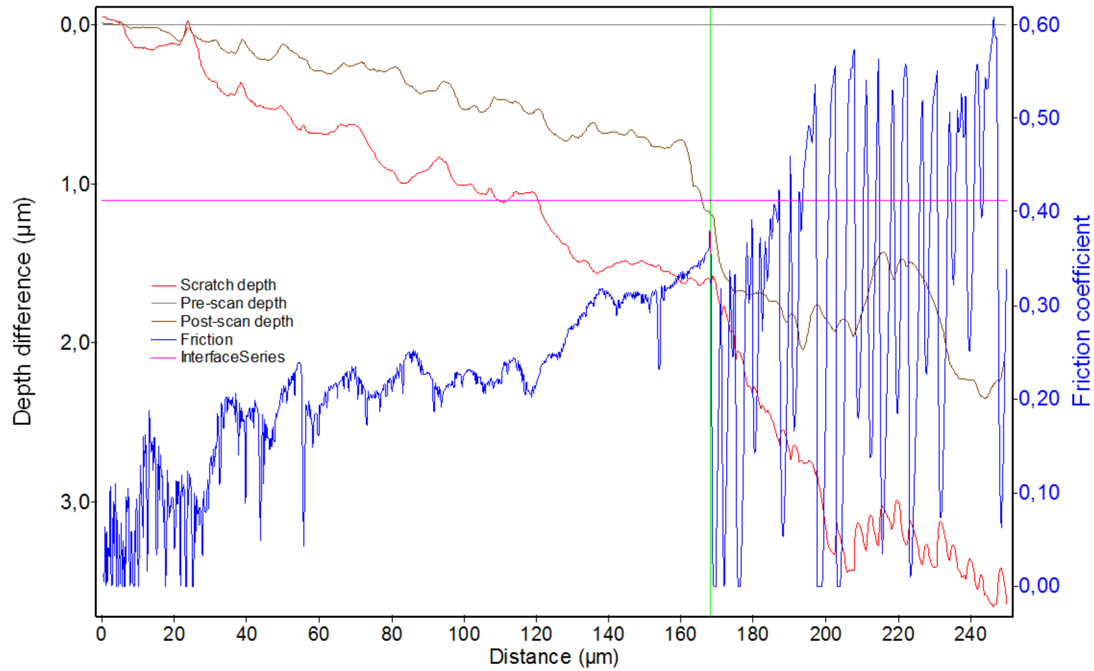
For macro scratch test there is a clear dependency from the orientation of the roughness profile.
parallel = pp perpendicular = II.

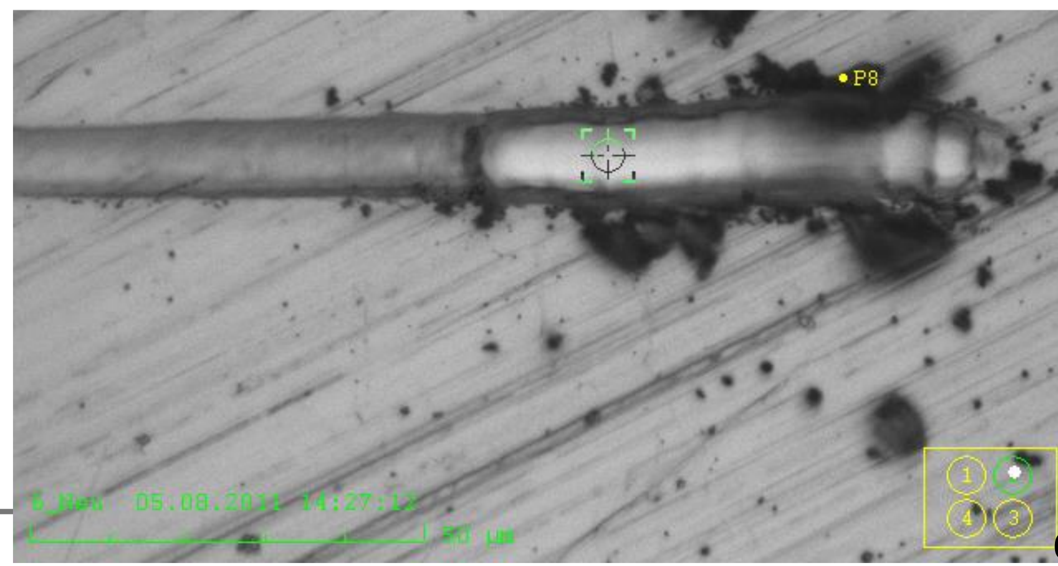
Example 1:

1,1 µm TiN on steel

10,4 µm tip radius

F_{C3} = 662 mN



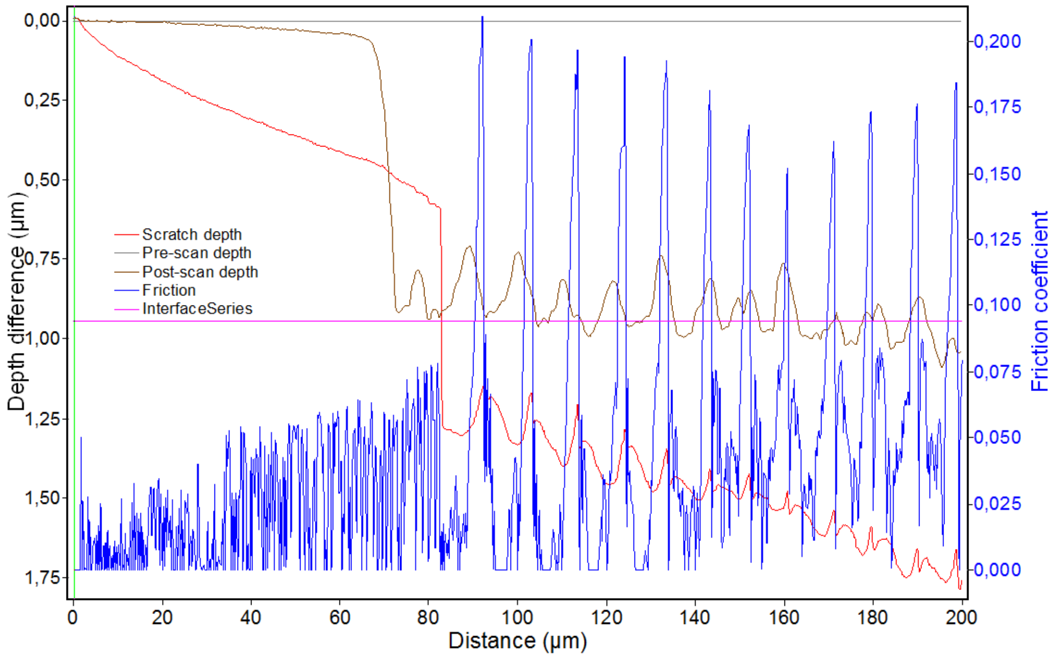


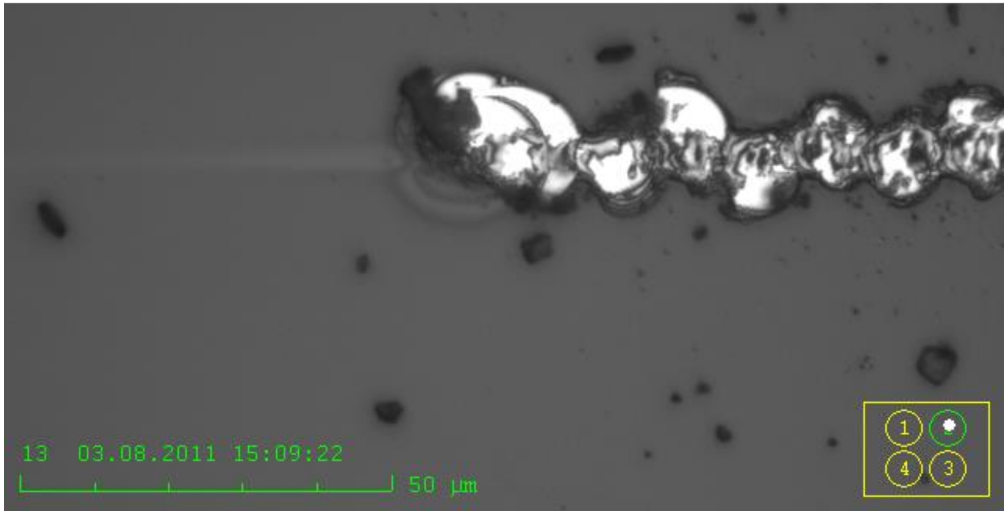
Example 2:

0,94 µm soft DLC on Si

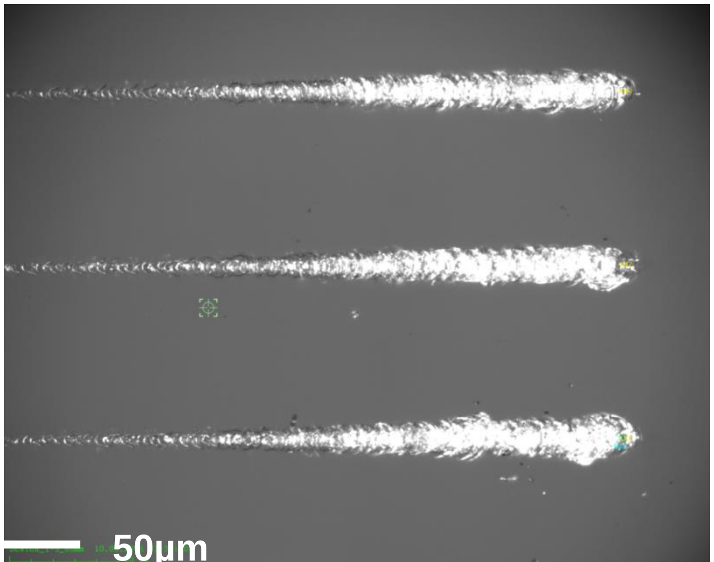
10,4 µm tip radius

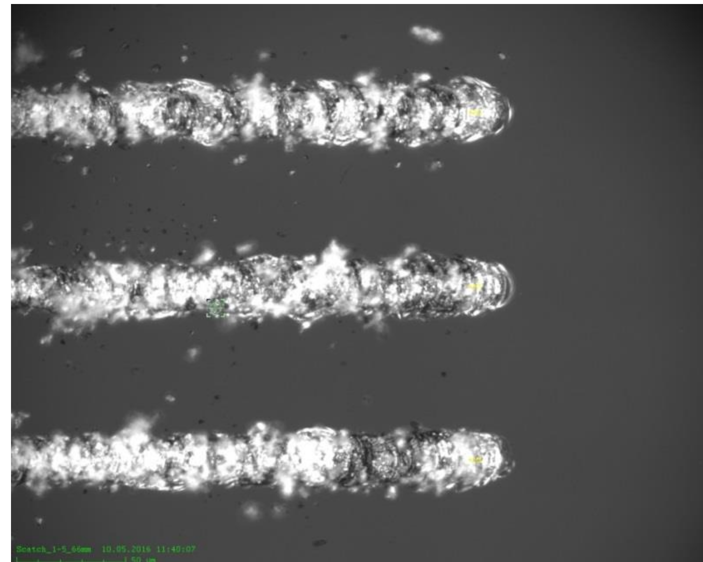
F_{C3} = 136 mN

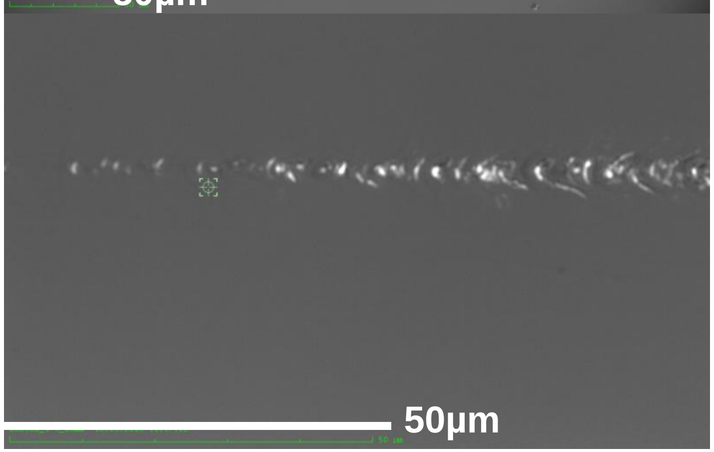


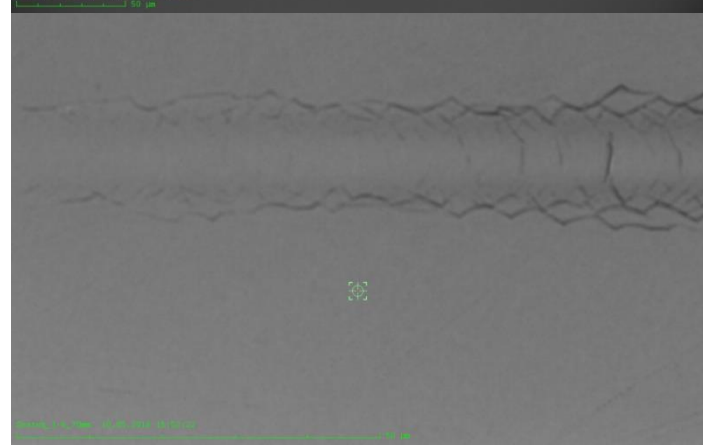


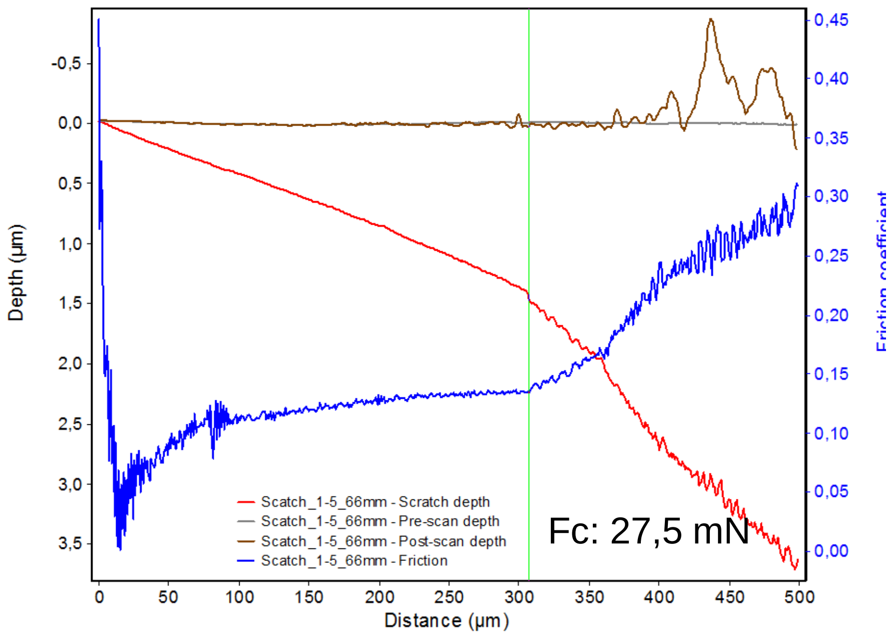
Example 3: Polymer lenses with hard varnish and anti reflective coating

