Zwick / Roell Intelligent Testing

asmec ADVANCED SURFACE MECHANICS

3D micro scratch tests in combination with a comprehensive stress analysis – a new tool for the understanding of surface failures

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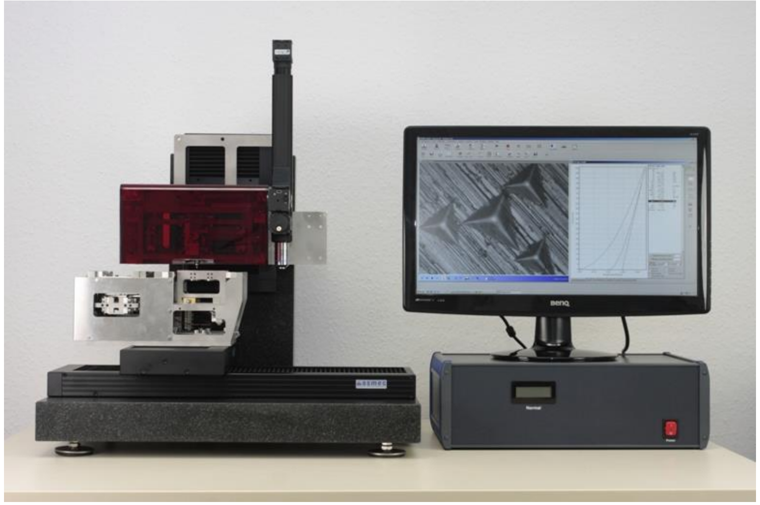
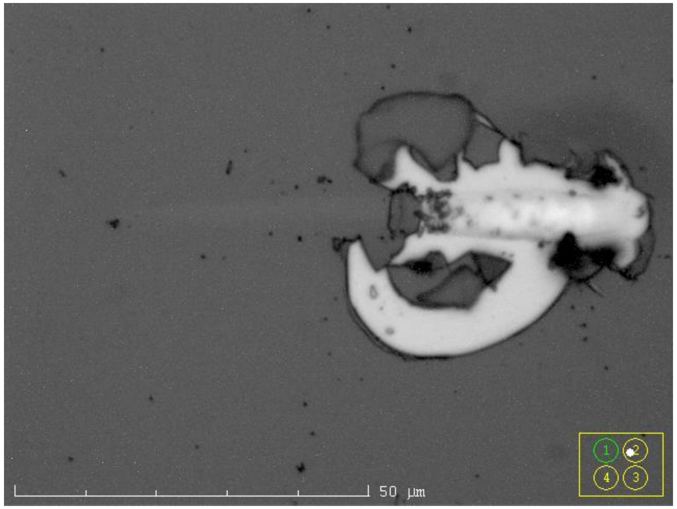


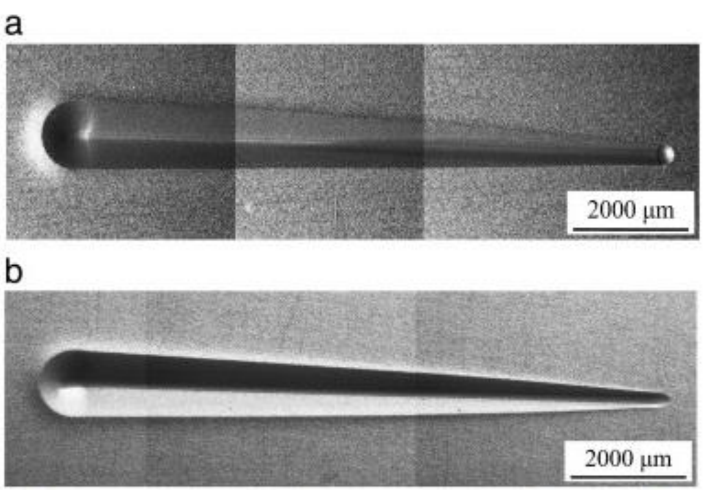


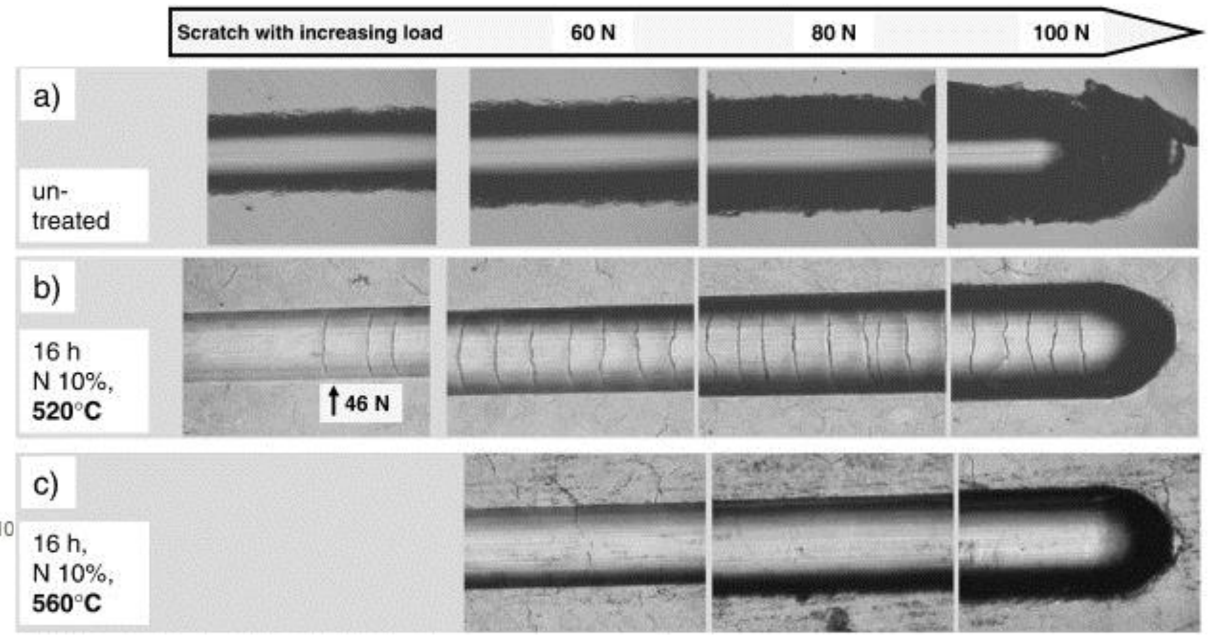
Agenda

- Motivation of micro scratch tests
- Determination of real tip geometry
- Analysis of repeated scratch tests at same position with increasing load
  - Detection of elastic – plastic transition (first failure)
  - Detection of severe fracture
- Stress analysis
- Conclusions

It depends on the sharpness of the counterpart if coatings can be damaged or not.

This shall be analyzed in laboratory with scratch tests.





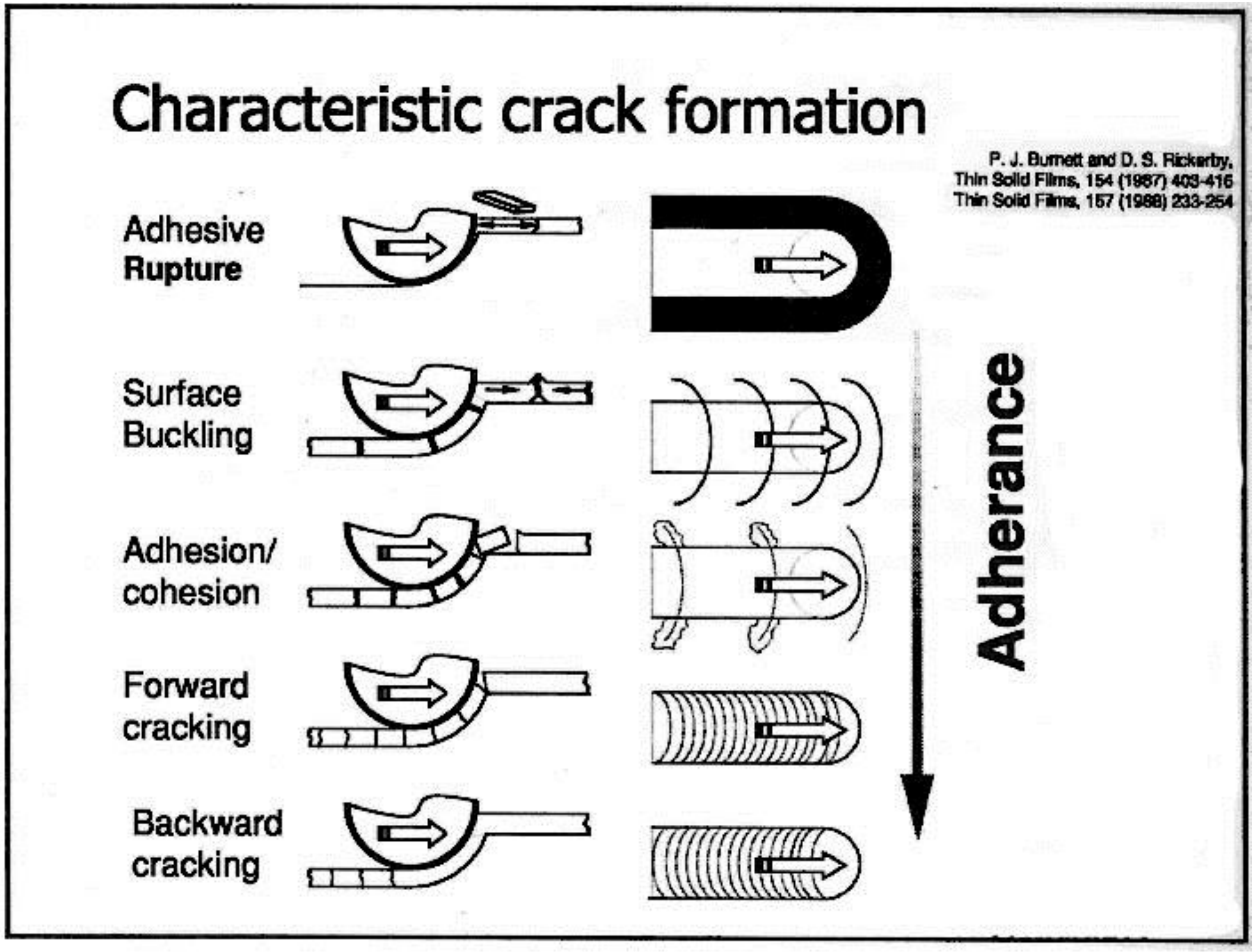
Scratch test with common Rockwell-C indenter, linearly increasing load 0–100 N on soft ASA polymer substrate and thin hard layers (a) 0.52µm CrN and (b) 1.51µm TiN.

T. Sander, S. Tremmel, S. Wartzack
Surf. Coat. Techn. 206 (2011) 1873-1878

Scratches evaluated in scratch test (CSM Revetest-RST) showing the influence of the nitriding temperature (520 °C (b) and 560 °C (c) respectively) on the crack behavior depending on the generated surface hardness. (a) shows scratch pattern on the untreated reference.

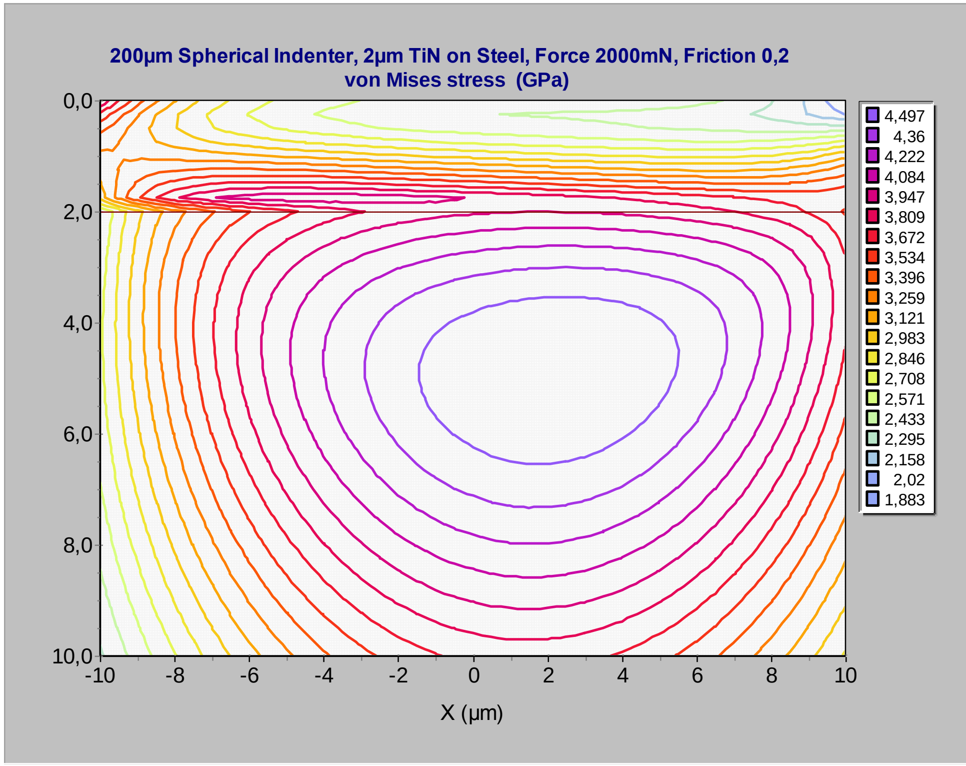
H. Paschke, M.Weber, P.Kaestner, G.Braeuer
Surf. Coat. Techn. 205 (2010) 1465-1469

Conventional scratch tests produce coating failures but one can not understand why the failures occur

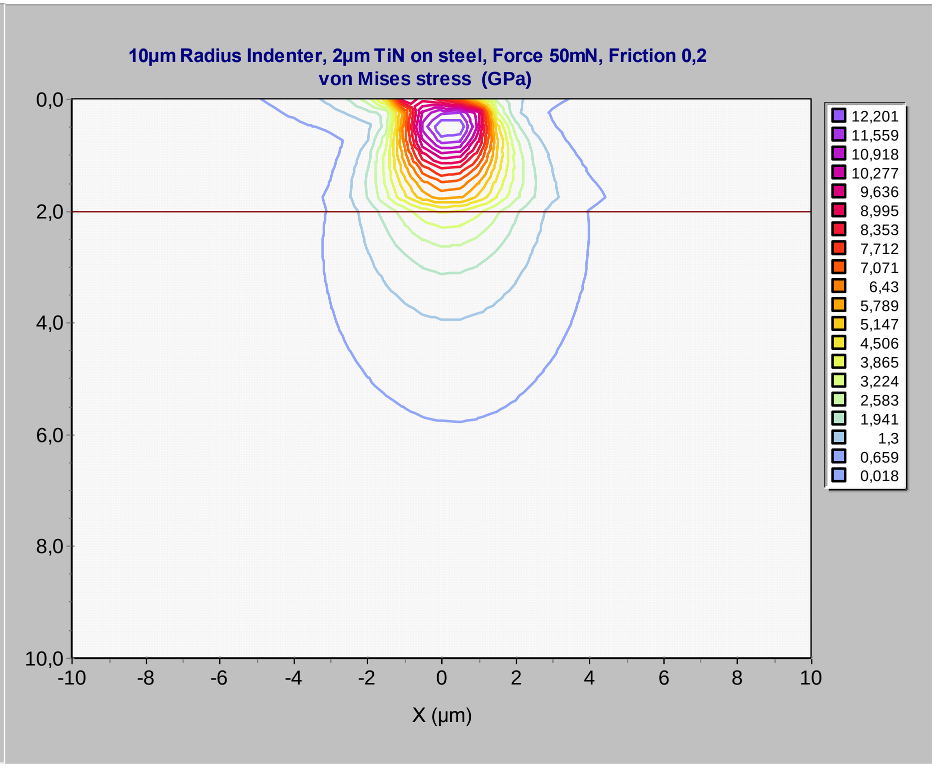


Failure modes for a standard scratch test with Rockwell C indenter

Von Mises stress field in the surface of a 2µm thick hard coating on steel for a scratch test with tips of different radius.



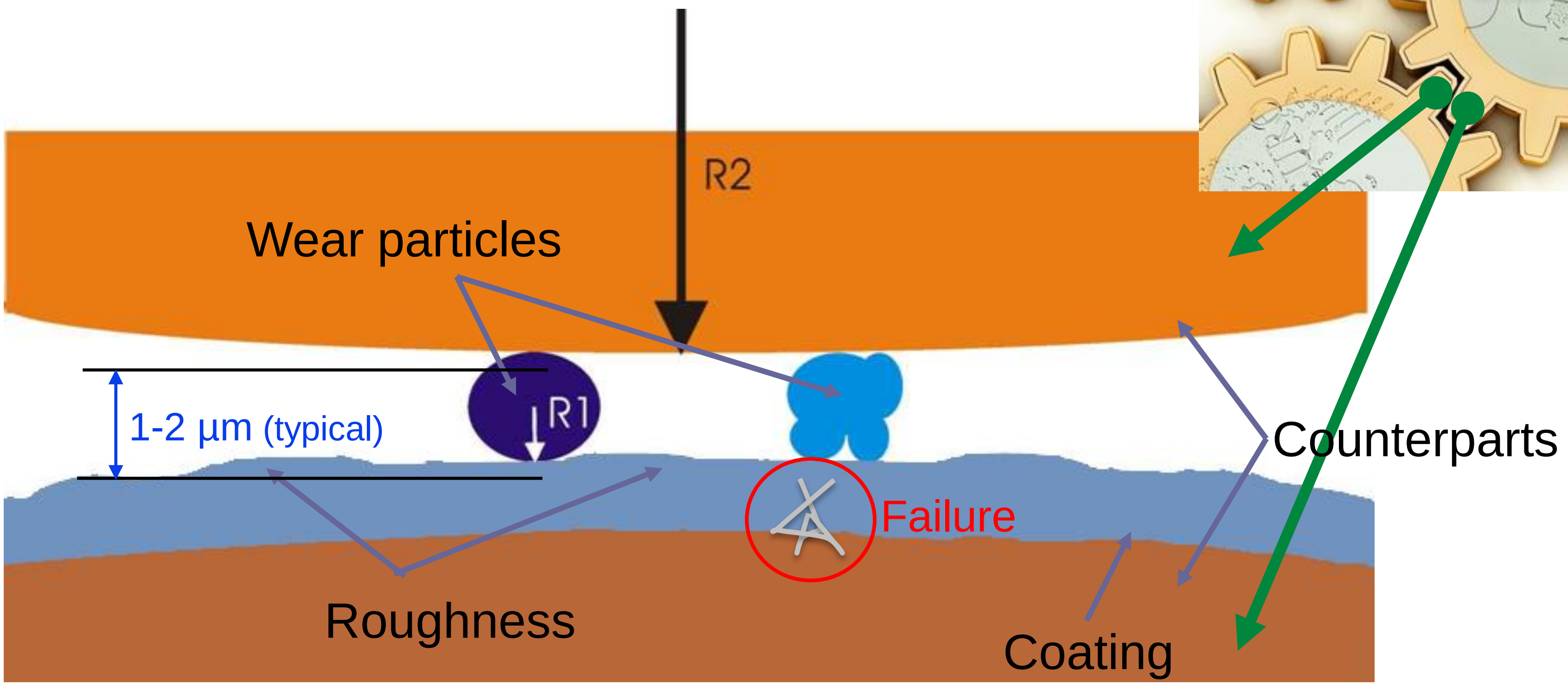
200µm radius



10µm radius

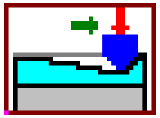
The first failure of a conventional scratch test occurs normally in the substrate.