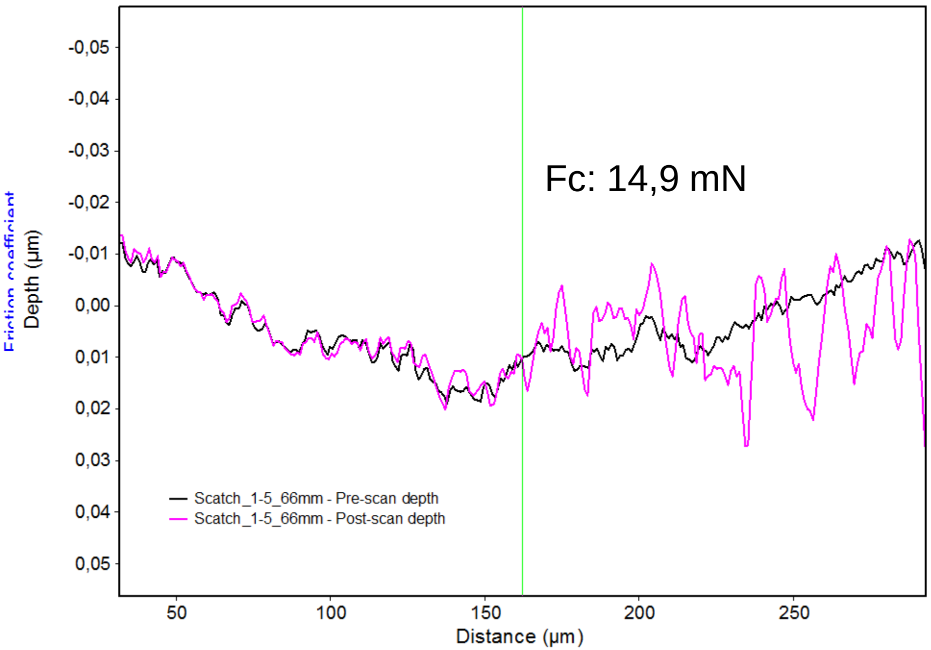


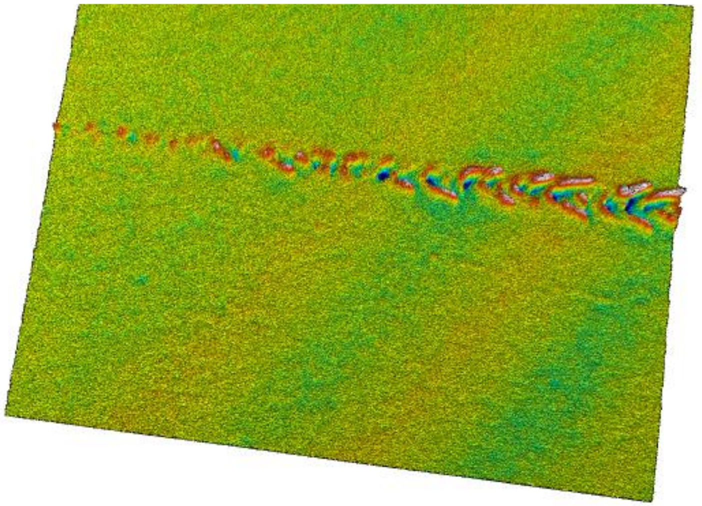




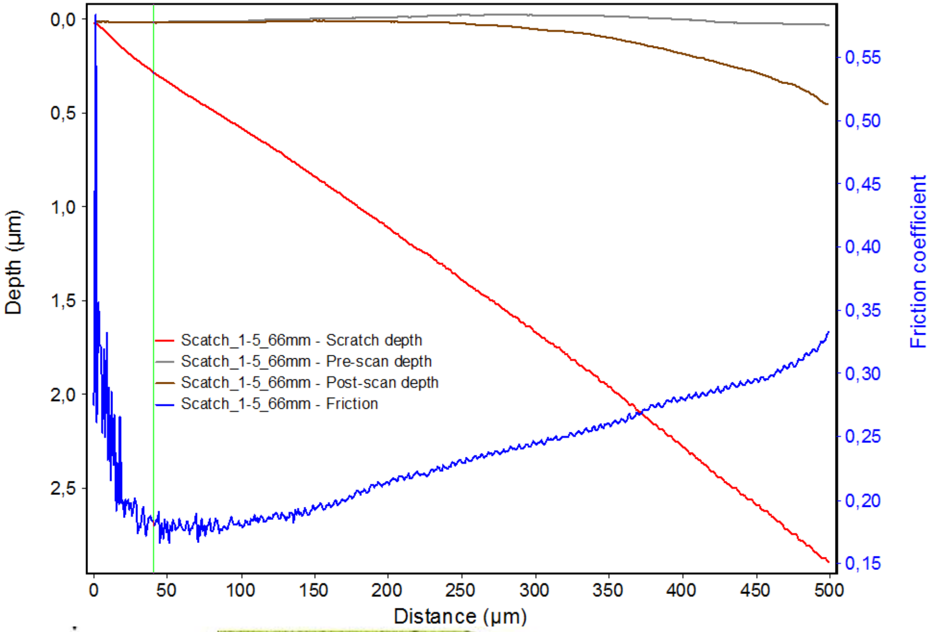



Depth (µm)
-0,5
0,0
0,5
1,0
1,5
2,0
2,5
3,0
3,5
Friction coefficient
0,45
0,40
0,35
0,30
0,25
0,20
0,15
0,10
0,05
0,00
0 50 100 150 200 250 300 350 400 450 500
Distance (µm)
Scatch_1-5_66mm - Scratch depth
Scatch_1-5_66mm - Pre-scan depth
Scatch_1-5_66mm - Post-scan depth
Scatch_1-5_66mm - Friction
Fc: 27,5 mN

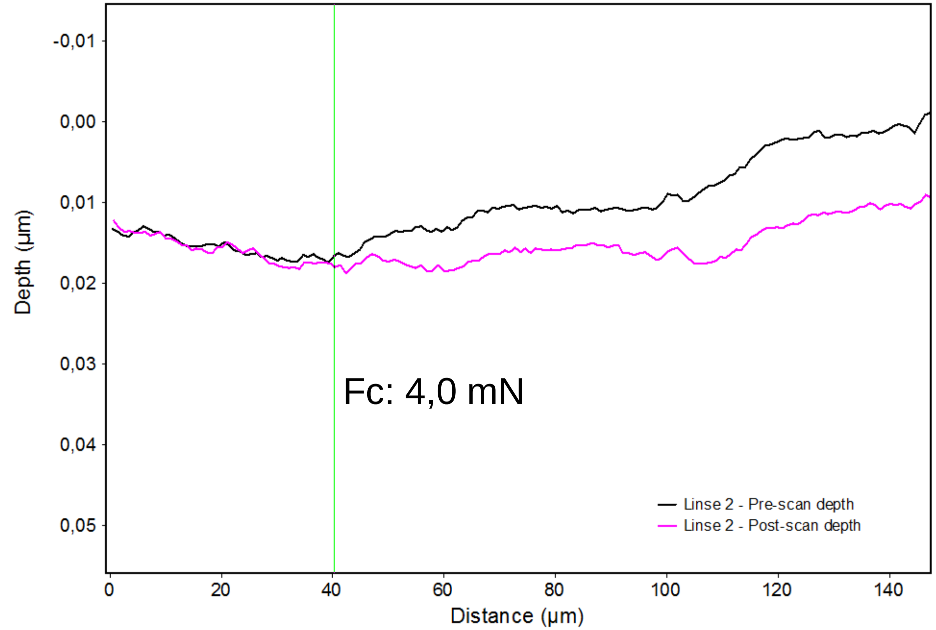

Depth (µm)
-0,05
-0,04
-0,03
-0,02
-0,01
0,00
0,01
0,02
0,03
0,04
0,05
50 100 150 200 250
Distance (µm)
Fc: 14,9 mN
Scatch_1-5_66mm - Pre-scan depth
Scatch_1-5_66mm - Post-scan depth



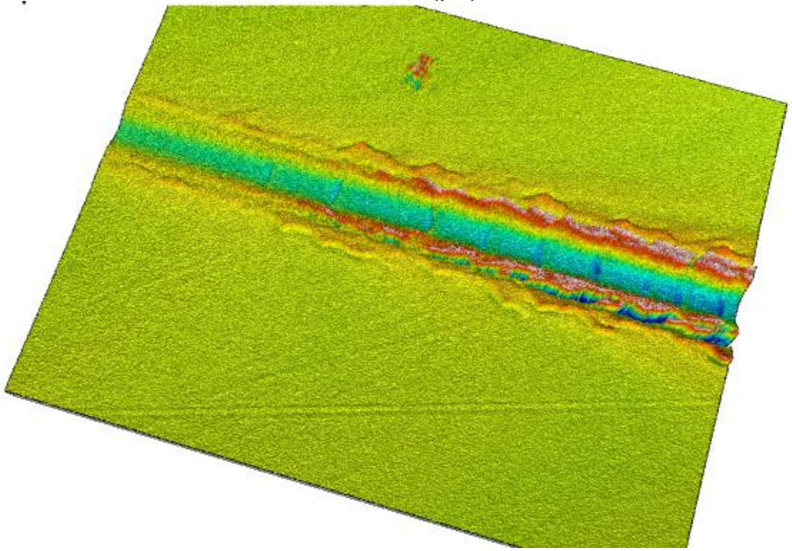
Lens 2

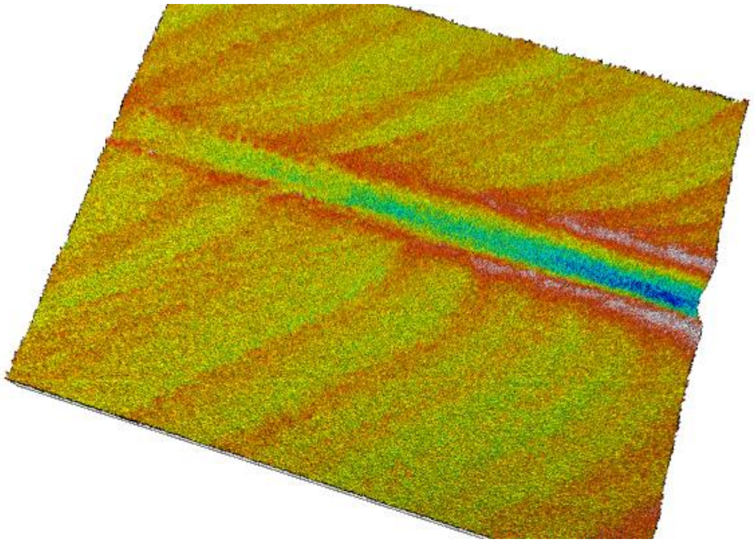


Depth (µm)
Friction coefficient
Distance (µm)
Scatch_1-5_66mm - Scratch depth
Scatch_1-5_66mm - Pre-scan depth
Scatch_1-5_66mm - Post-scan depth
Scatch_1-5_66mm - Friction



Depth (µm)
Distance (µm)
Fc: 4,0 mN
Linse 2 - Pre-scan depth
Linse 2 - Post-scan depth





Mapping of mechanical properties

Local versus global properties

The weakest link is deciding about the durability of a system.
Global tests are therefore advantageous.