Analysis of characteristic loading conditions



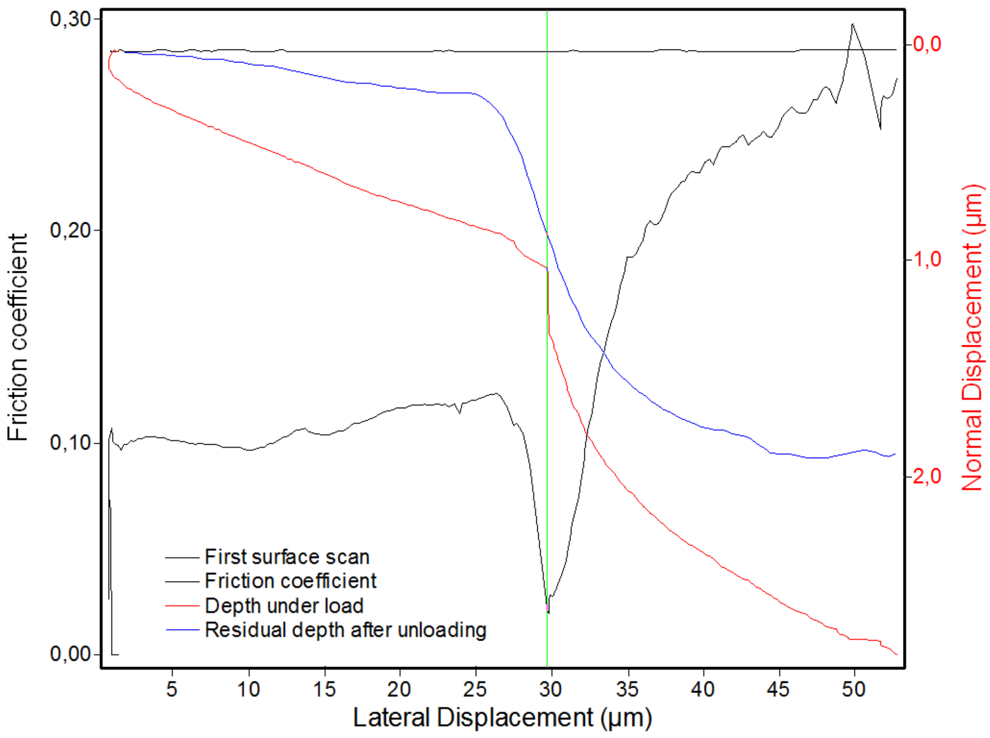
To understand the failure reasons one has to go down to the dimension of roughness and wear particles → This requires high resolution.

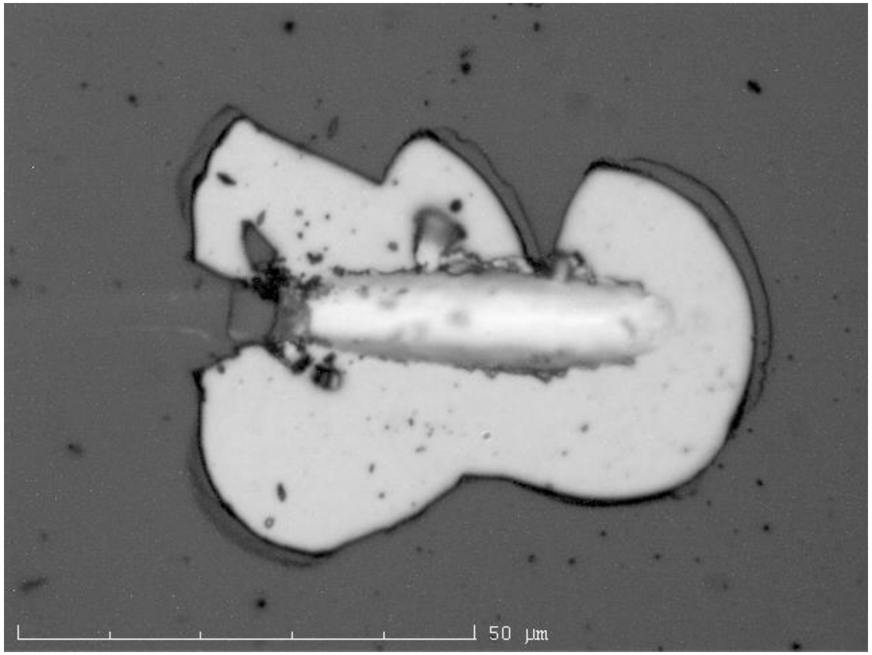
Micro scratch test



Parameter (typical)	Micro scratch test	Conventional scratch test
Indenter radius	2 – 20 µm	200 µm (Rockwell)
Normal force	0,01 – 2 N	1 – 100 N
Scratch length	10 - 500 µm	5000 µm
Scratch speed	1-20 µm/s	300 µm/s
Normal displacement	Available	Not available
Stress maximum	In coating / Interface	In substrate
Roughness influence	Low	High
Tip wear	Medium	High

≈ 2µm DLC on steel, tip radius 7.2µm





Example for bad adhesion

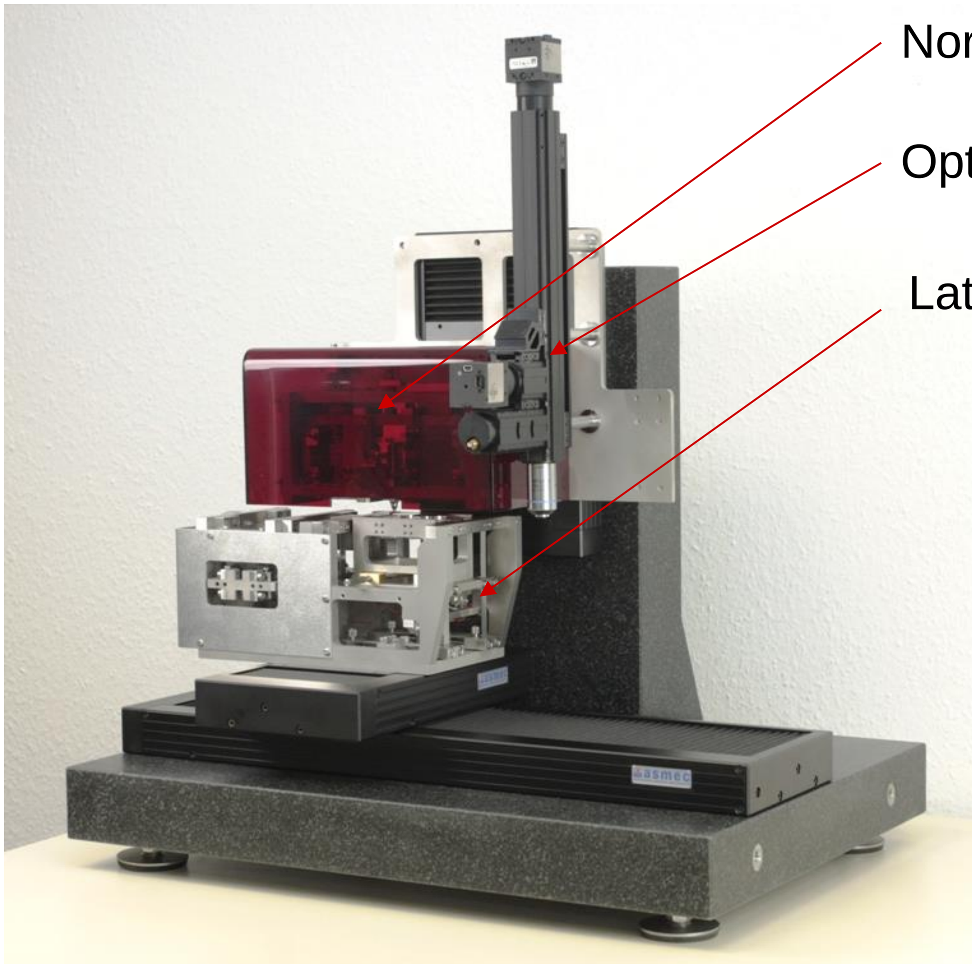
The delamination can be seen in the curve by a step in the displacement curve and a minimum in the friction curve

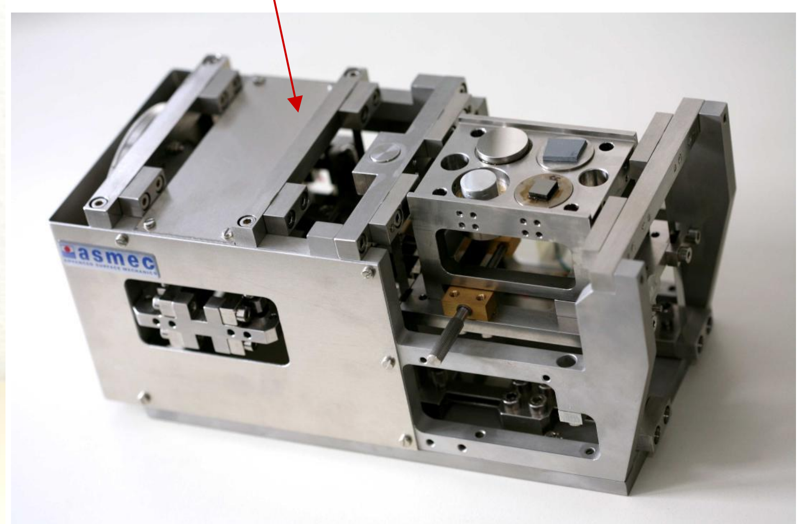
UNAT – Universal Nanomechanical Tester

Normal force unit NFU

Optics

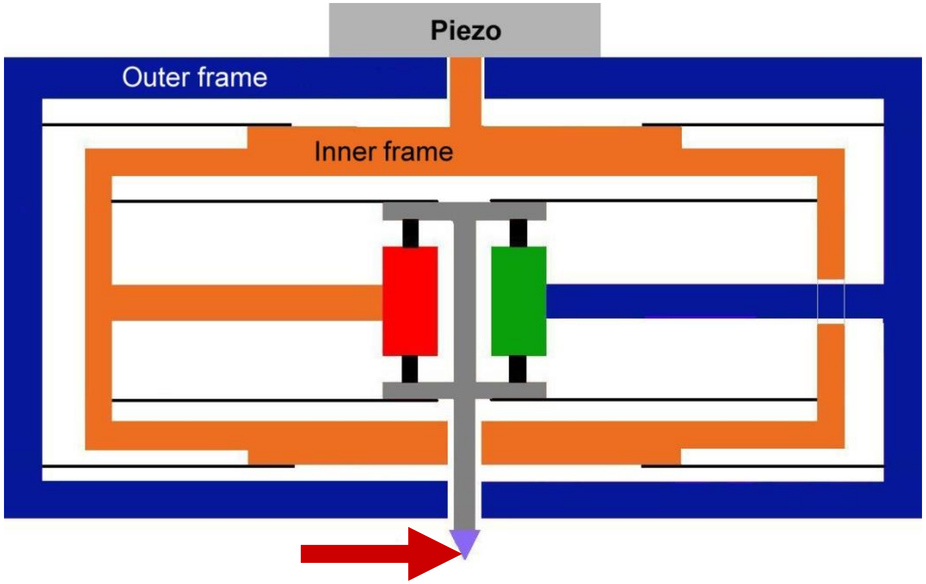
Lateral force unit LFU



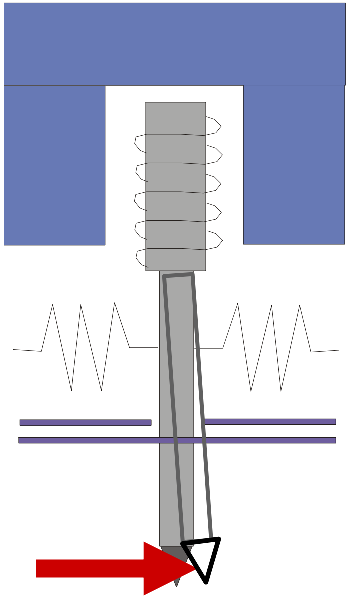


Nanoindenter heads

UNAT principle



Other principles



High lateral stiffness in scratch direction

Shaft bending in nm range is determined and corrected in the data

Low lateral stiffness in scratch direction

Shaft bending in µm range prevent correct determination if tip position on surface

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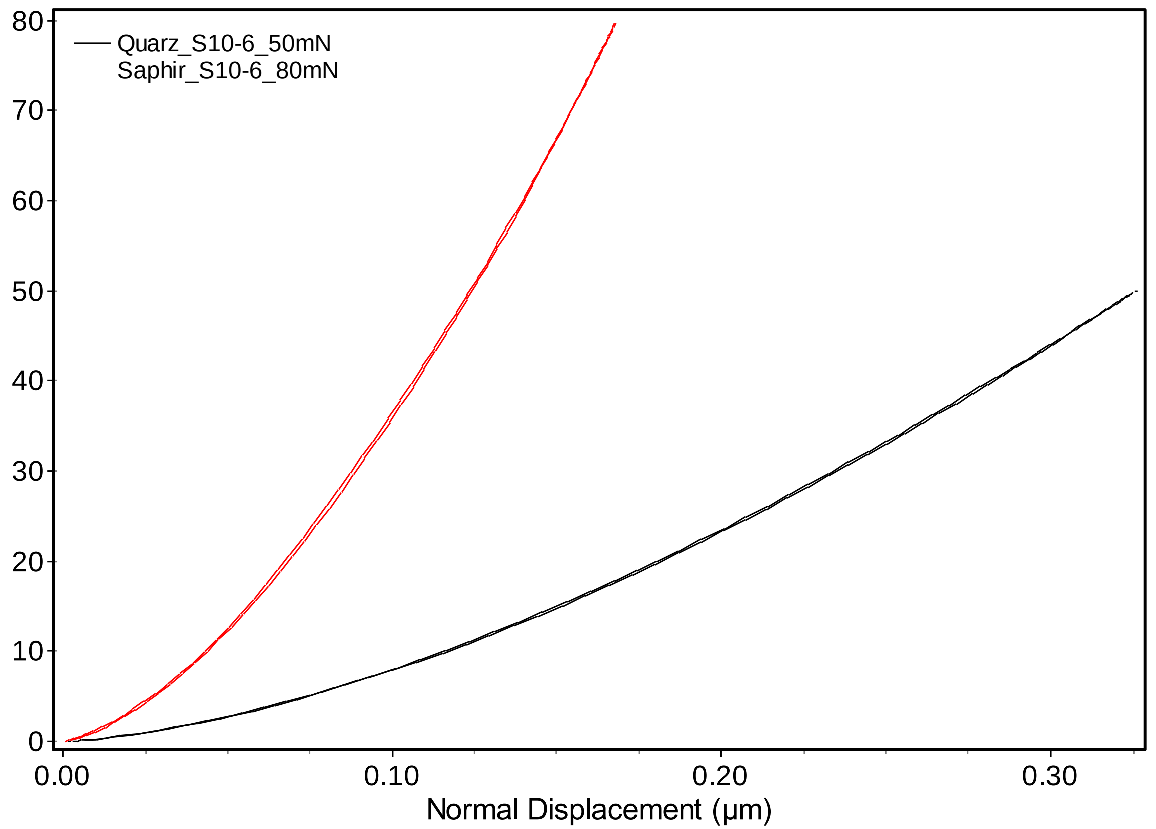
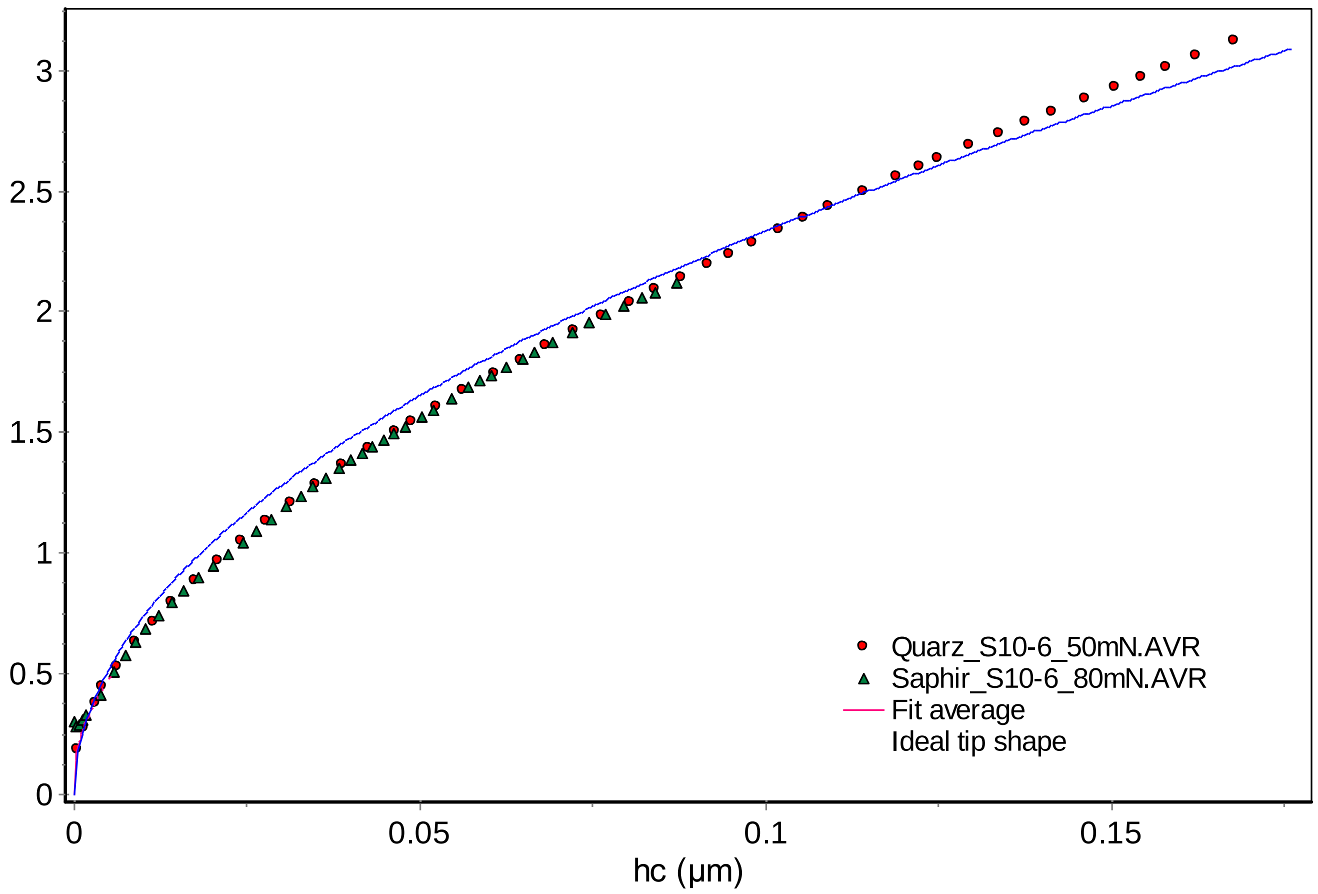
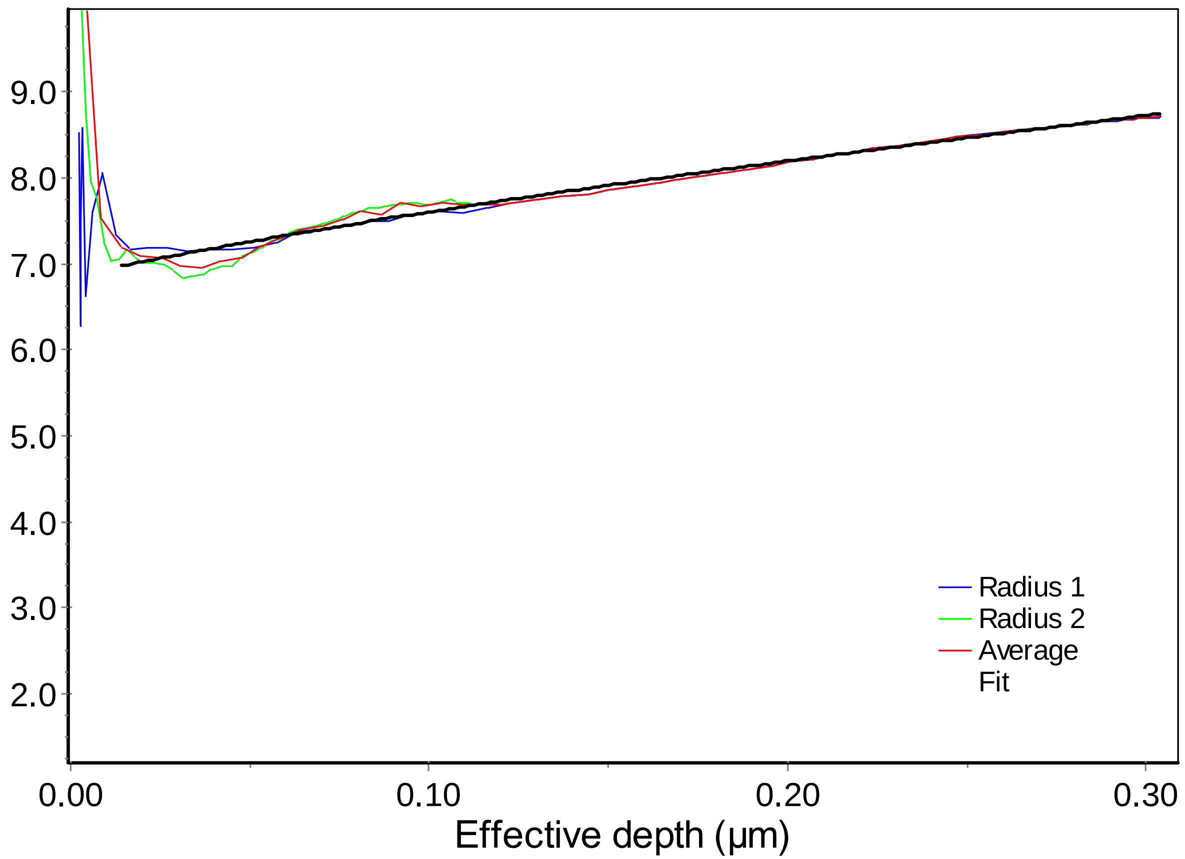
Determination of the real tip geometry by using fully elastic indentations into two different reference materials



Fully elastic load-displacement curves, measured on fused silica + sapphire



Area function of indenter S10-6, nominal radius 10µm, obtained from elastic measurements on fused silica + sapphire

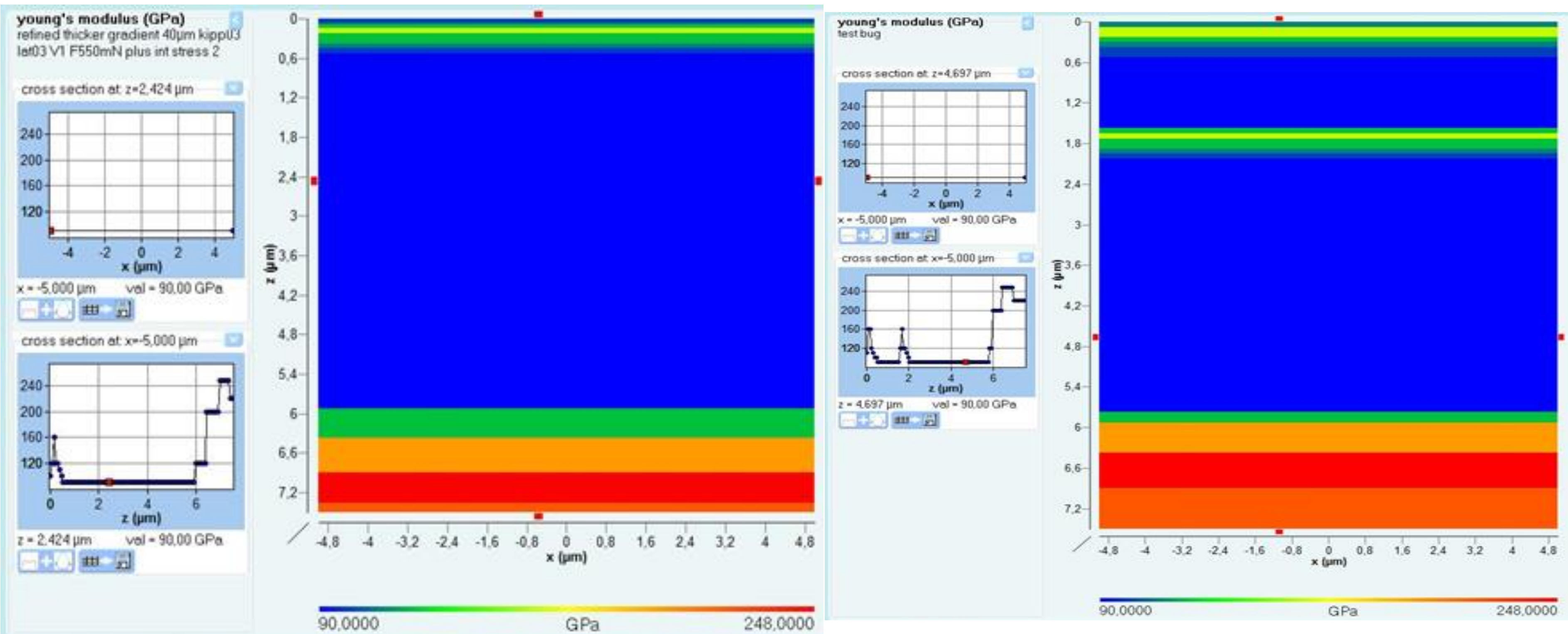


Radius function of indenter S10-6, nominal radius 10µm, obtained from elastic measurements on fused silica + sapphire

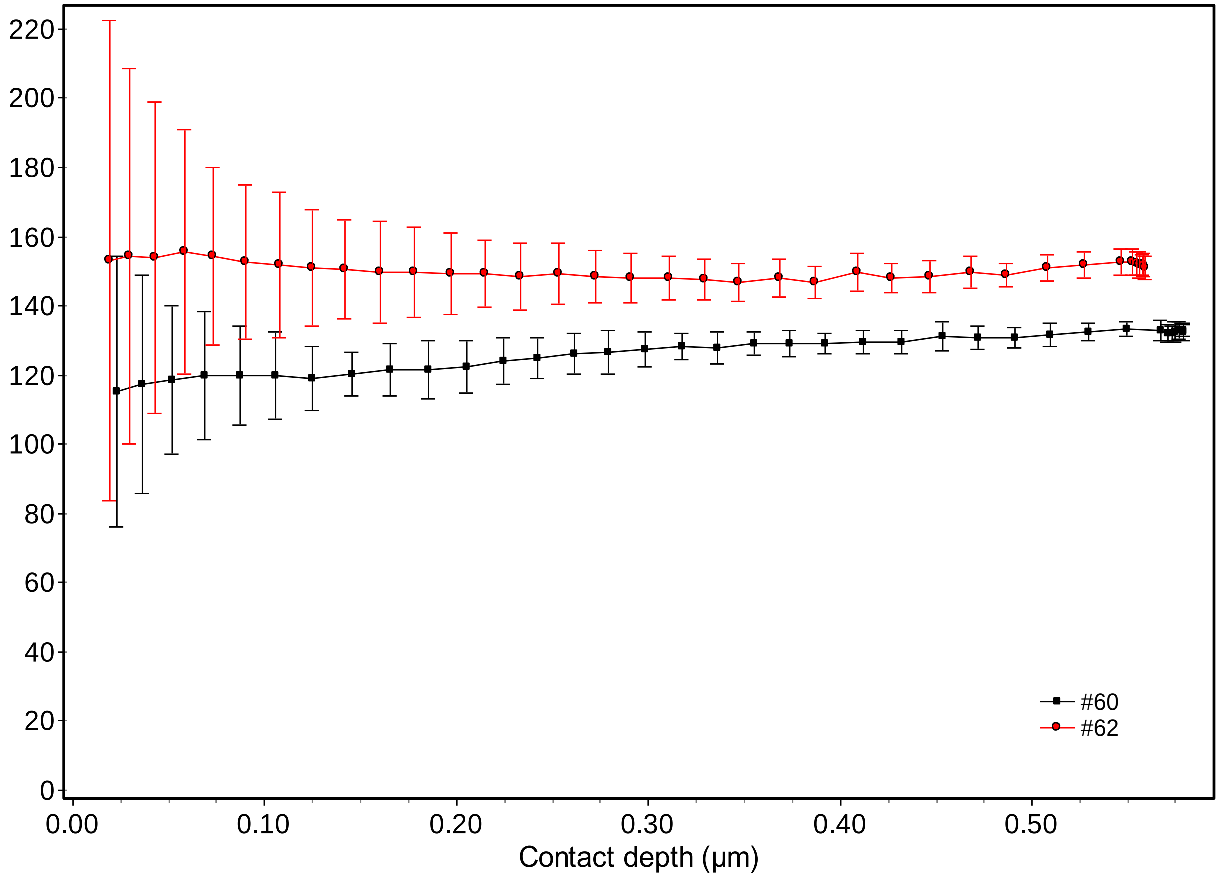
The effective radius at the outermost tip is only 7µm

Investigation of two samples with **a:C:H:W** coatings (DLC)

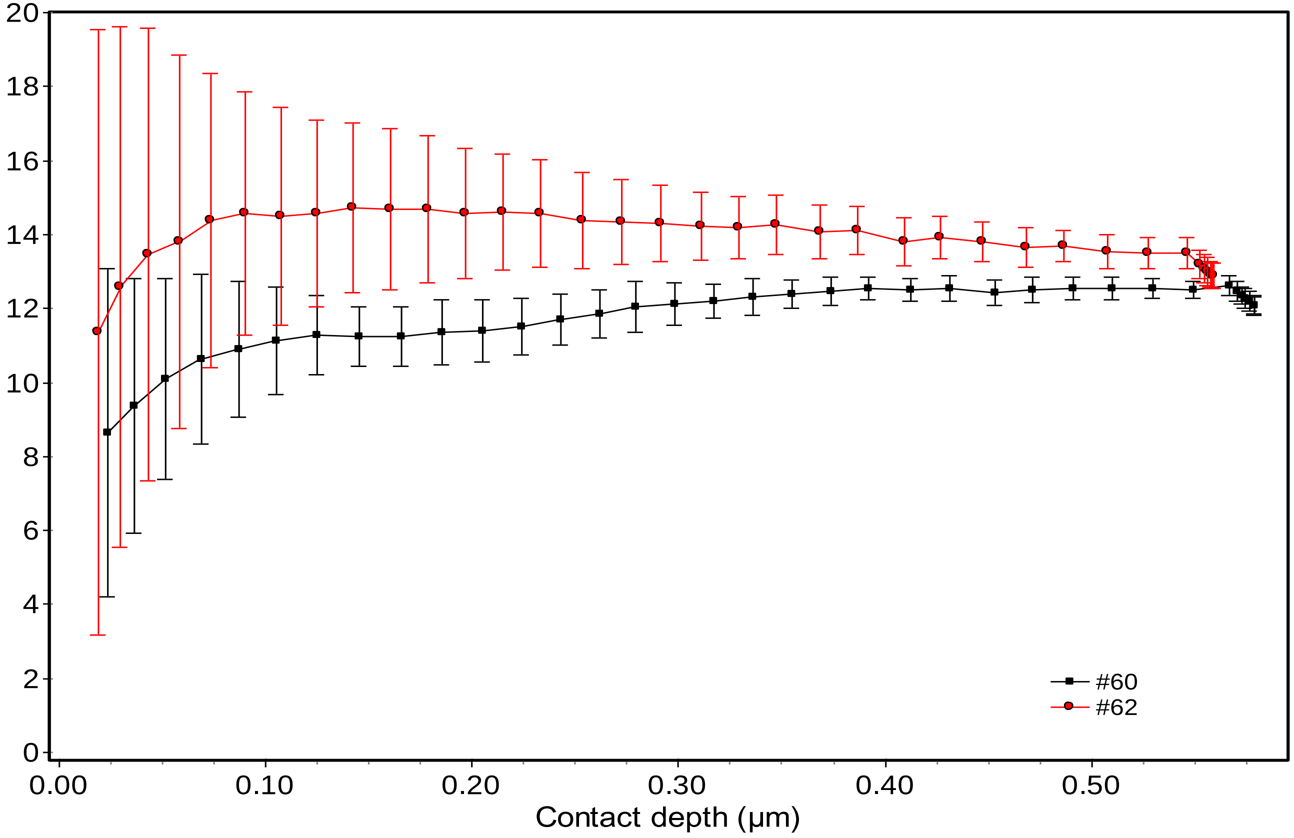
Sample #60 = gradient coating thickness 6.5µm
Sample #62 = homogeneous coating thickness 7.8µm



2 examples for an optimized layer structure for a certain roughness range and loading condition



Indentation modulus as function of depth, measured with dynamic QCSM method of ASMEC



Indentation hardness as function of depth, measured with dynamic QCSM method of ASMEC

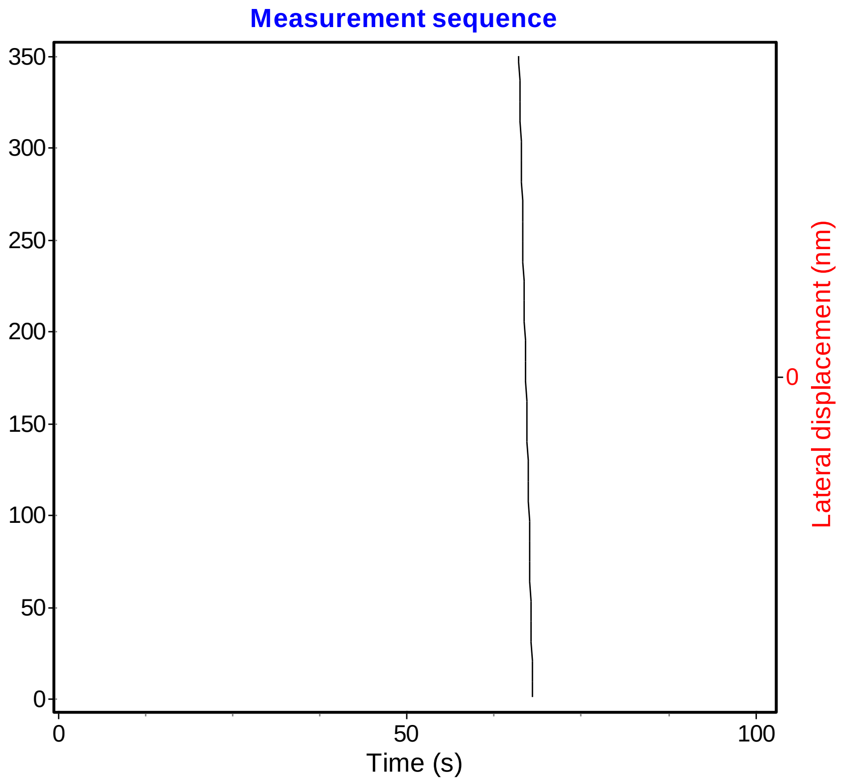
Scratch parameter:

Distance: 300µm
Speed: 10µm/s

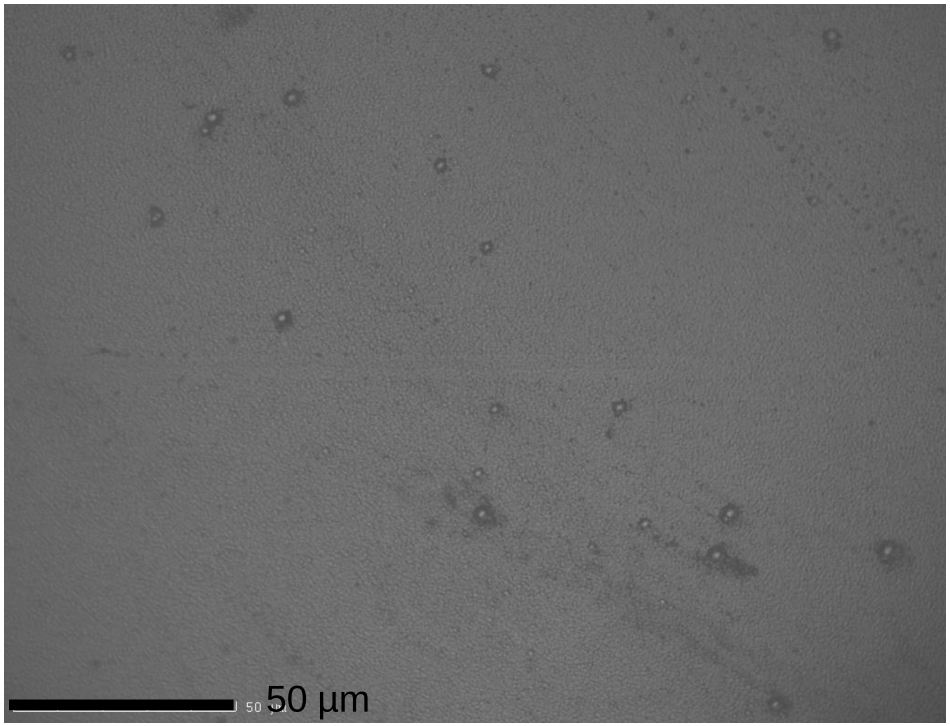
Pre and post scan with contact force of 2.8mN

Increasing force from contact force to maximum

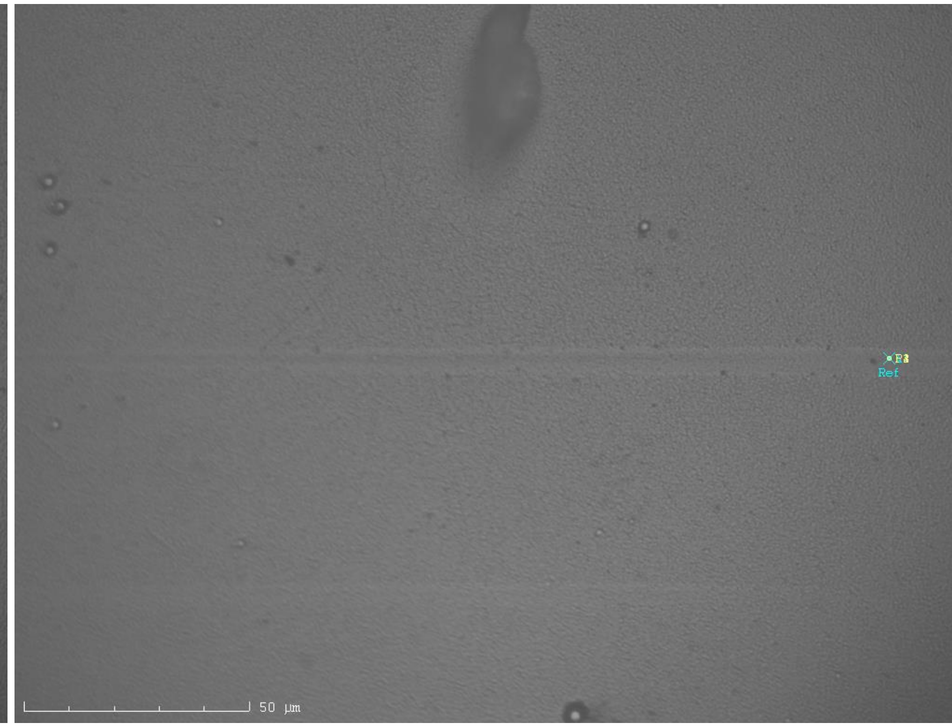
3 test at same position with 350mN
3 test at same position with 800mN
2 test at same position with 1500mN