Property	***Global tests***	***Local tests***
Yield strength	Tensile test	Indentation test
Young's modulus	Tensile test	Indentation test, Ultrasonic test
Adhesion	Cavitation test	Scratch test

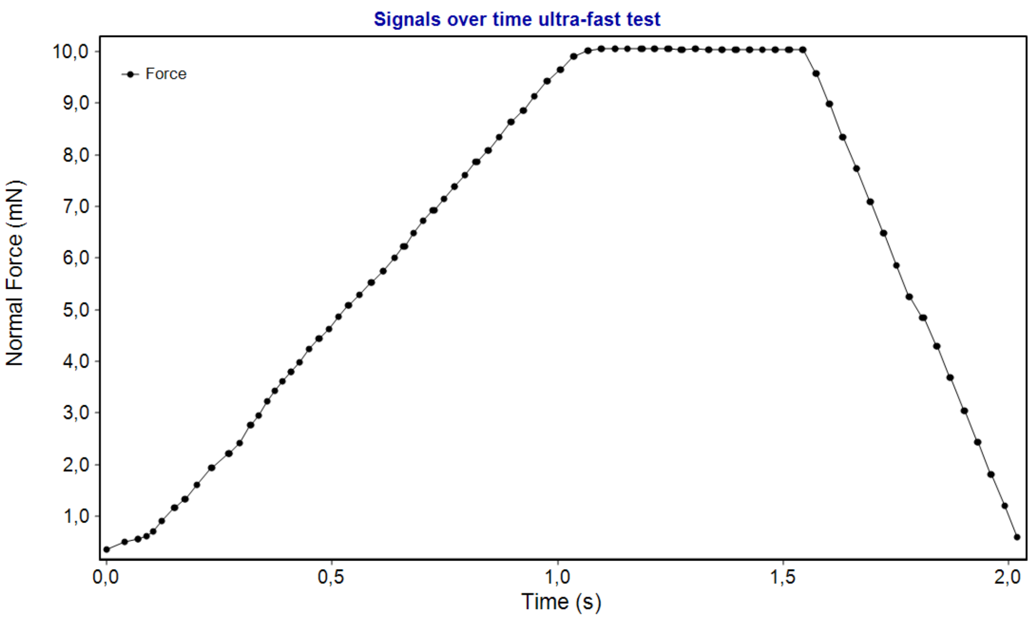
The mapping of local mechanical properties is a step towards a global characterization.

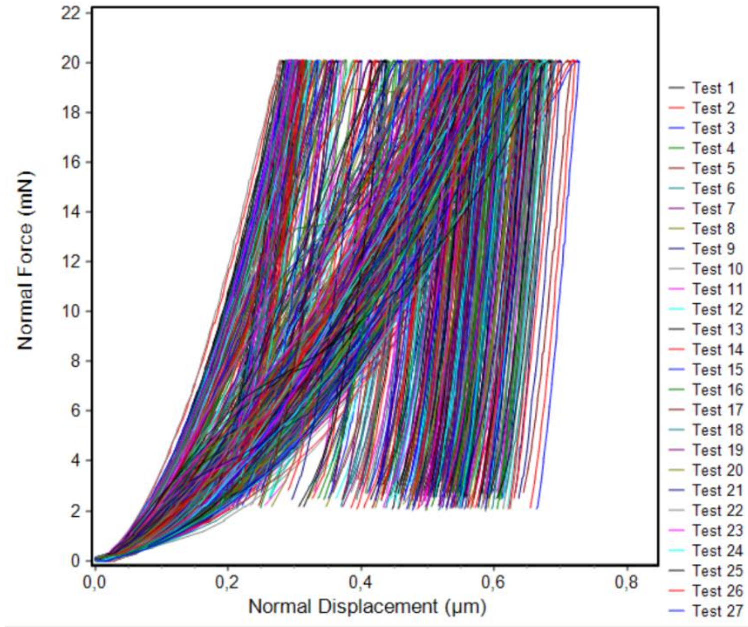
Ultra fast hardness tests with mapping function

- Test time recommended according standard: 130s + 30s Approach = 160s * 400 = 1067min = 44h
- Test time for fast hardness test: 19s + 30s Approach = 49s * 400 = 327min = 13,6h
- Test time for ultra fast hardness tests: 2s + 3s Approach = 5s * 400 = 34min = 0,5h

Example: Ultra fast tests with a typical data rate of 64 Hz

400 load-displacement curves





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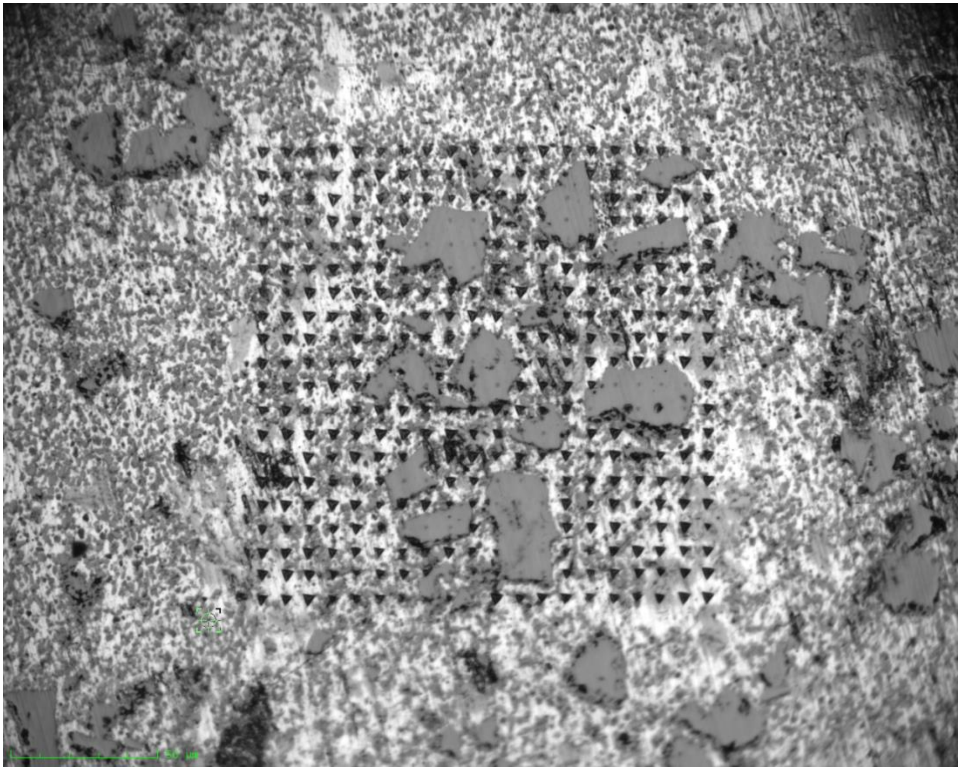
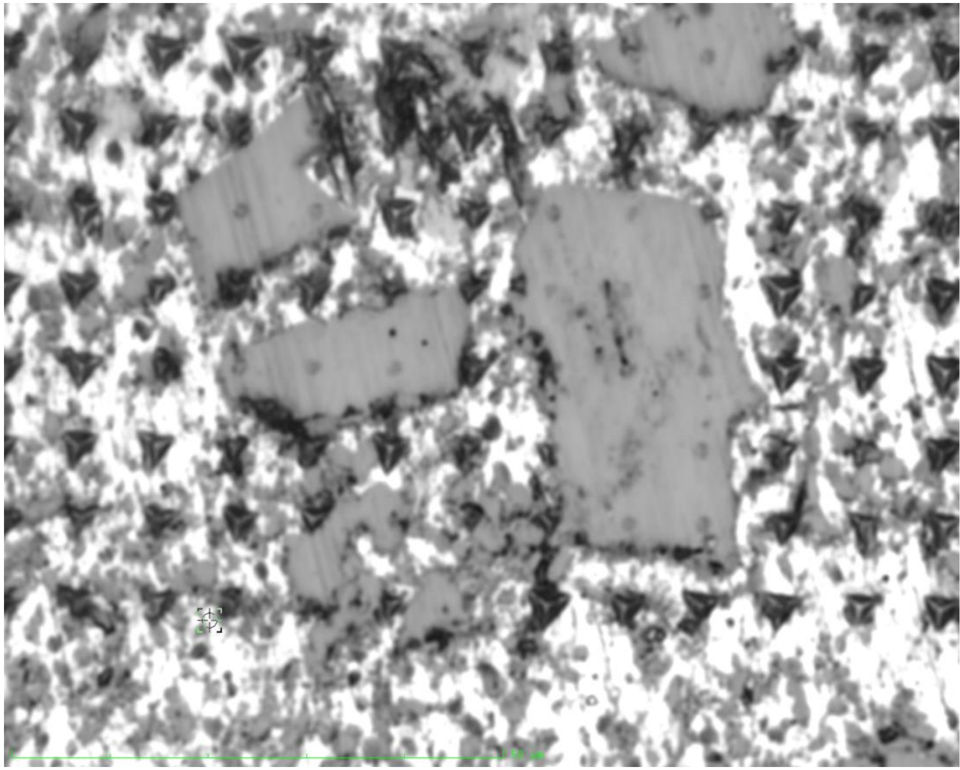
Example 1: Hardness and modulus mapping

Sample: Al-Si composite (Al matrix with Si particles) , experimental cylinder liner from Yamaha