Sample surface after three 350mN tests

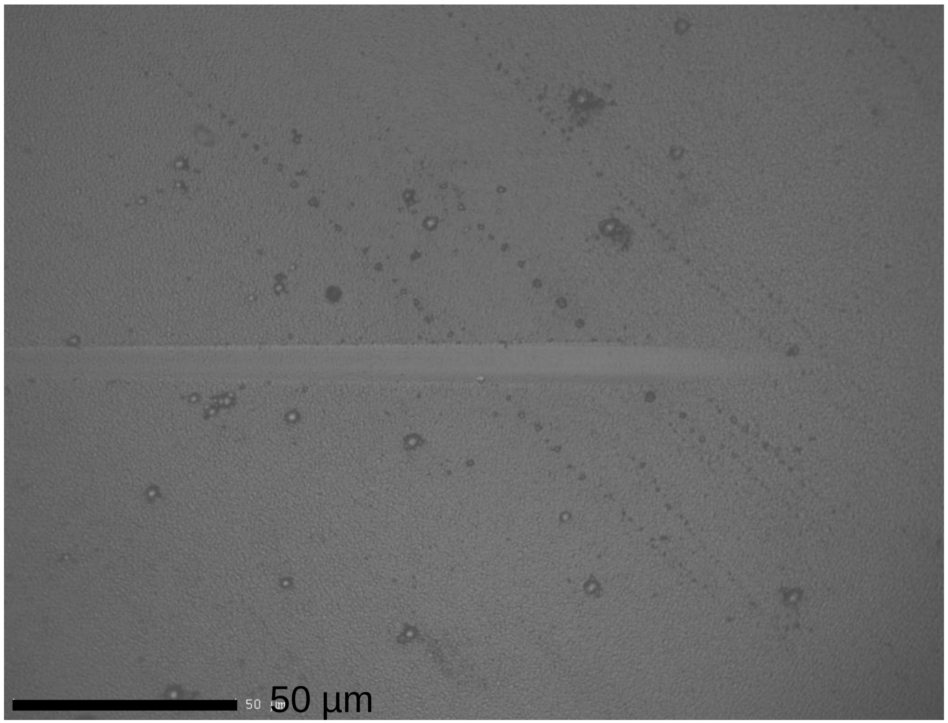


Sample #60

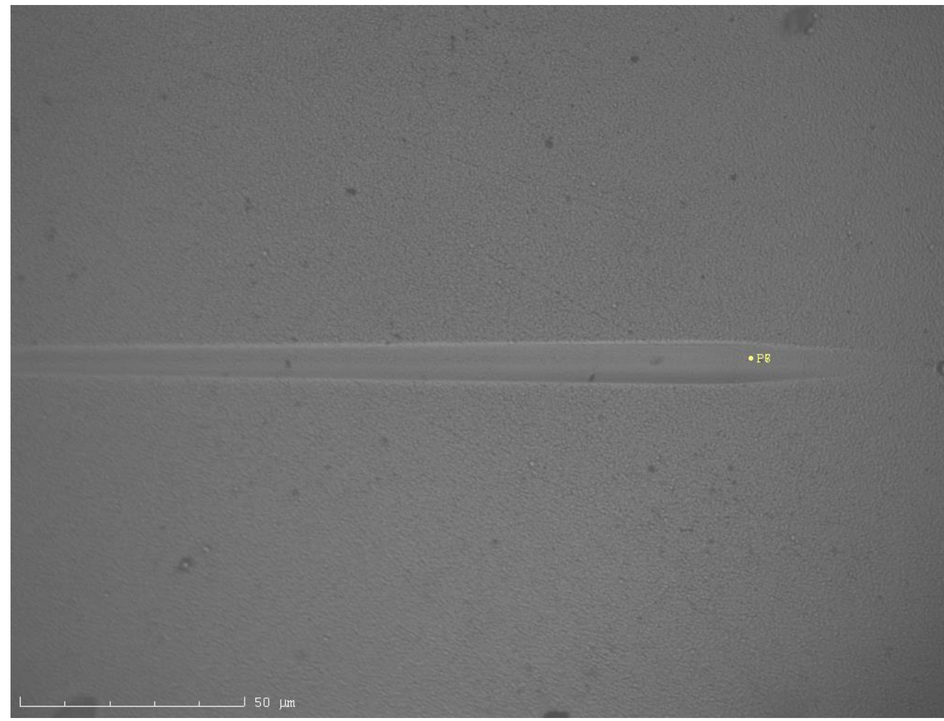


Sample #62

Sample surface after three 800mN tests

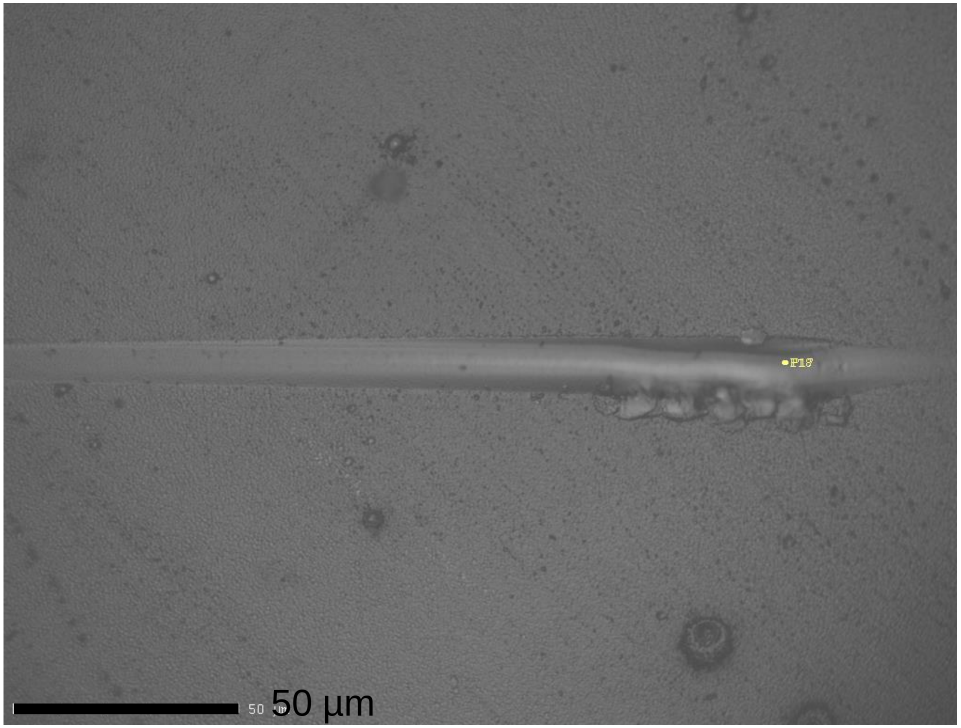


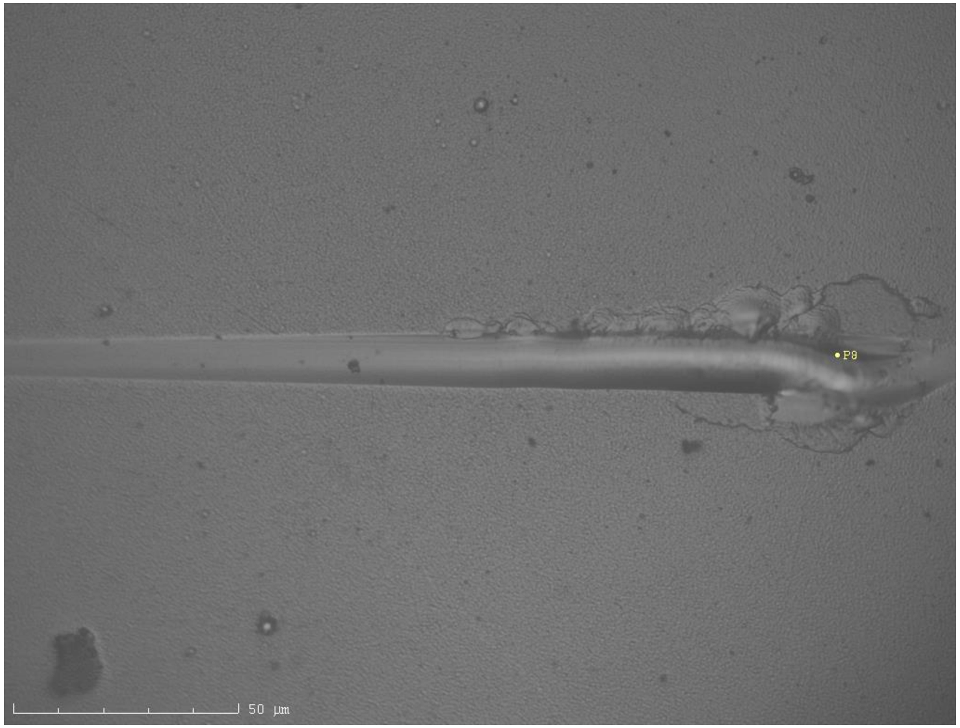
Sample #60



Sample #62

Sample surface after two 1500mN tests

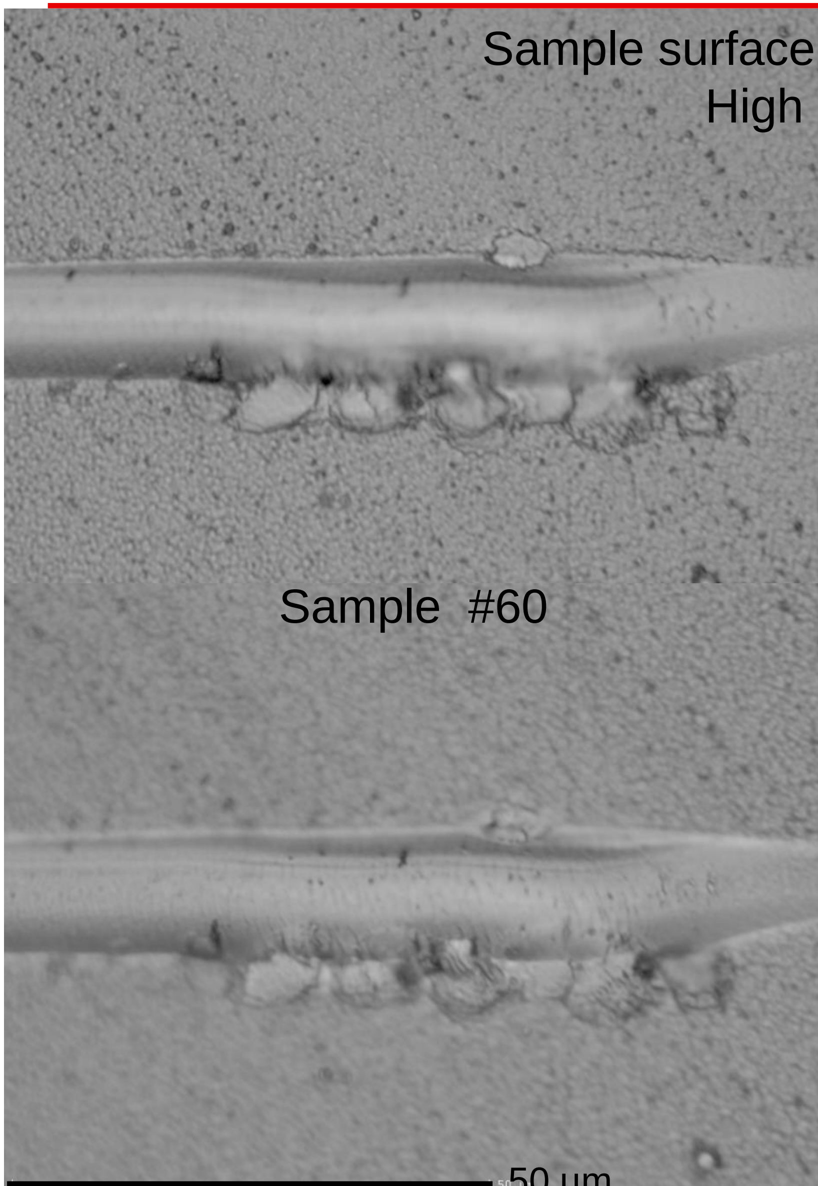




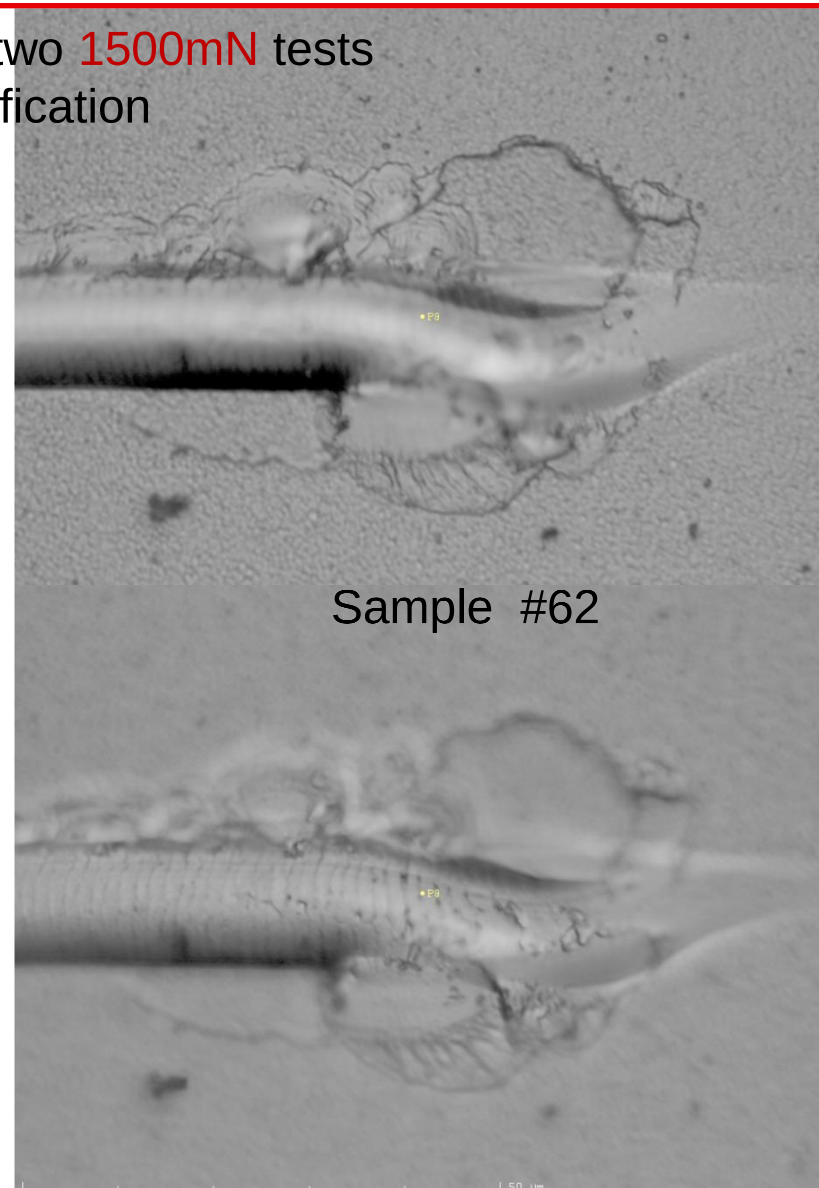
Sample #60

Sample #62

Sample surface after two 1500mN tests
High magnification



Sample #60

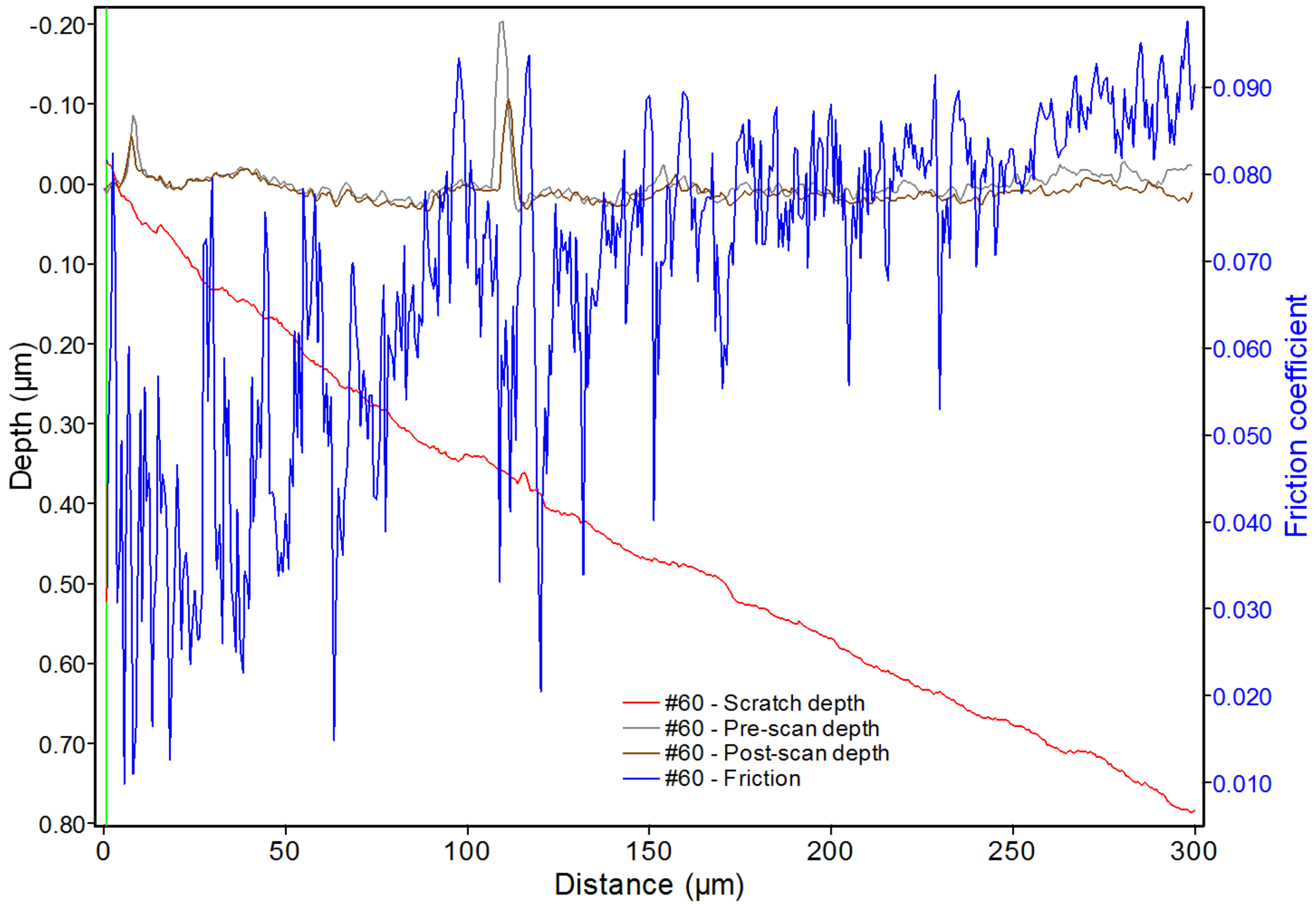


Sample #62

Test 1

350mN

#60

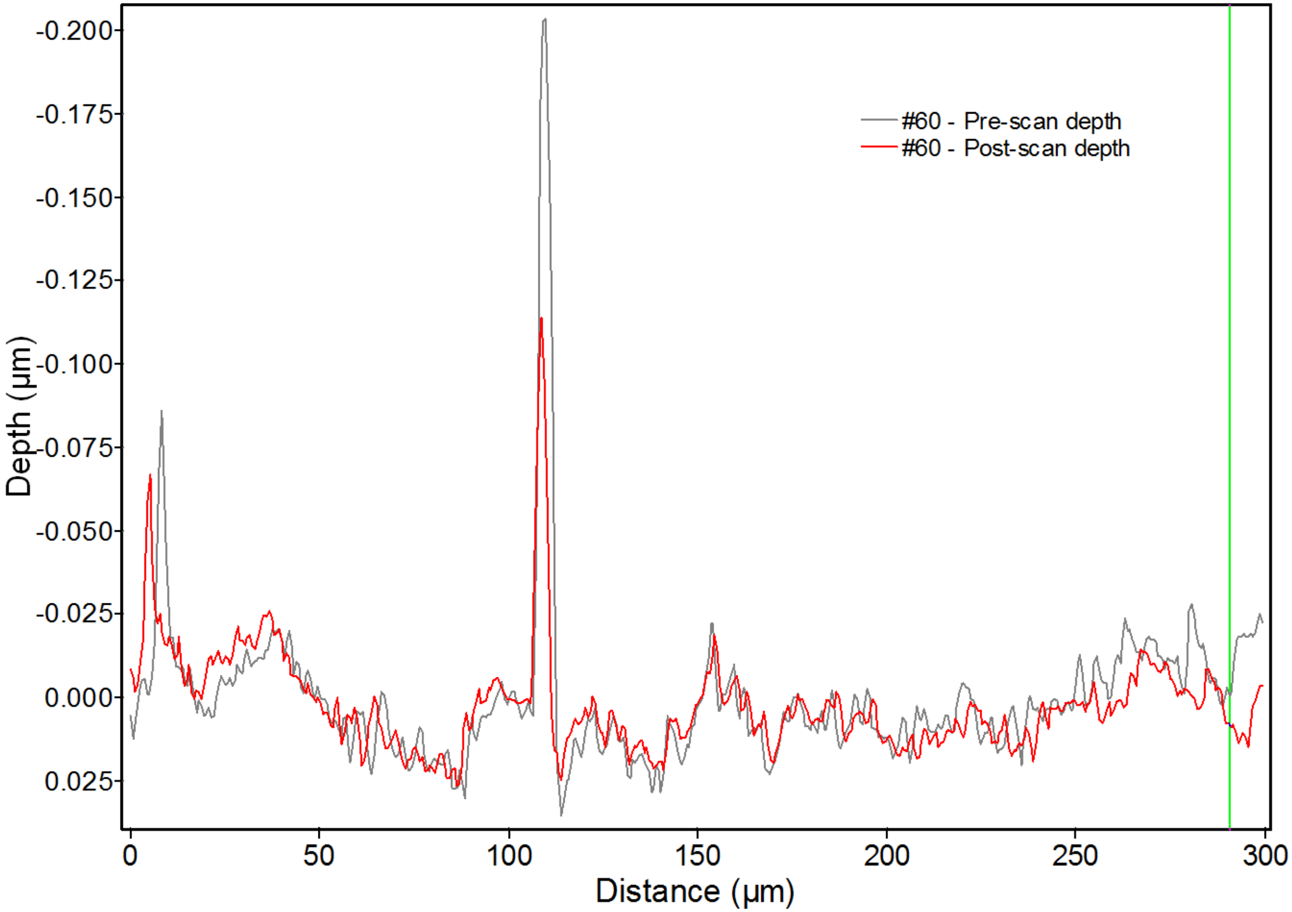


Sample #60 350mN

Test 1

350mN

#60



Comparison of surface position before and after test

Peak height is reduced

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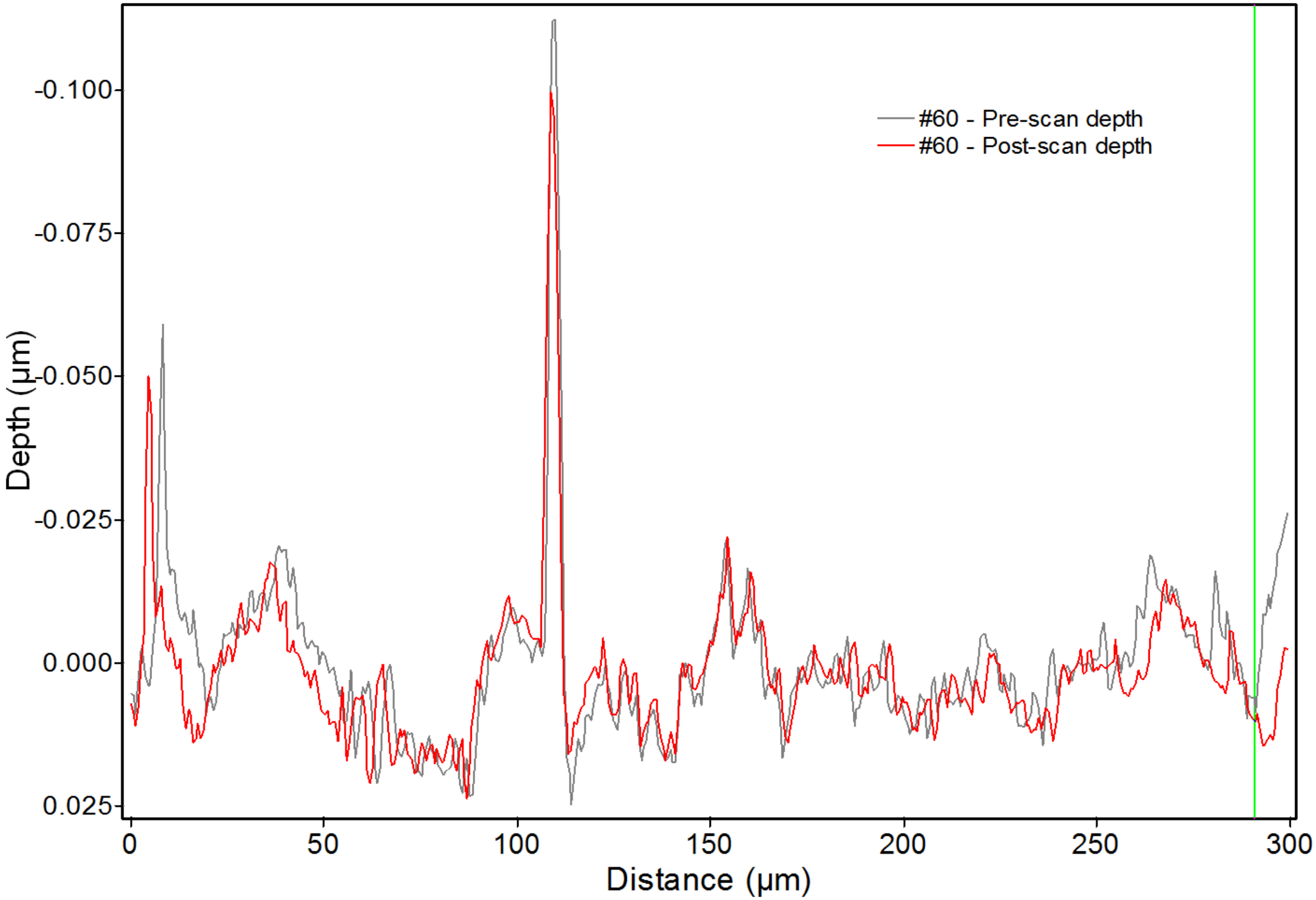
Test 2

350mN

#60



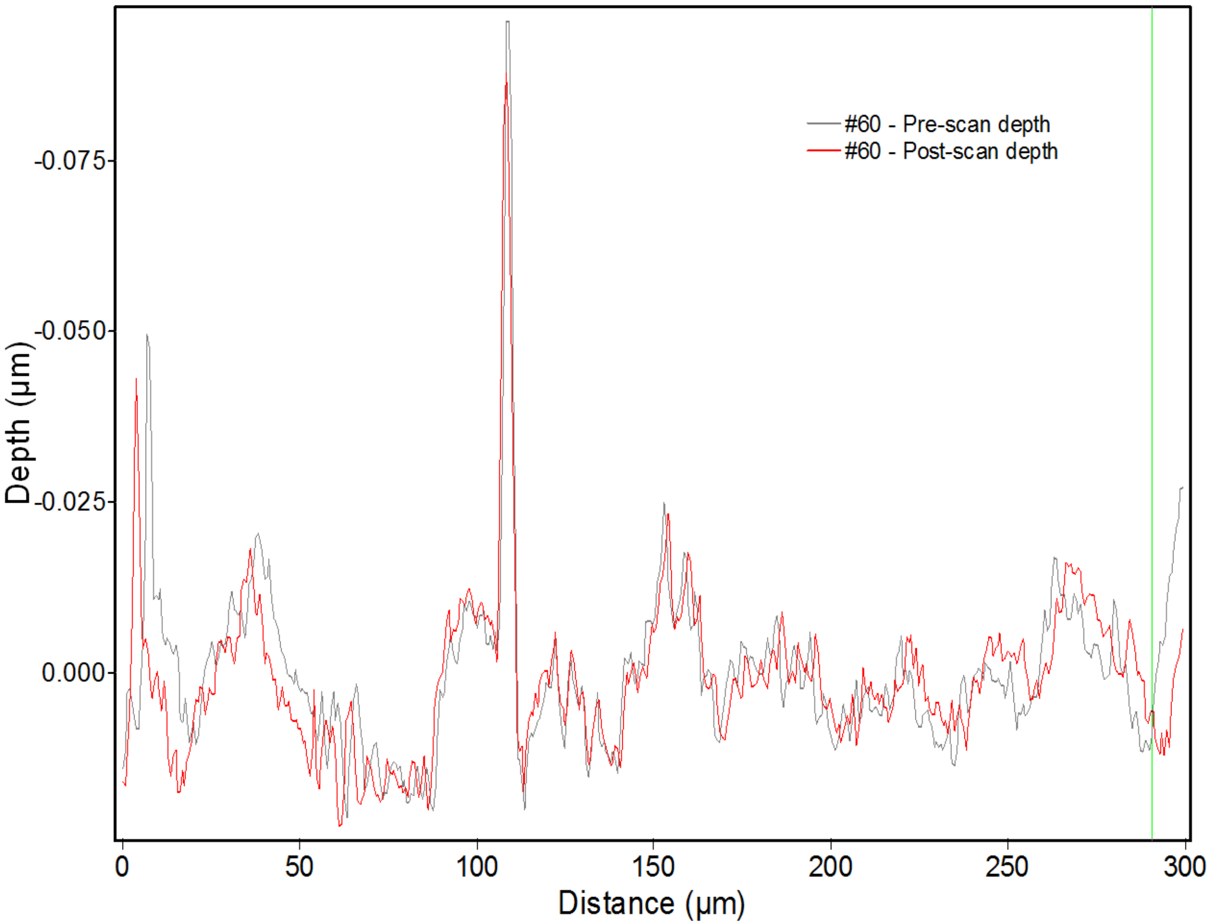
Comparison of surface position before and after test

Peak height is further reduced

Test 3

350mN

#60



Comparison of surface position before and after test

Peak height is further reduced

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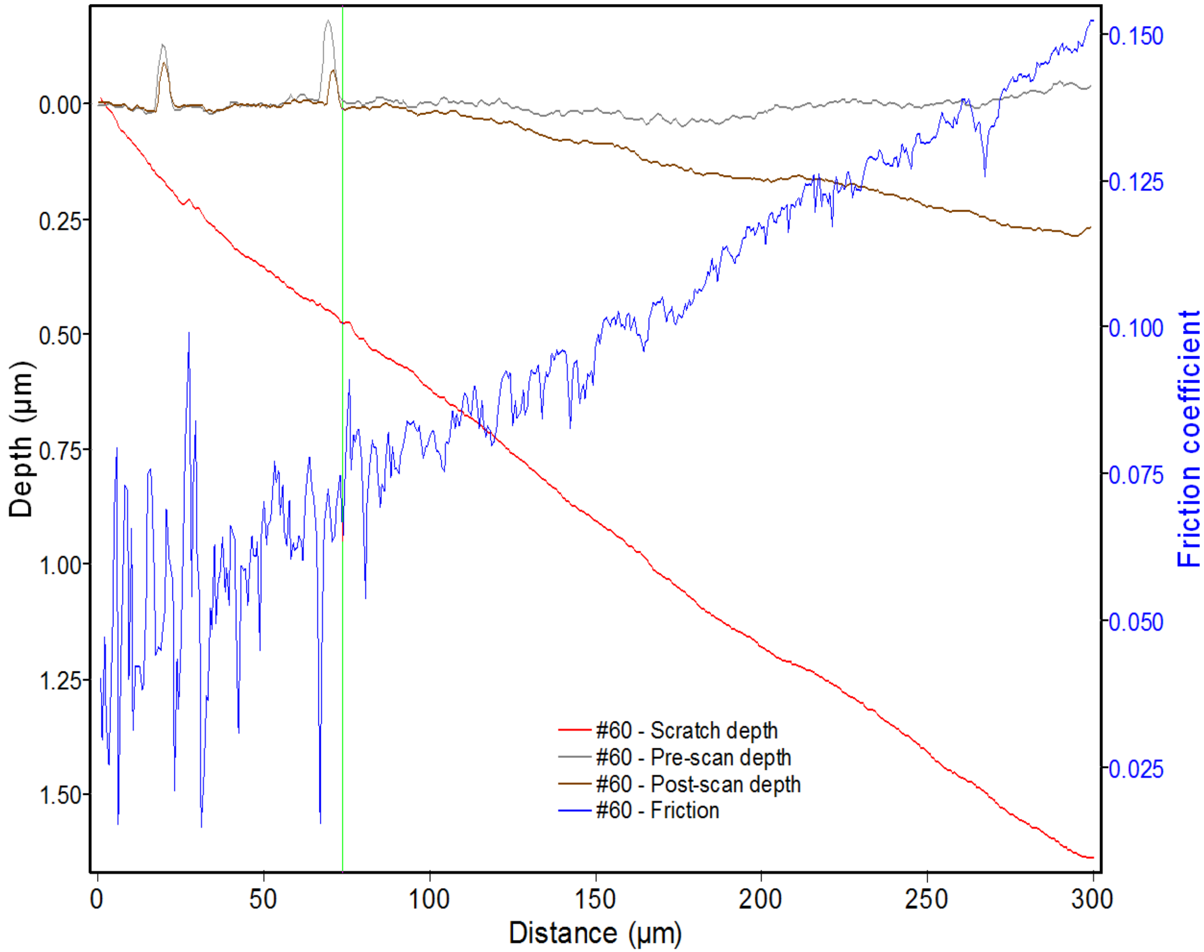
Test 1

800mN

#60

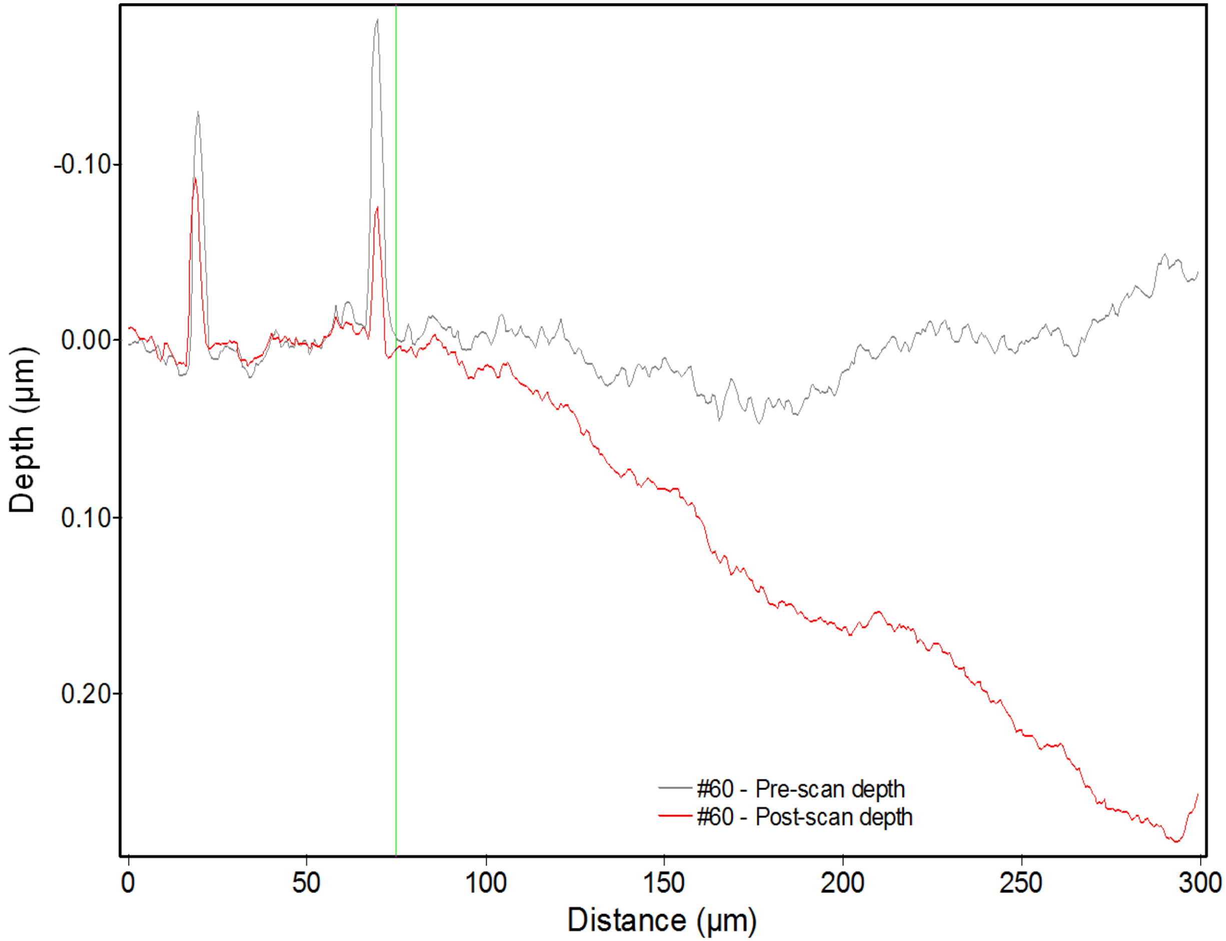


Plastic deformation is clearly visible

Test 1

800mN

#60



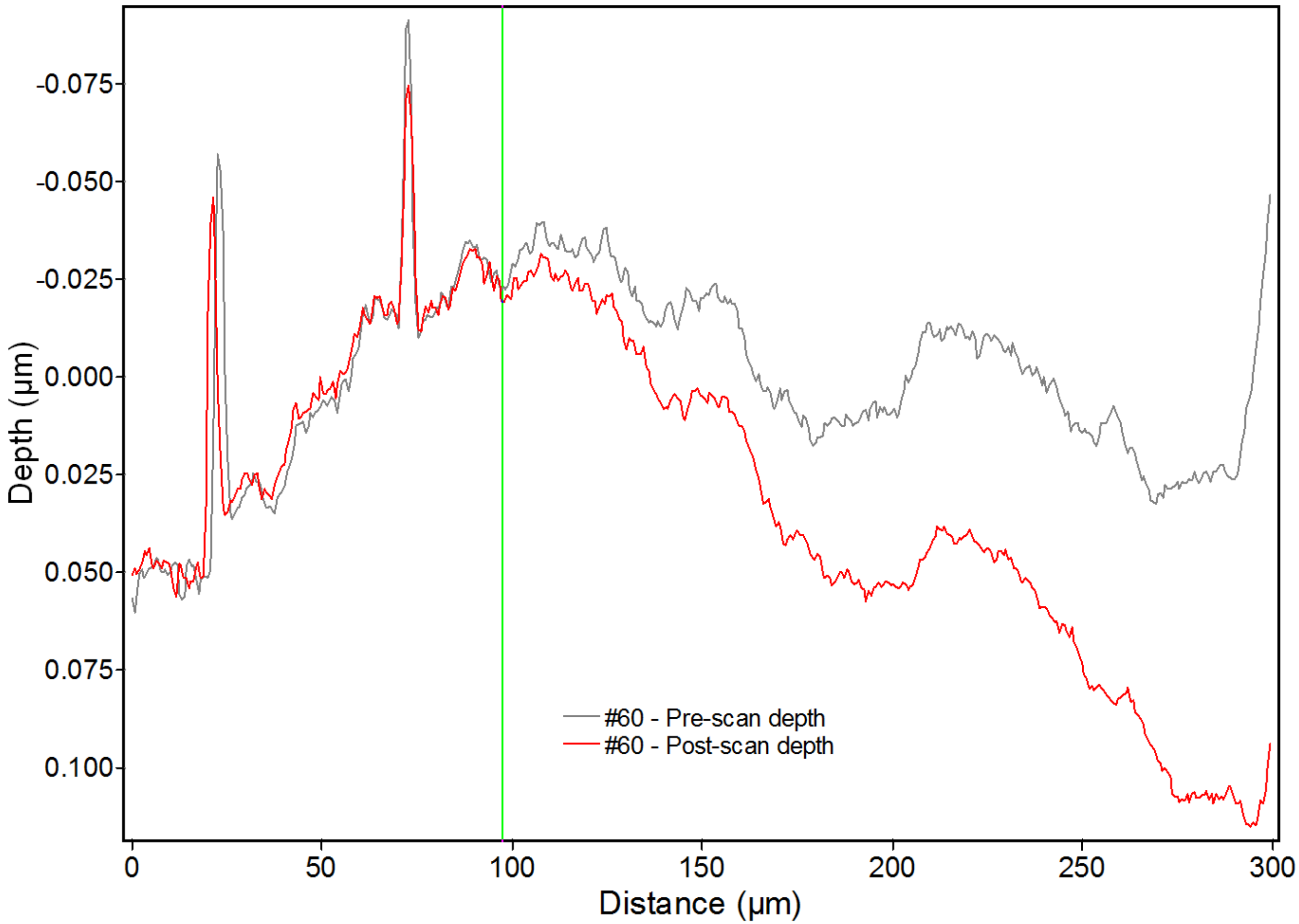
Comparison of surface position before and after test