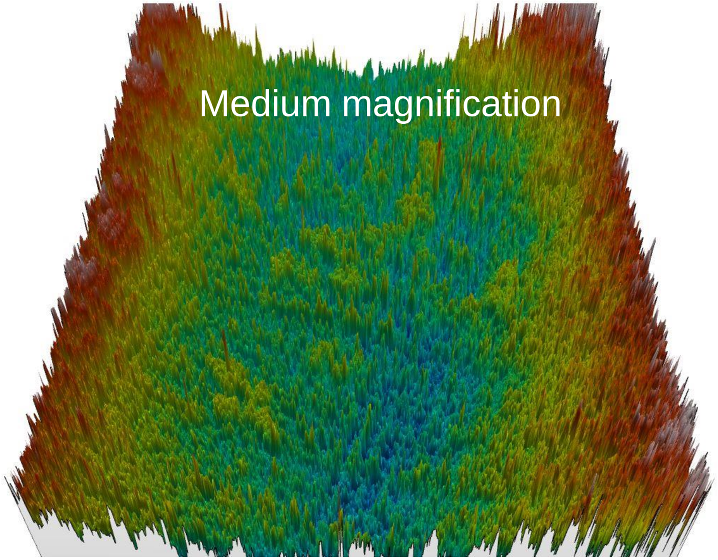
Test conditions: Maximum force: 20mN, Test time: 2s