Plastic deformation starts at 207mN, position 75µm

Test 2

800mN

#60



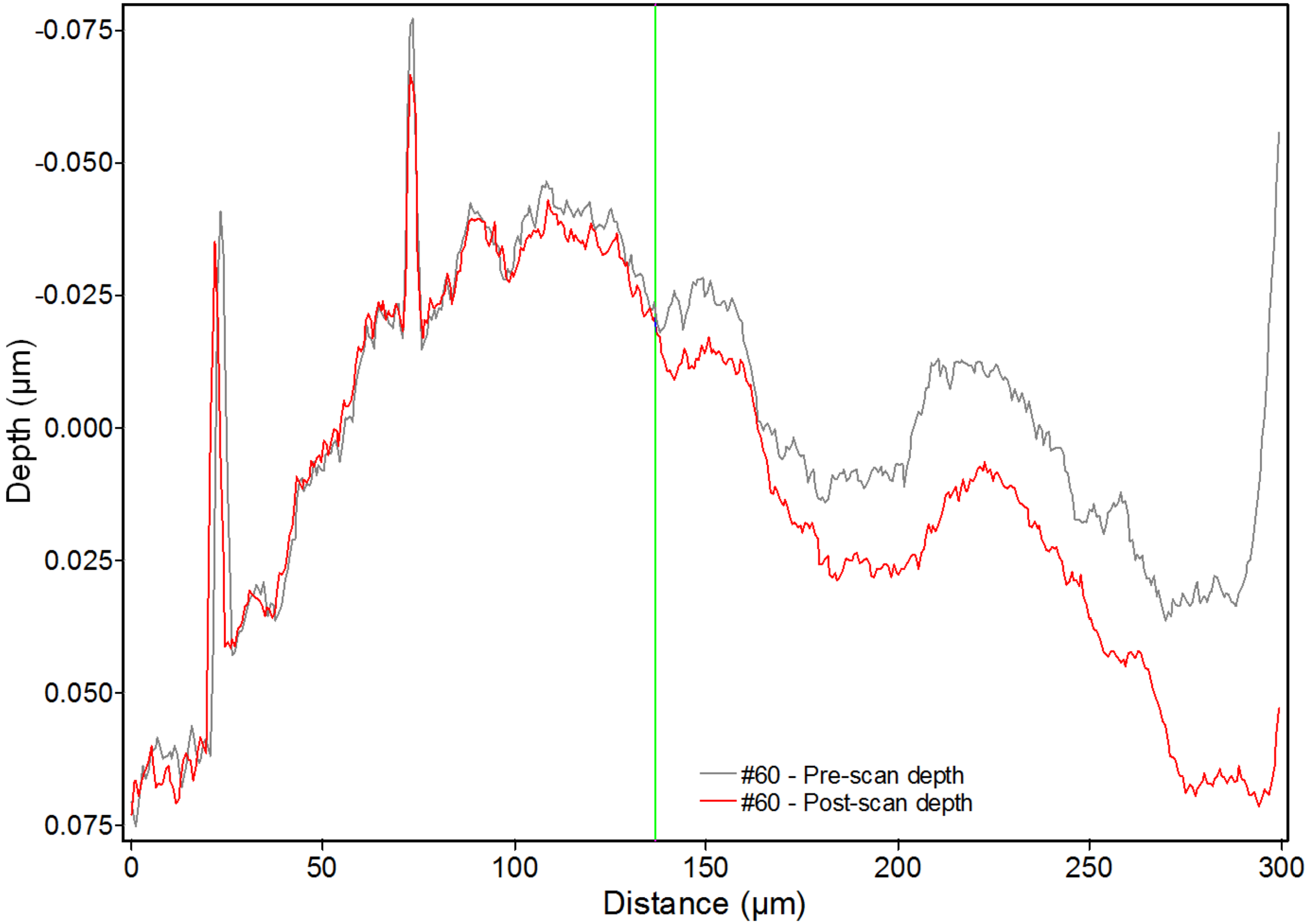
Comparison of surface position before and after test

Plastic deformation starts again at 263mN, position 97.5µm

Test 3

800mN

#60



Comparison of surface position before and after test

Plastic deformation starts again at 367mN, position 137µm

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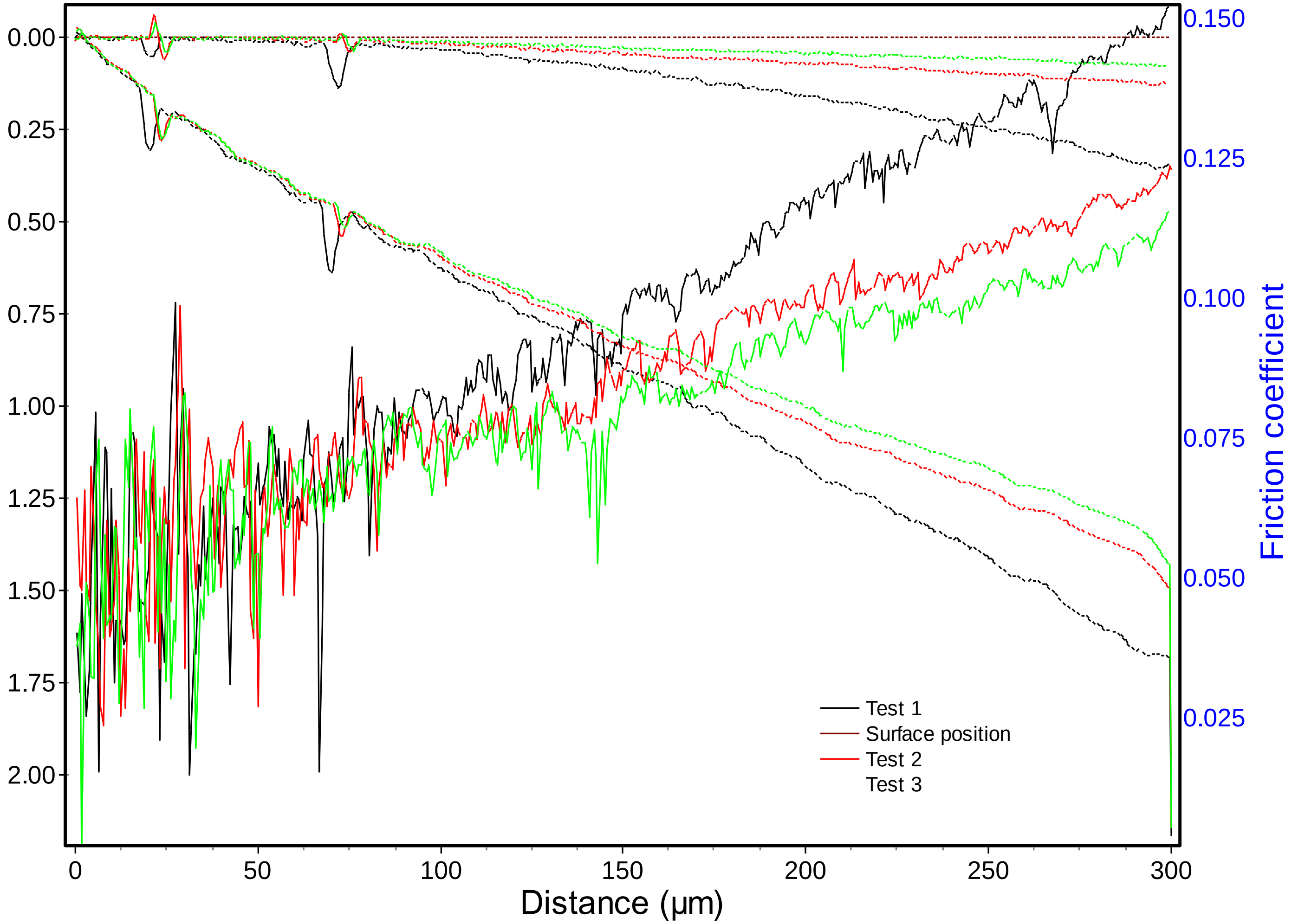

Sample #60; comparison of Test 1-3 with 800mN

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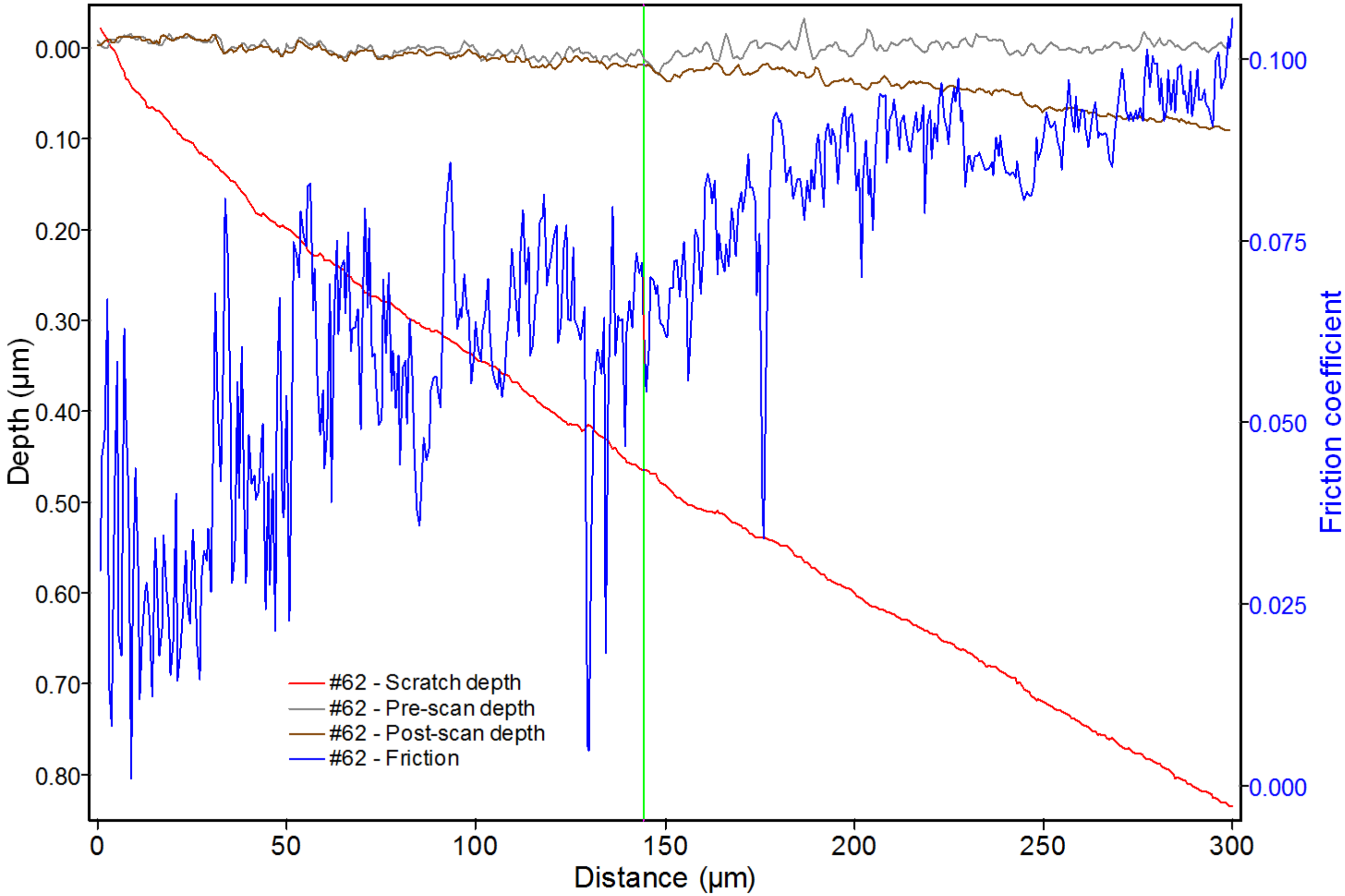
Test 1

350mN

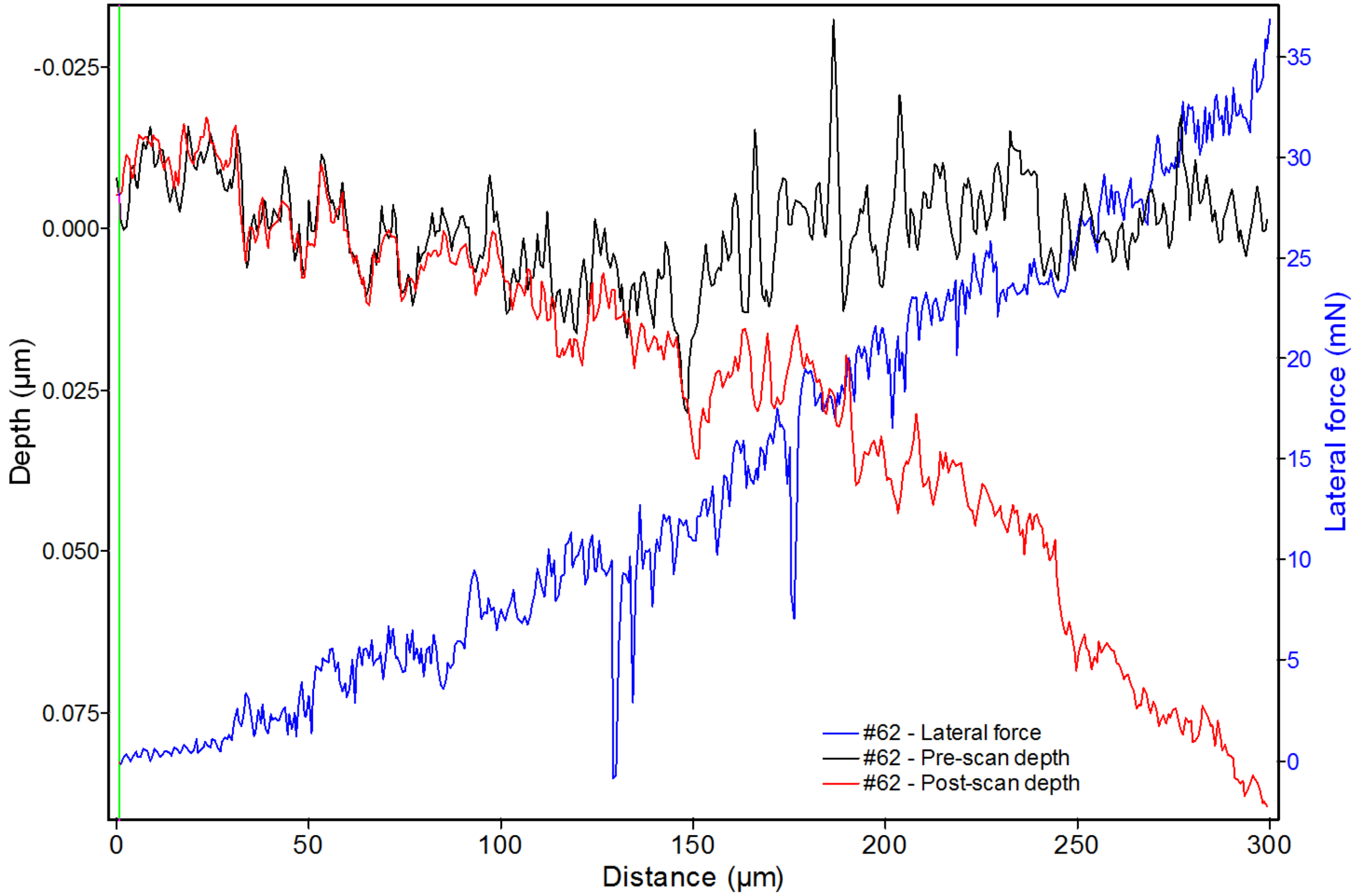
#62



Test 1

350mN

#62



Comparison of surface position before and after test

Plastic deformation starts at 173mN, position 147.5µm

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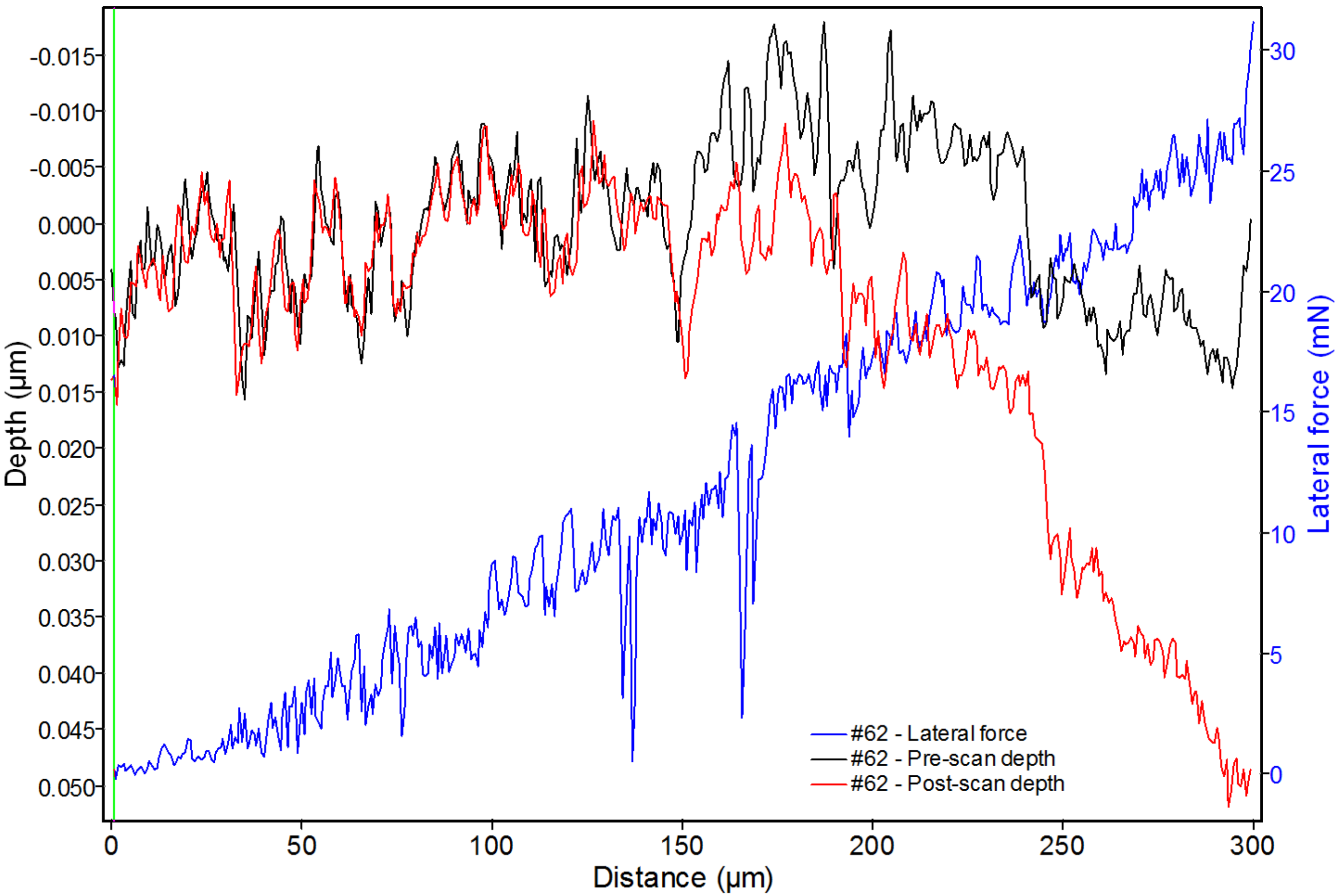
Test 2

350mN

#62



Comparison of surface position before and after test

Plastic deformation starts again at 176mN

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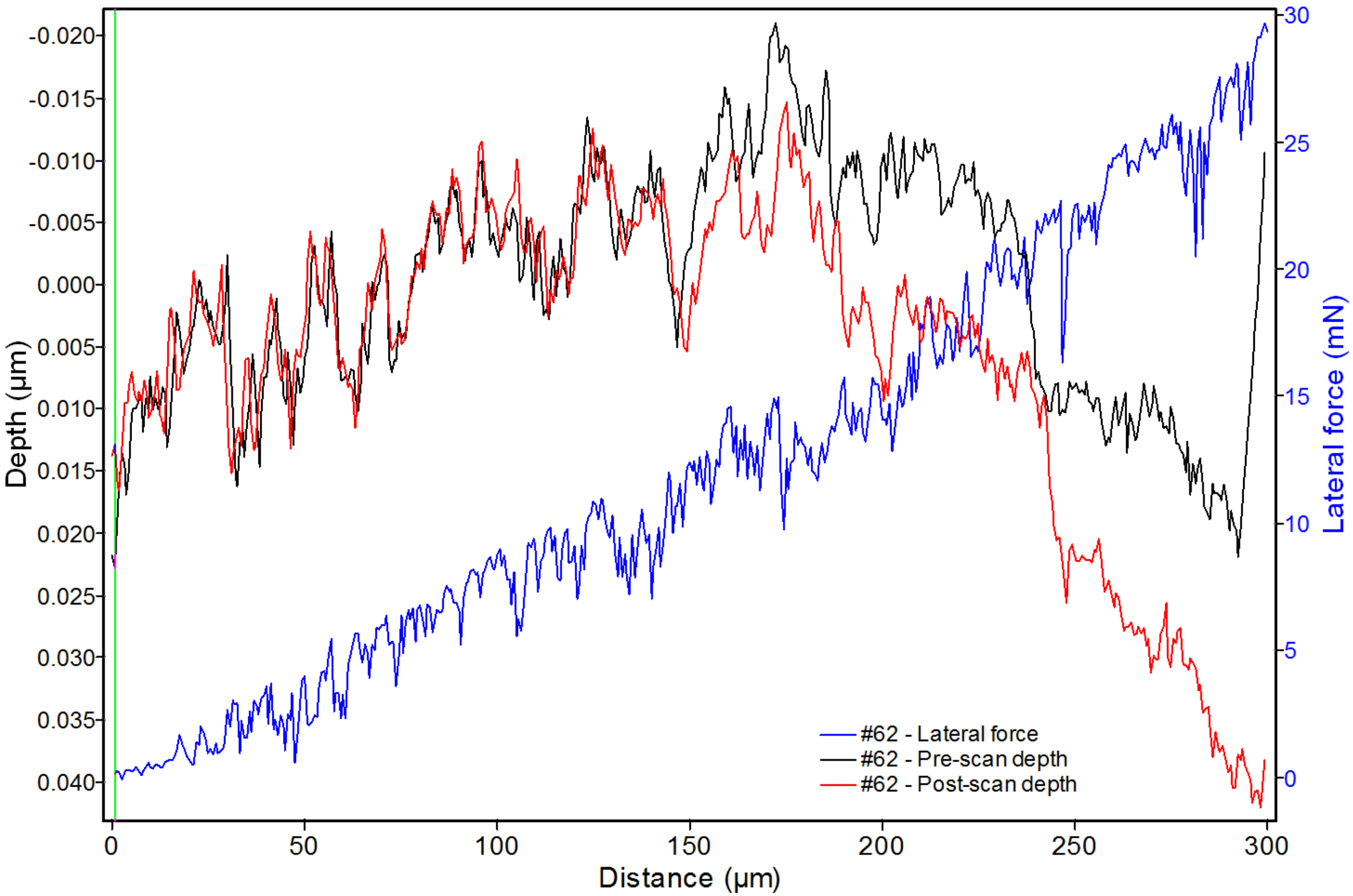
Test 3

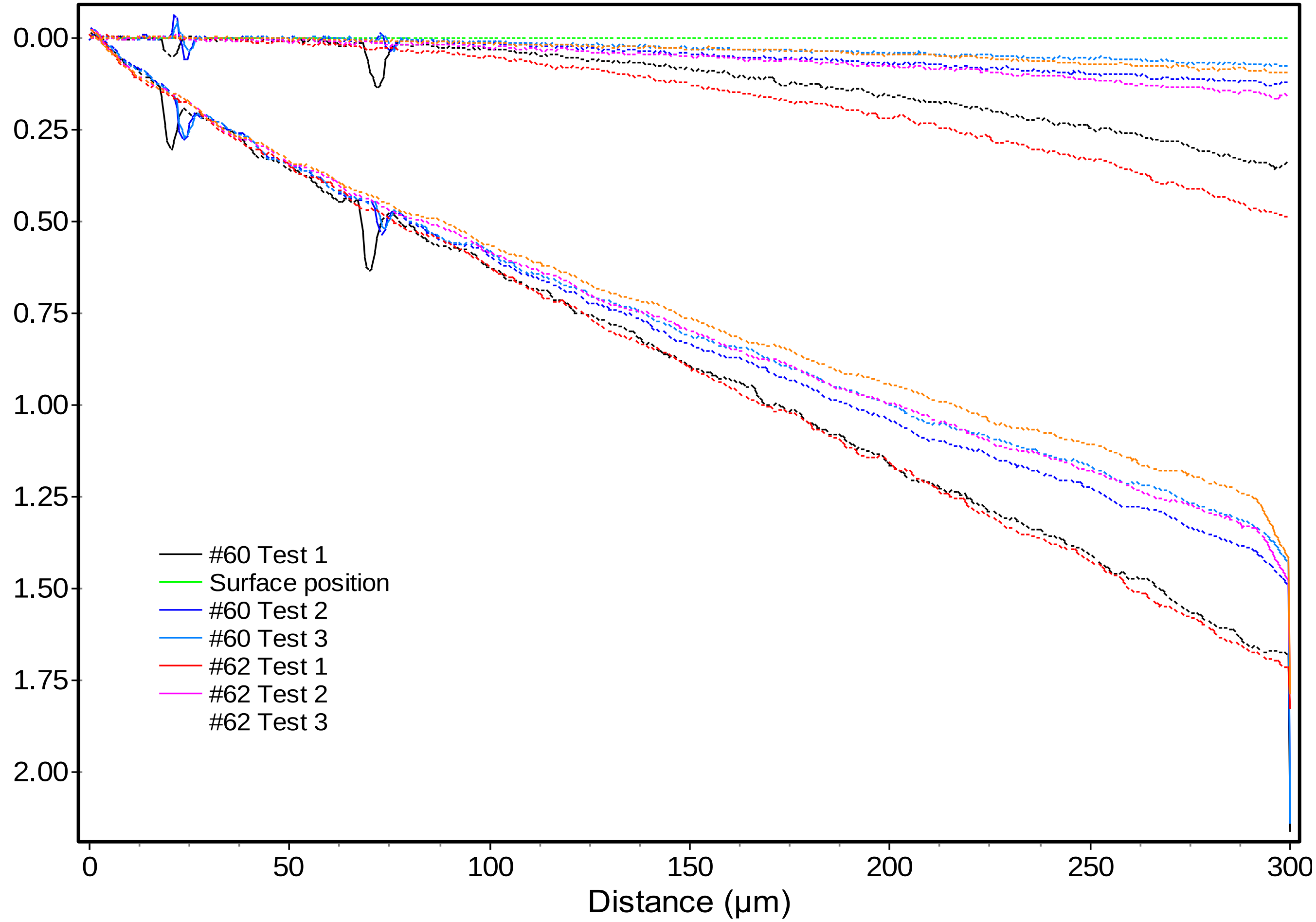
350mN

#62

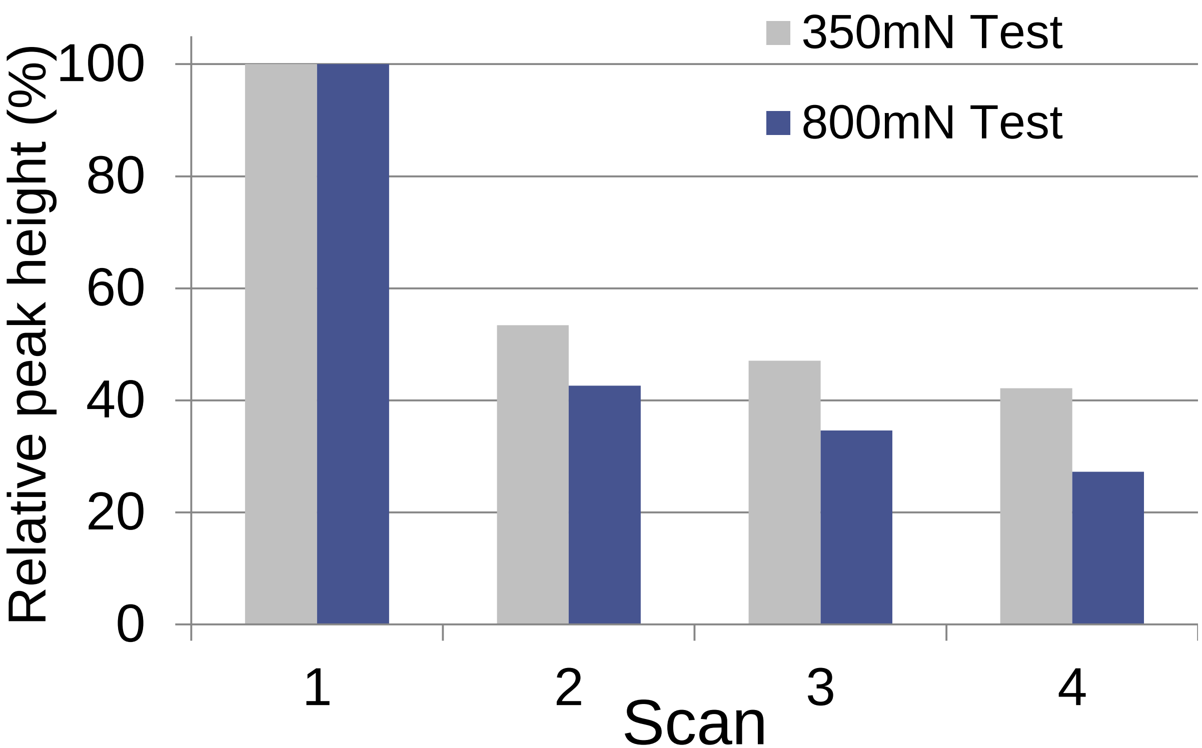


Comparison of surface position before and after test

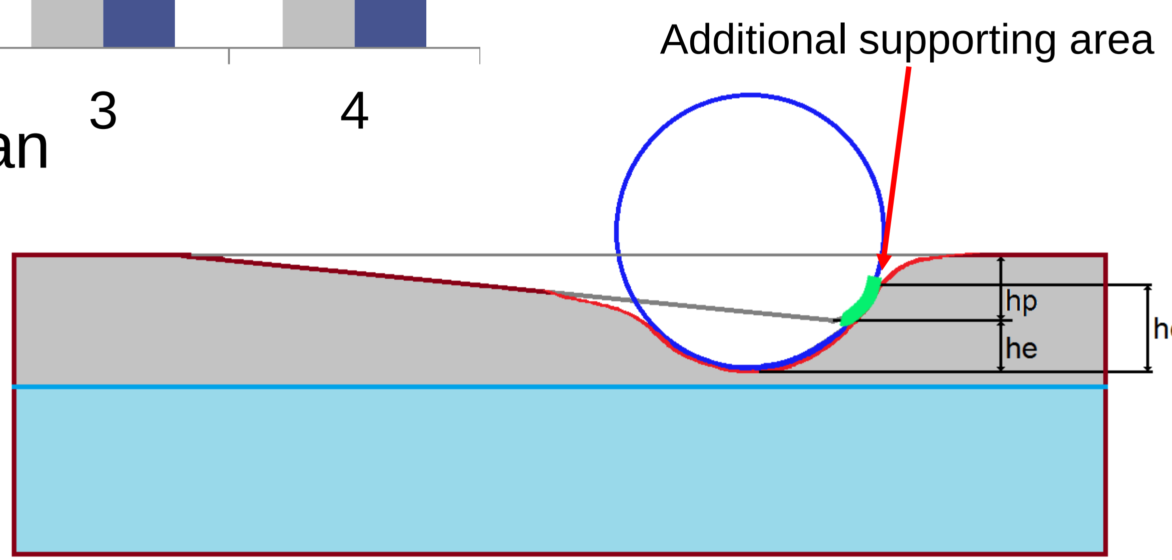
Plastic deformation starts at 177mN



Comparison of depth under load and remaining depth from 800mN tests on sample #60 and 62



Reduction of the relative peak height of sample #60 during repeated loading

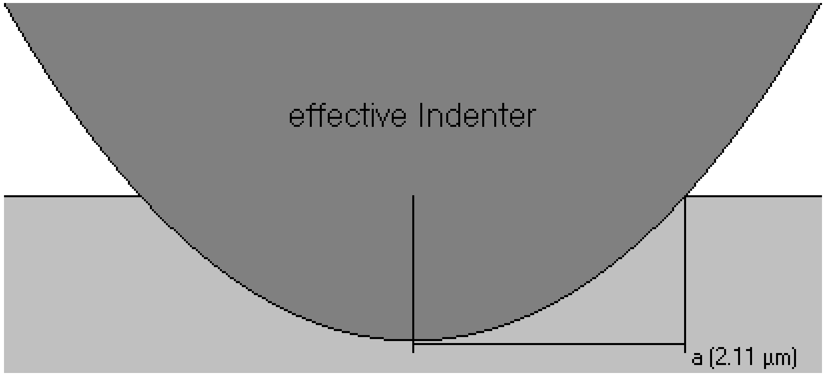


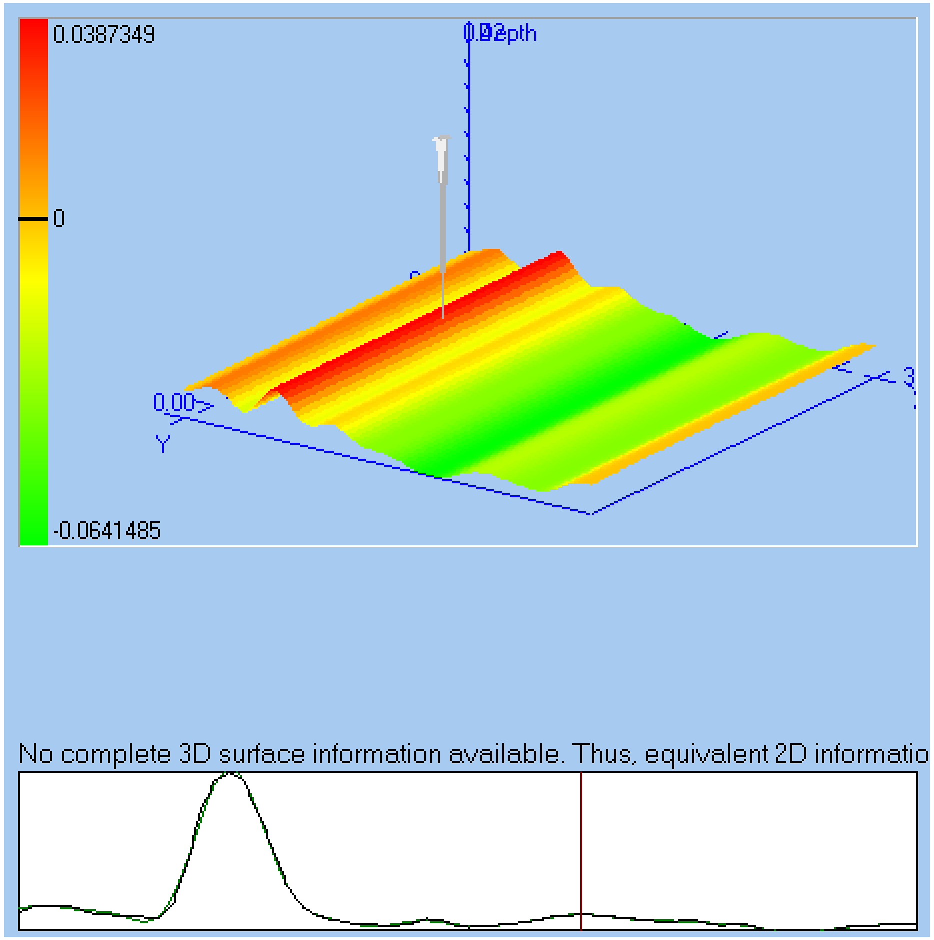
Steady depth increase during repeated loading can be explained by the change of the load carrying area.

Calculations with software **FilmDoctor**

Indenter radius 7µm

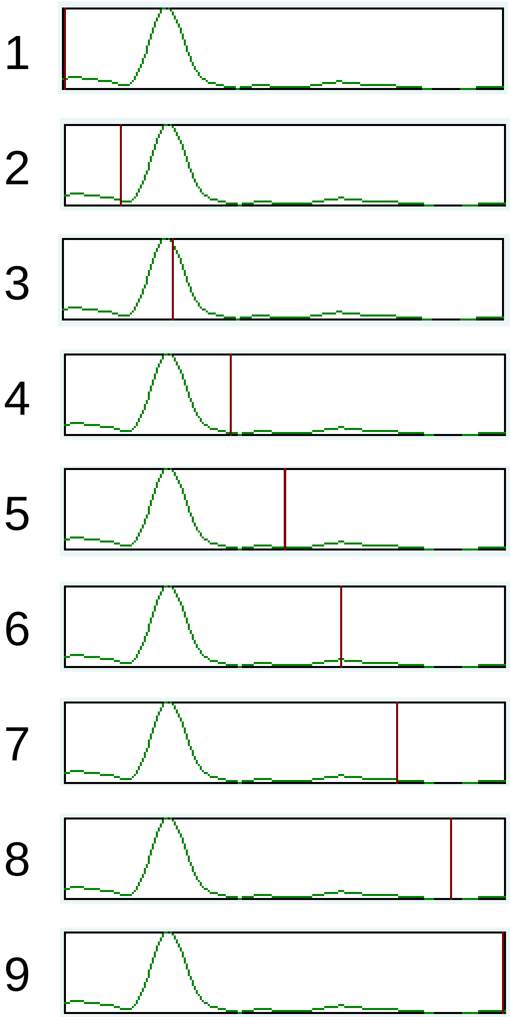
Film 1:	115 GPa	0.22	2.0µm
Film 2:	125 GPa	0.22	4.6µm
Substrate	210 GPa	0.30	