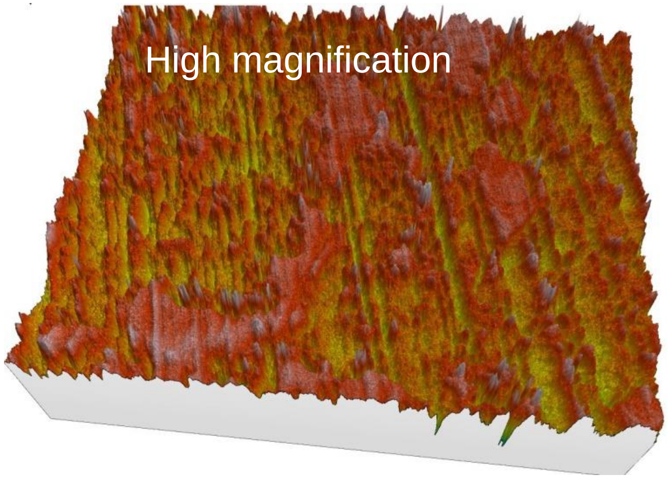
50 µm

50 µm

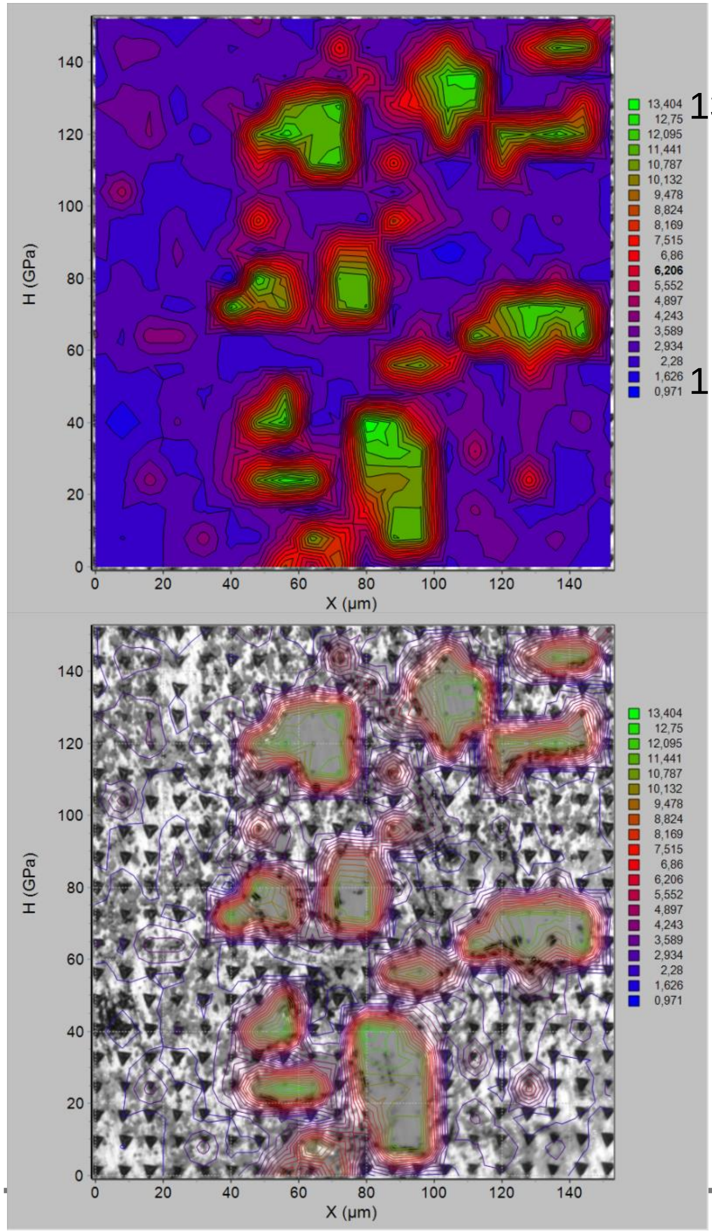


Measurement of topography with green light interferometer

High surface roughness is making tests more difficult

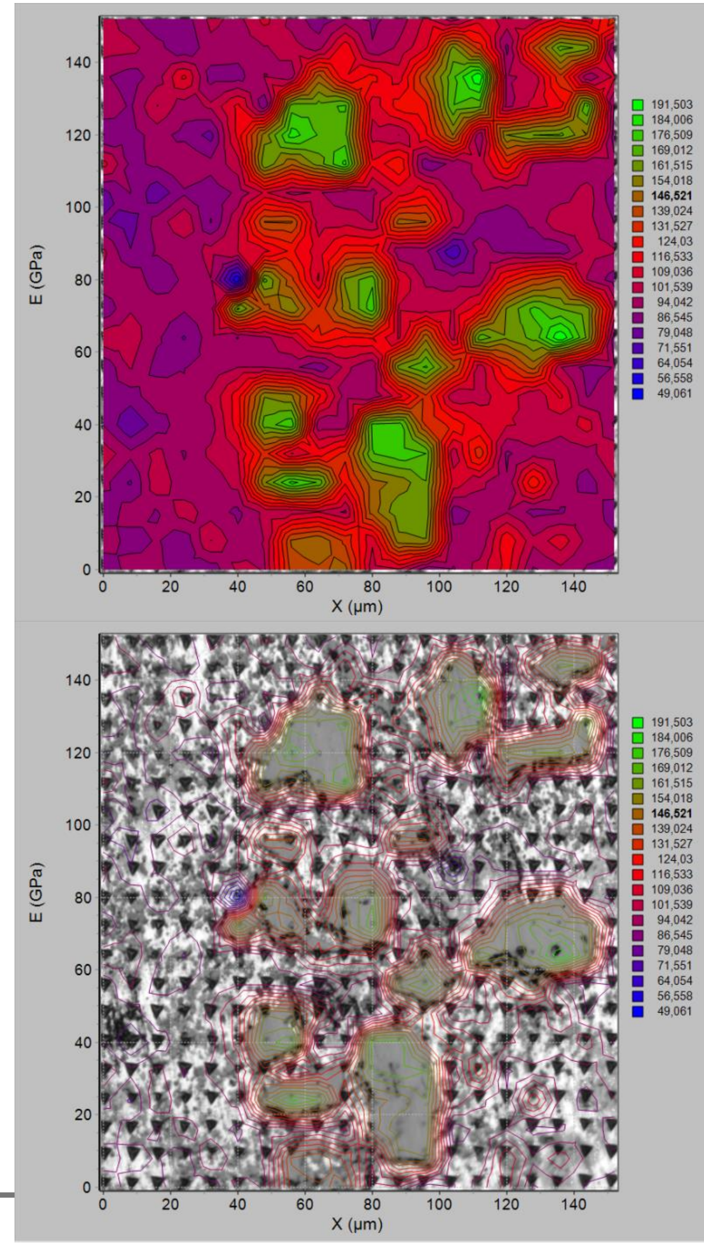


Hardness (left) and E-Modulus



13 GPa

1 GPa

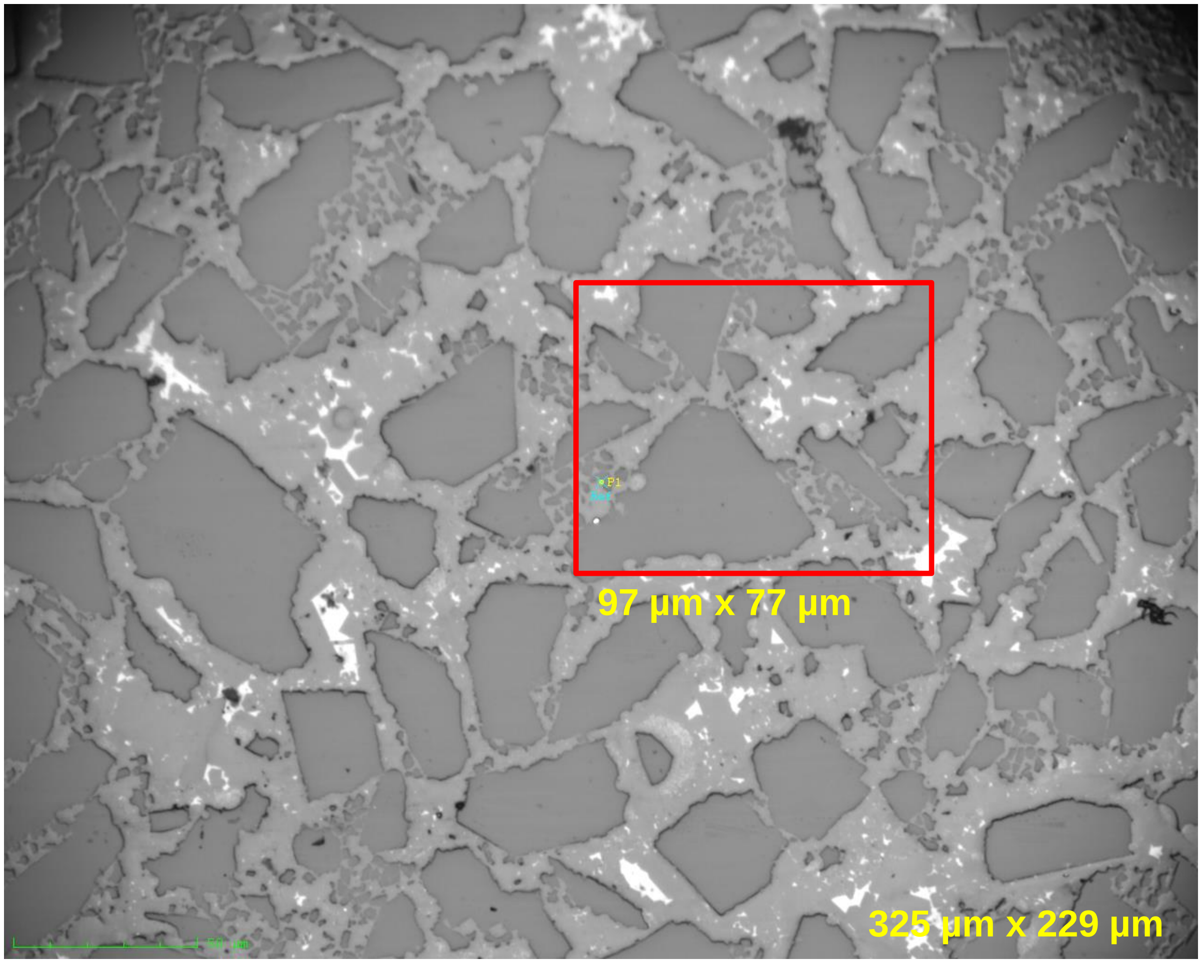


190 GPa

50 GPa

Example 2: Topography, modulus and friction mapping

Sample: Diamond - SiC composite



Test parameters:
Indenter:
Sphere with 10,48µm radius
Contact diameter
on Diamond: 1,5 µm
on SiC: 1,8 µm

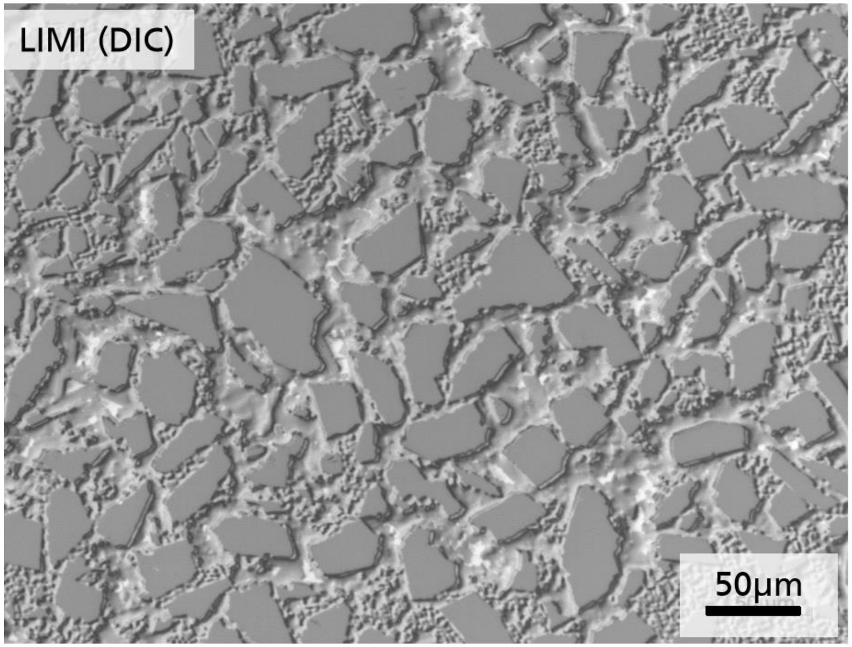
45 lines in a distance of 1.7µm
Test time: 1600s

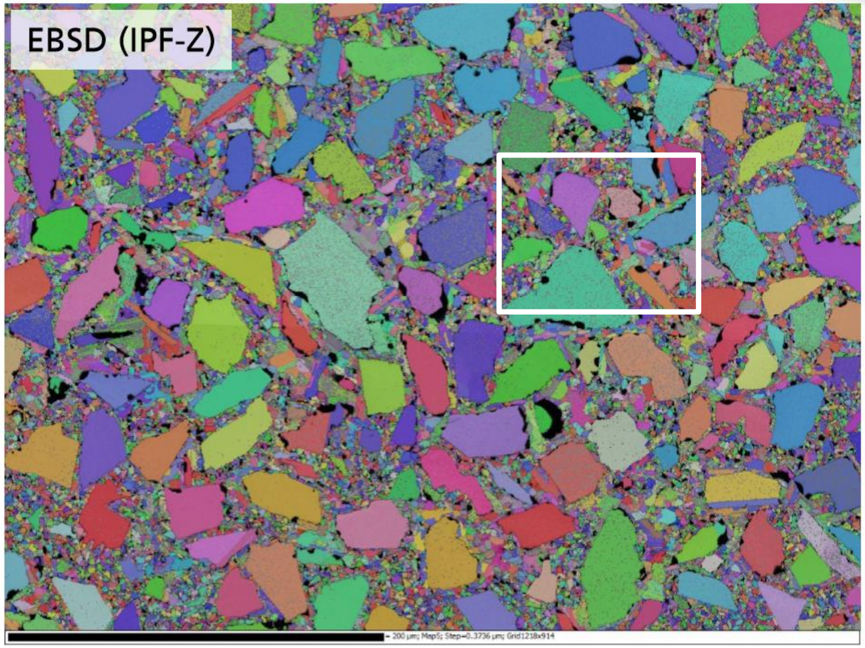
Scan time per line: 20s
Scan force: 30mN
Data rate 8 Hz
Oscillation frequnecy: 40 Hz
Amplitude: 0,22V ≈ 3nm

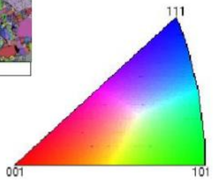
EBSD measurements of diamond crystals done at Fraunhofer IKTS

Diamant - SiC

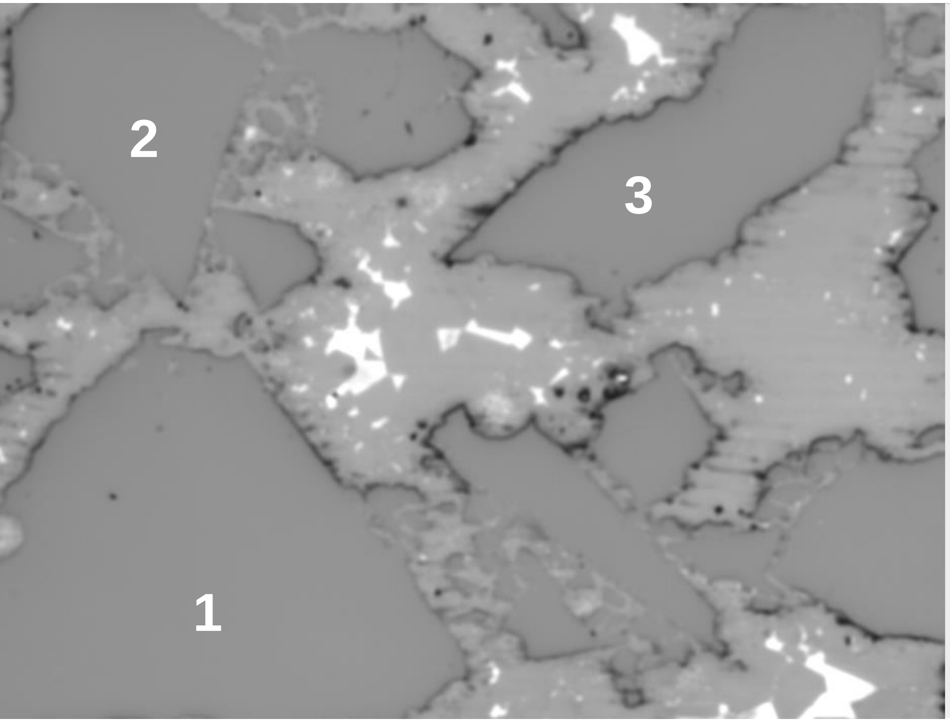
LIMI

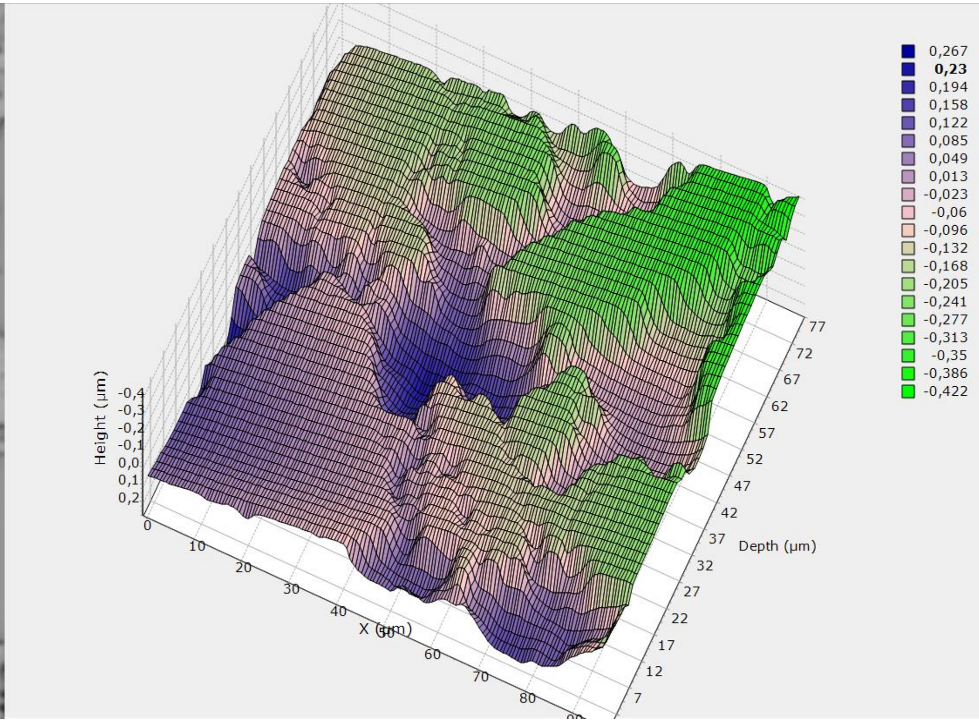




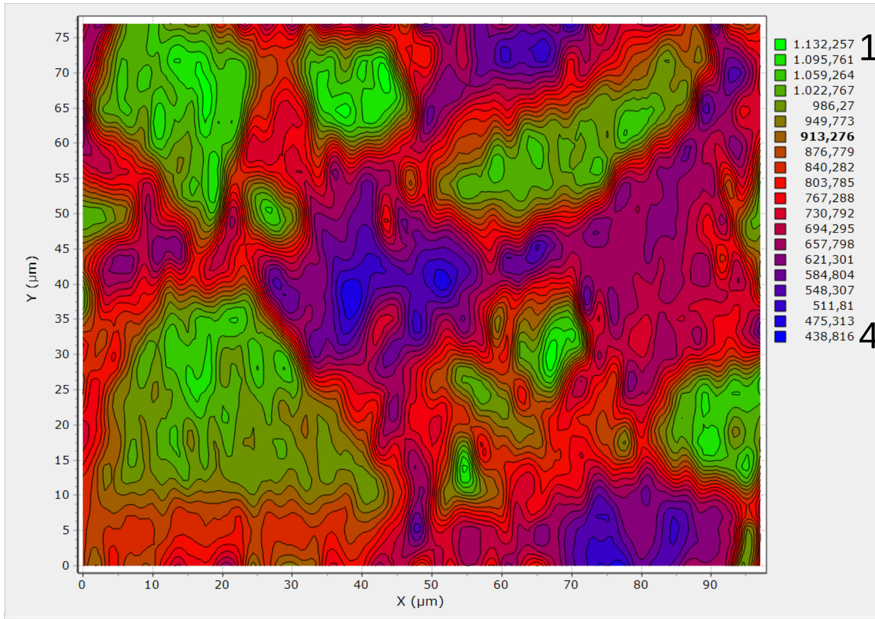


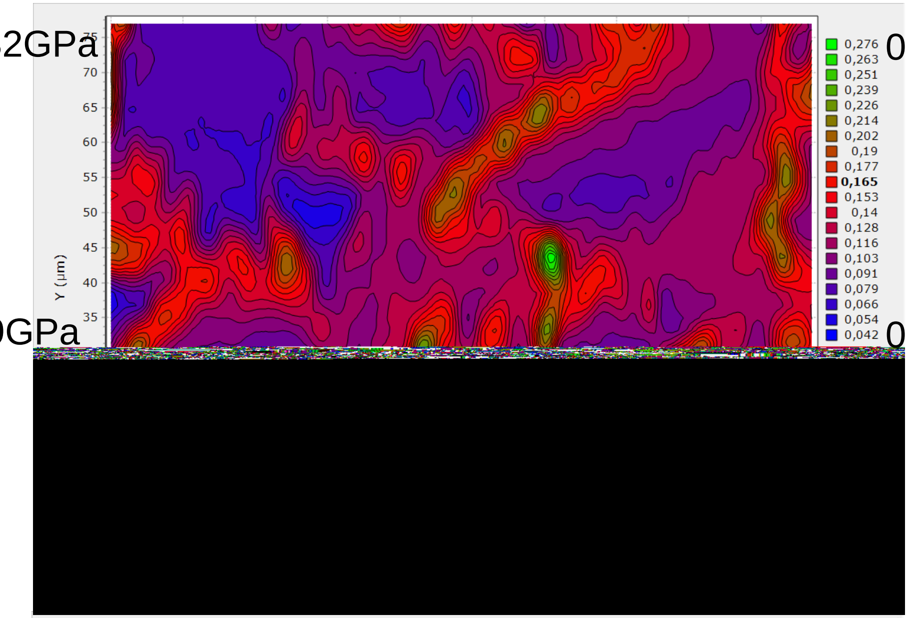
Topography

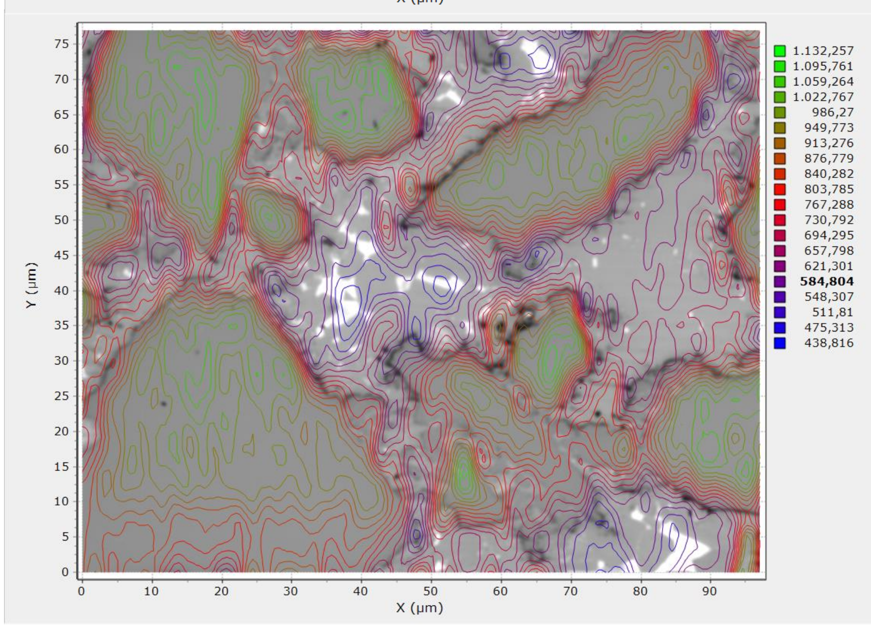

2
3
1

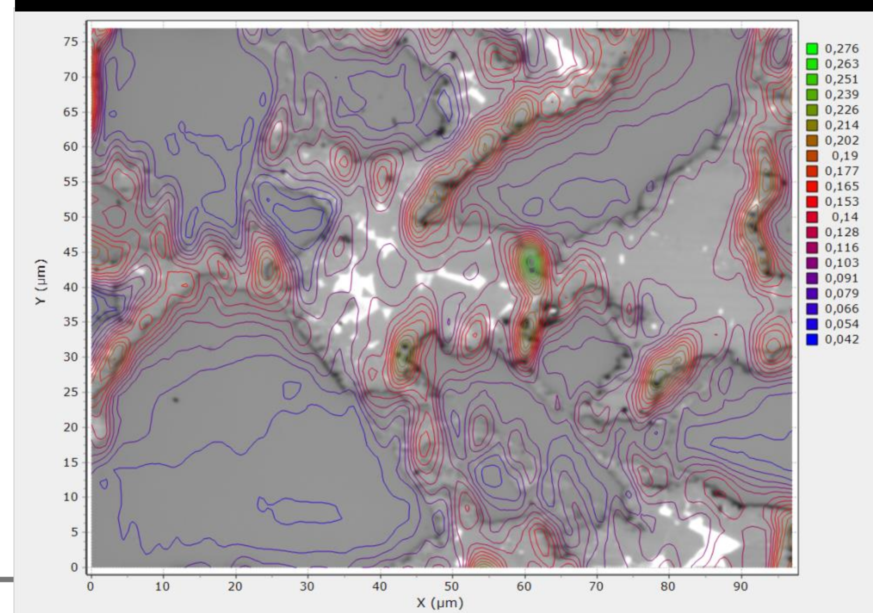

0,267
0,23
0,194
0,158
0,122
0,085
0,049
0,013
-0,023
-0,06
-0,096
-0,132
-0,168
-0,205
-0,241
-0,277
-0,313
-0,35
-0,386
-0,422
Height (µm)
Depth (µm)
X (µm)

E-Modulus (left) and Friction

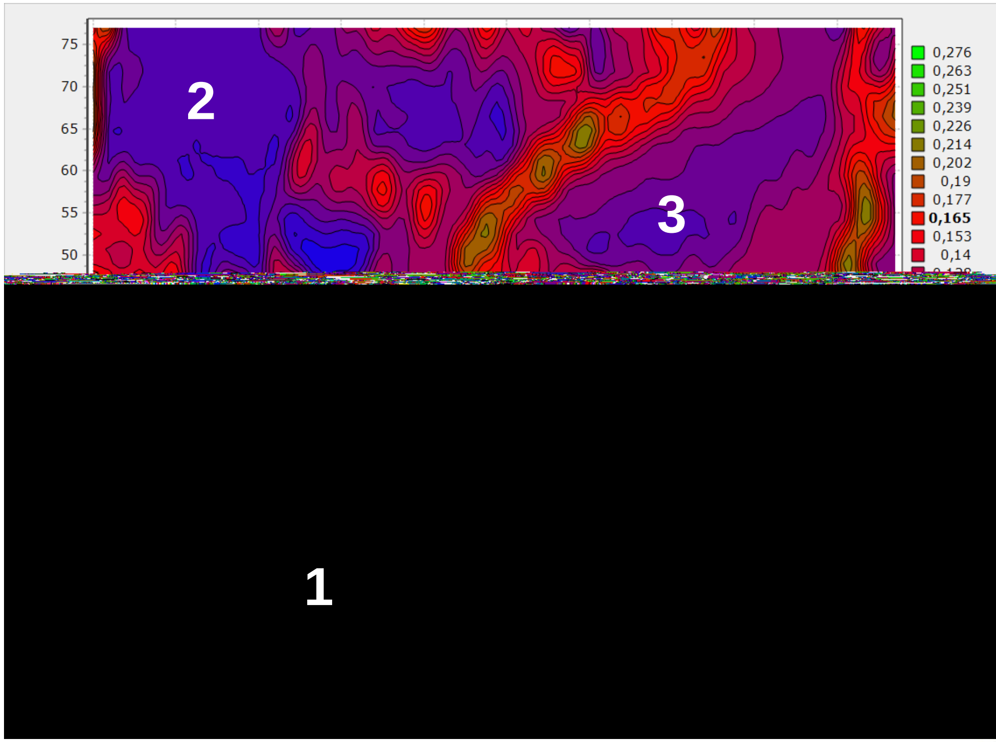


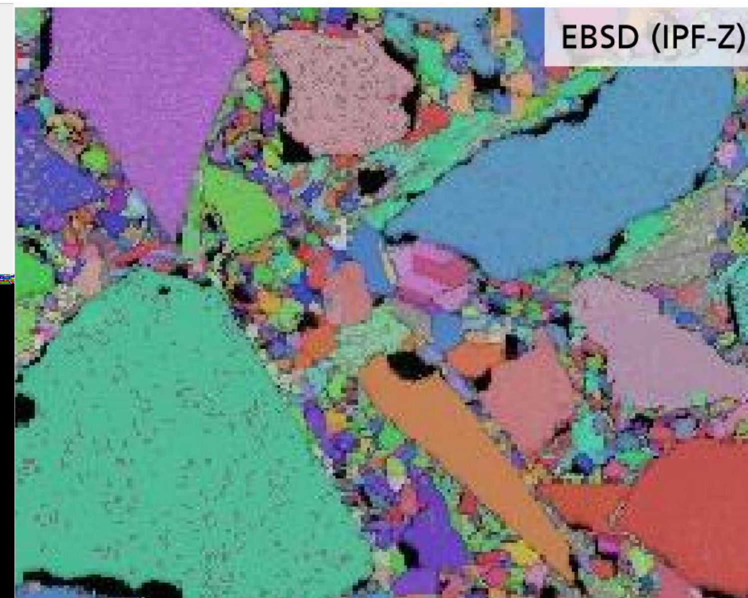


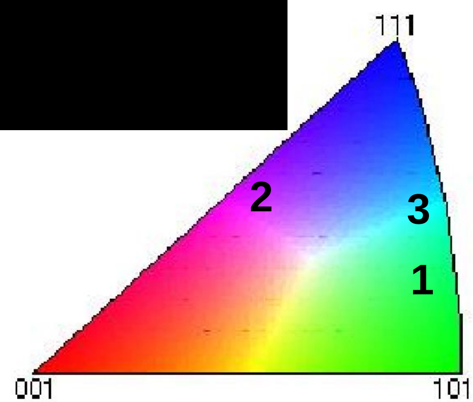


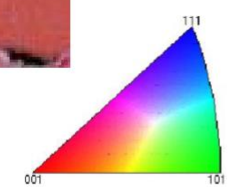


Friction coefficient





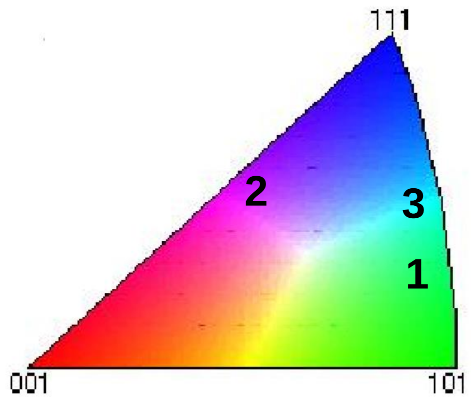


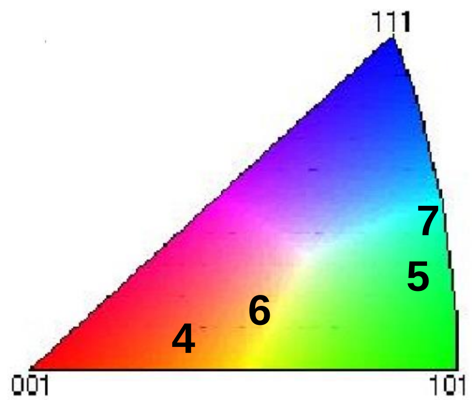


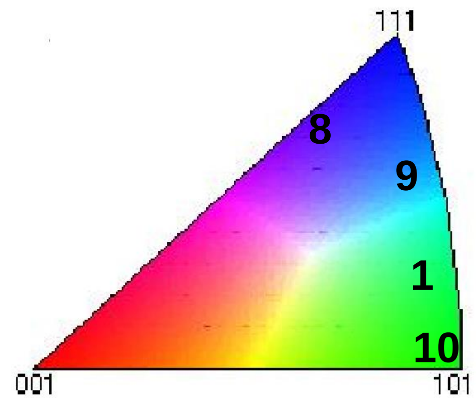
Friction coefficient in dependence on diamond crystal orientation

Measurement of 9 different grains

1	2	3	
0,064	0,071	0,085	
(101)-(111)	(001)-(111)	(101)-(111)	
4	5	6	7
0,065	0,051	0,077	0,072
(001)-(101)	(101)-(111)	(001)-(101)-(111)	(101)-(111)
1	8	9	10
0,046	0,058	0,081	0,027
(101)-(111)	(111)	(001)-(111)	(101)







Conclusions

- Finding the mechanically best behaving coating-substrate combination for a certain application is time consuming and expensive.
- The quantification of the mechanical behavior is still a challenge. Not all necessary mechanical parameters of coatings can be measured with standard test methods up to now.
- For the understanding of failure mechanisms it is necessary to measure with nanometer resolution. A lot of different micro or nano-mechanical test methods are available now in one and the same instrument.
- A larger inclusion of modelling tools (FE, analytical) can considerably shorten the development process. For the calculations accurate and relevant mechanical parameters are necessary.
- A better reproduction of the conditions in an application is necessary in laboratory tests. This requires the inclusion of lateral force-displacement measurements with high precision
- In the future the significance of multi-axial testing in combination with stress calculation will increase.

Thank you for your attention !

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