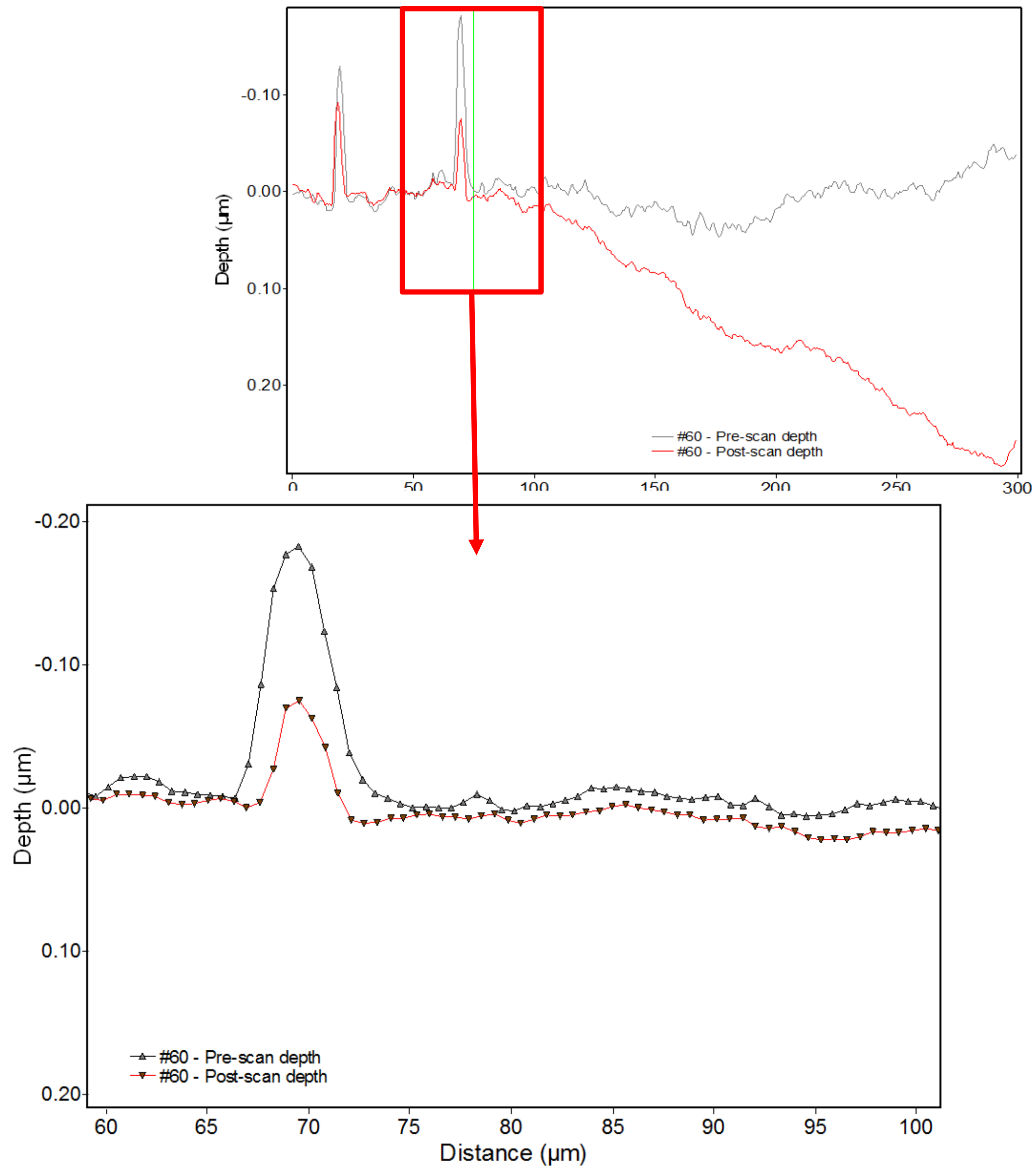


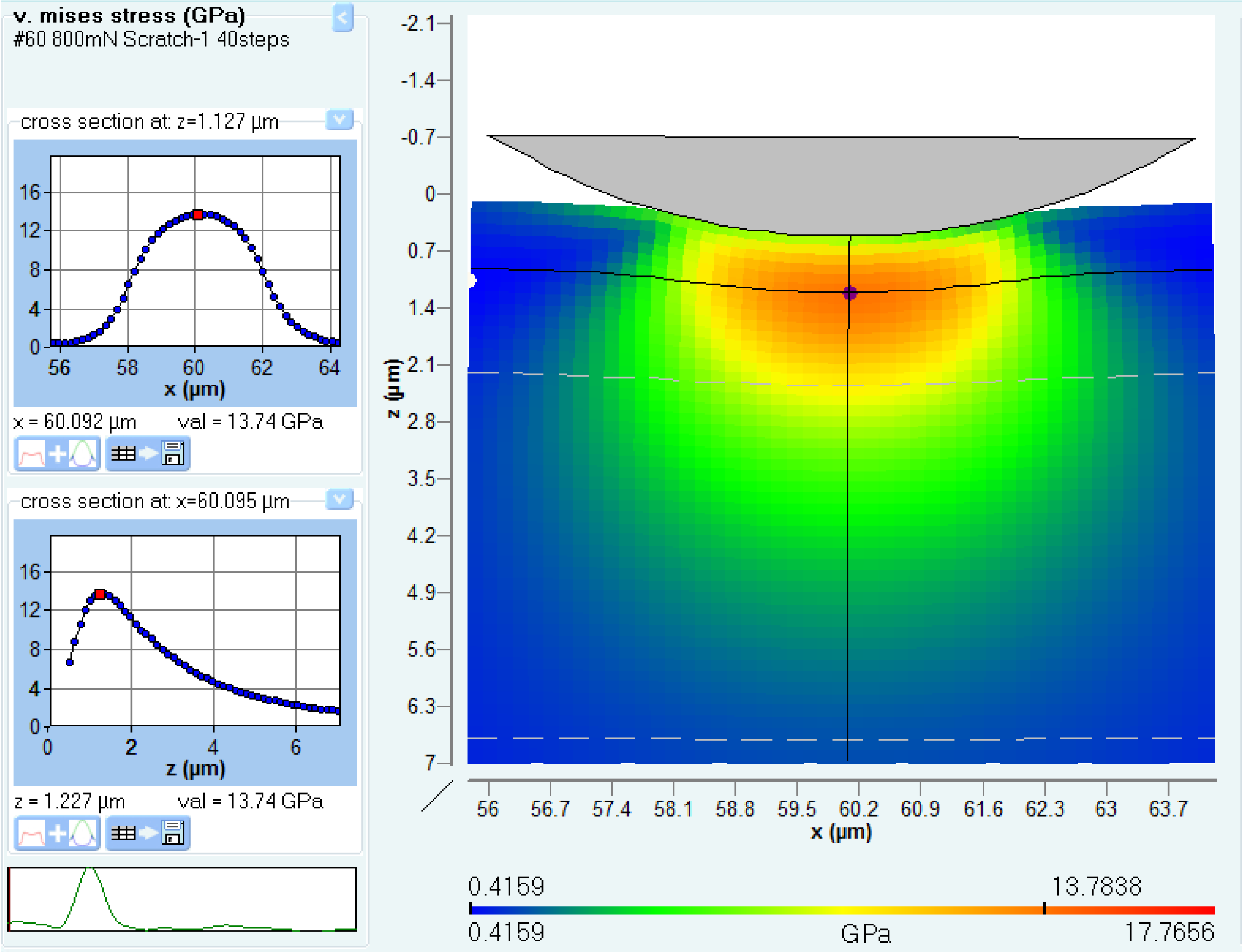


At present roughness information was only available in one direction → Pseudo 3D

Stress analysis is done in the region of beginning plastic deformation

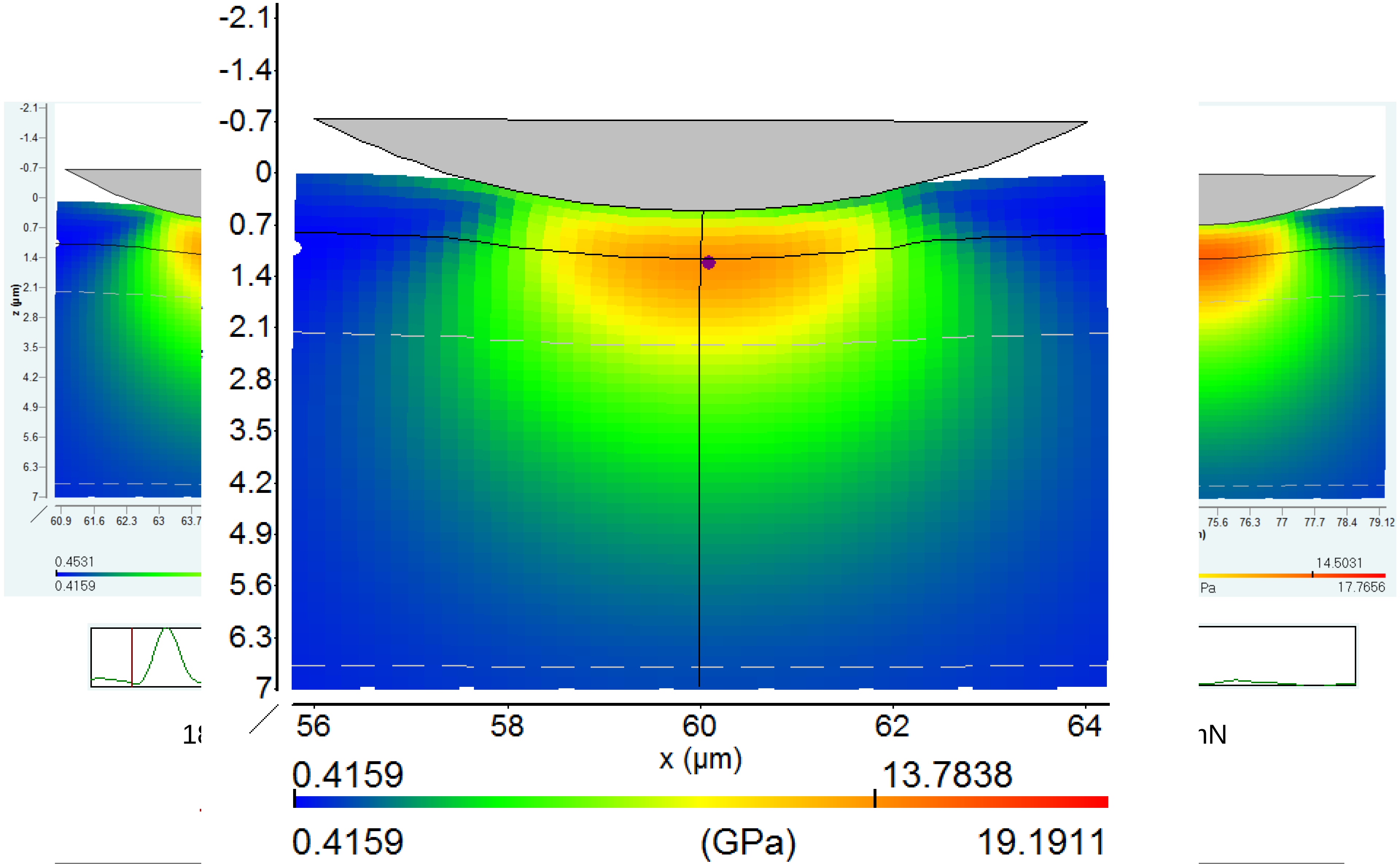

1
2
3
4
5
6
7
8
9


-0.10
0.00
0.10
0.20
Depth (µm)
0
50
100
150
200
250
300
#60 - Pre-scan depth
#60 - Post-scan depth
-0.20
-0.10
0.00
0.10
0.20
Depth (µm)
60
65
70
75
80
85
90
95
100
Distance (µm)
#60 - Pre-scan depth
#60 - Post-scan depth


v. mises stress (GPa)
#60 800mN Scratch-1 40steps
cross section at: z=1.127 µm
x (µm)
x = 60.092 µm val = 13.74 GPa
cross section at: x=60.095 µm
z (µm)
z = 1.227 µm val = 13.74 GPa
z (µm)
x (µm)
0.4159
13.7838
0.4159
GPa
17.7656

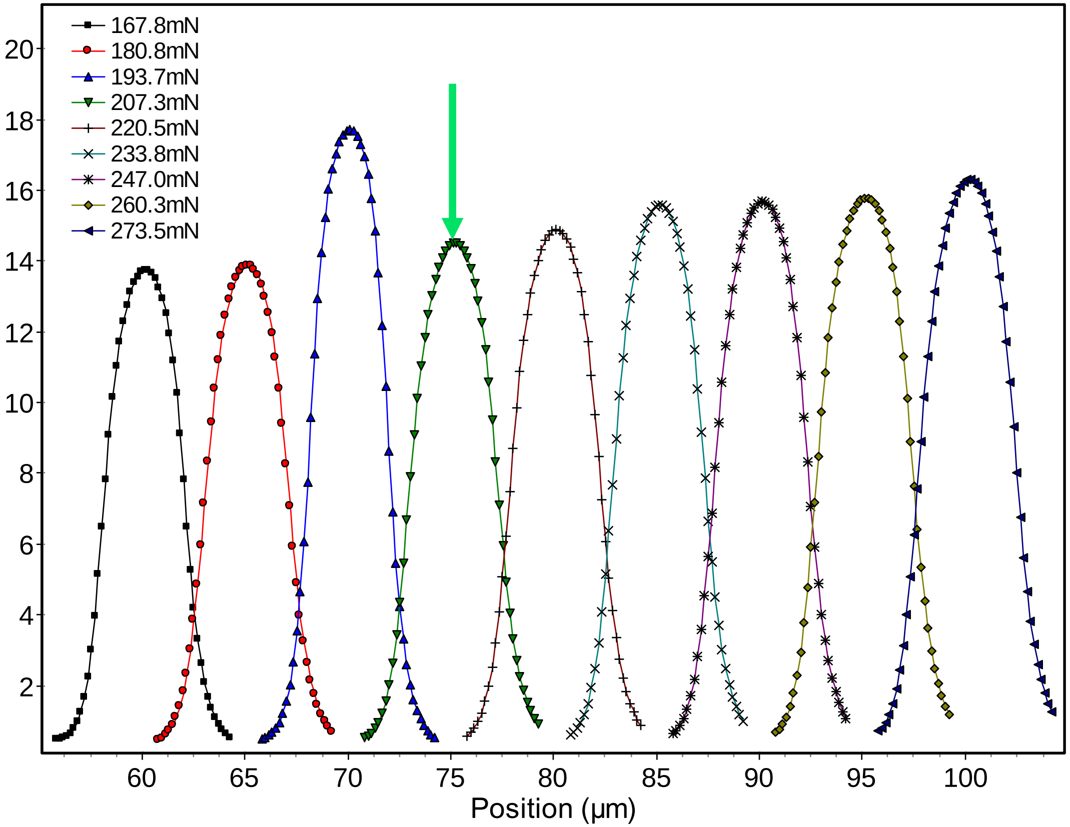
#60 800mN Scratch-1

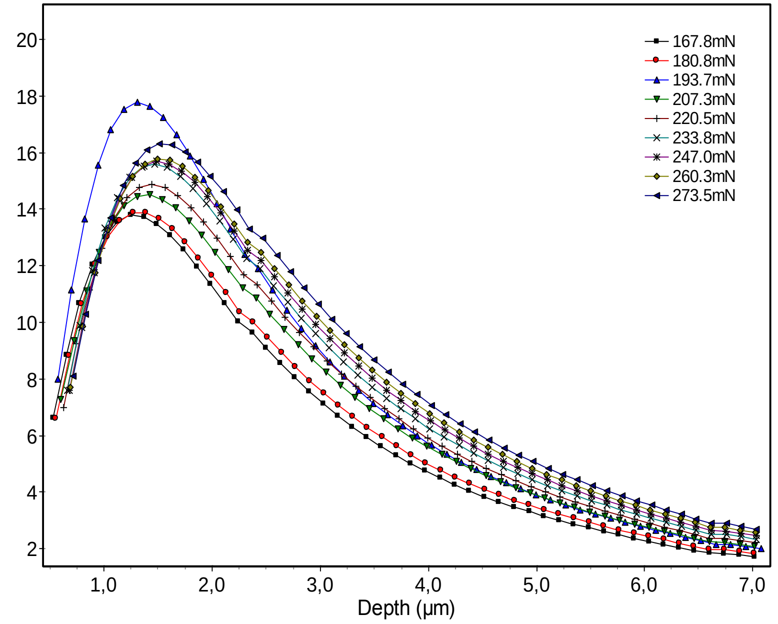
v. mises stress (GPa) at y=0 µm



Sample #60

168mN →→→→→→→→→→→→→→→→→→ → 274mN





Comparison of stress profiles from different positions

Plastic deformation starts at von Mises stress of 14.5 GPa

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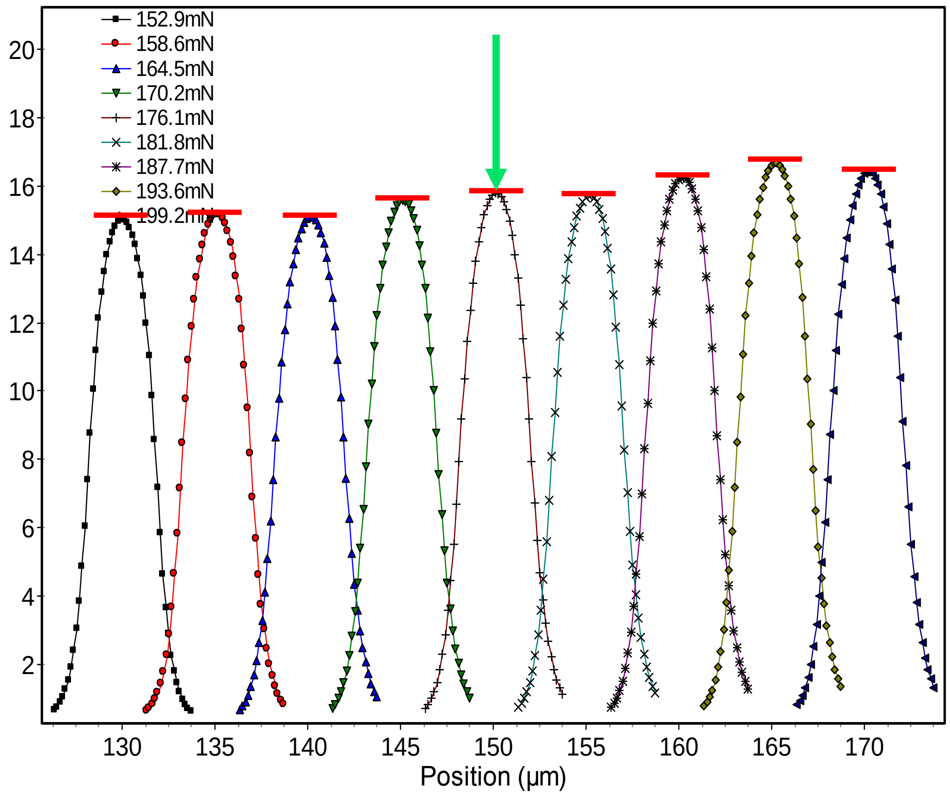
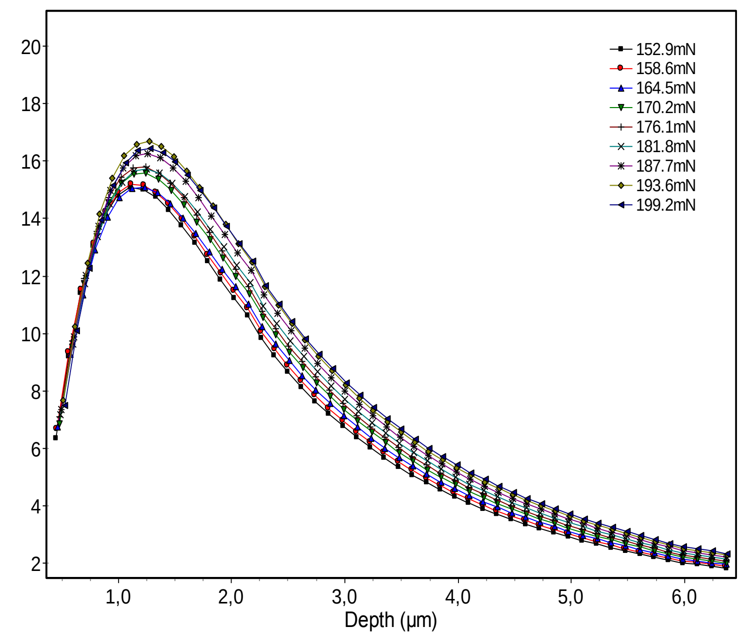
153mN →→→→→→→→→→→→→→→→ 199mN





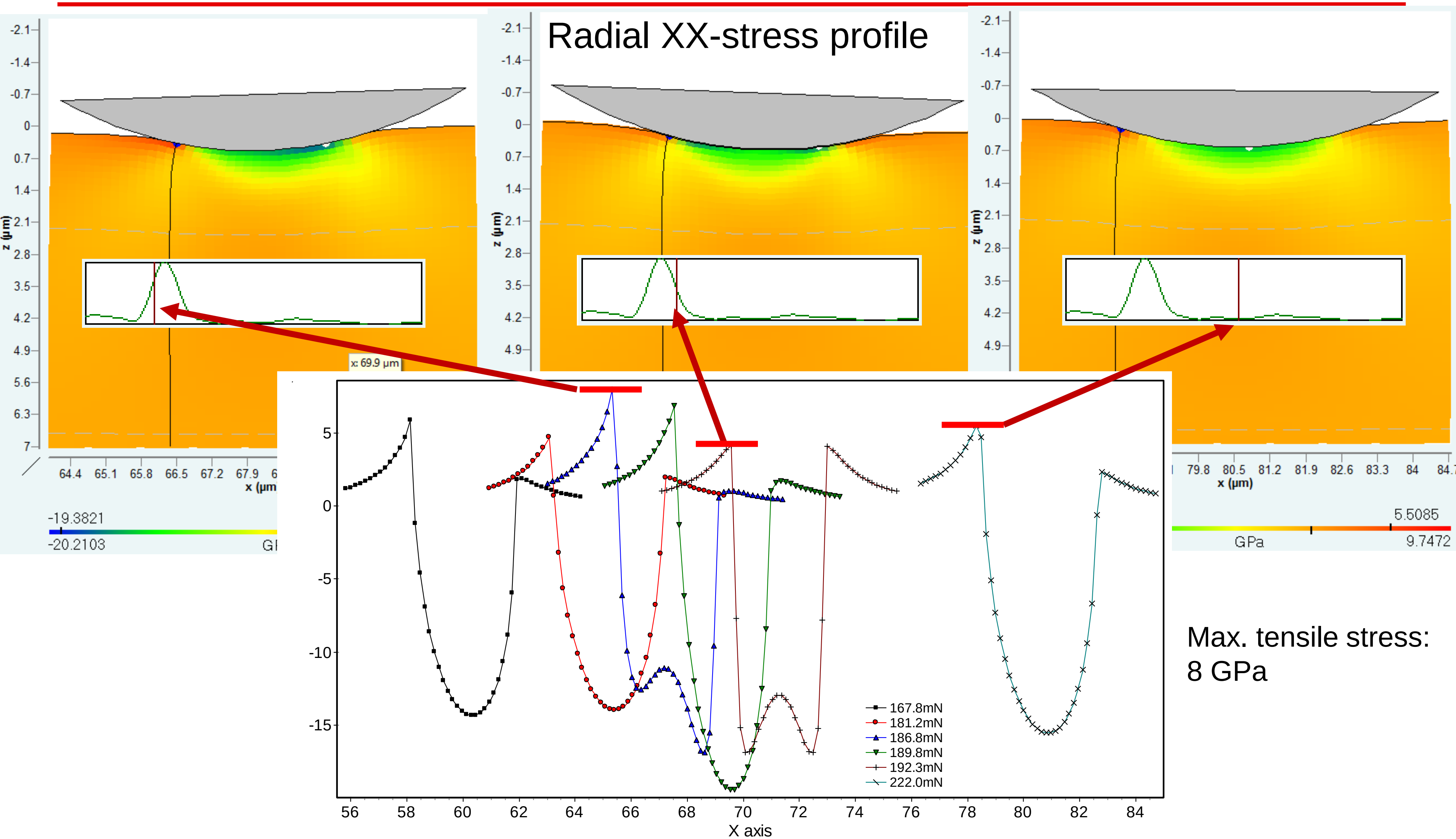
Comparison of stress profiles from different positions

The stress is not steadily increasing with force

Plastic deformation starts at von Mises stress of 15.8 GPa

Sample #60

Radial XX-stress profile



Max. tensile stress: 8 GPa

The tensile stress is considerably higher in regions with increasing surface profile

Zwick / Roell

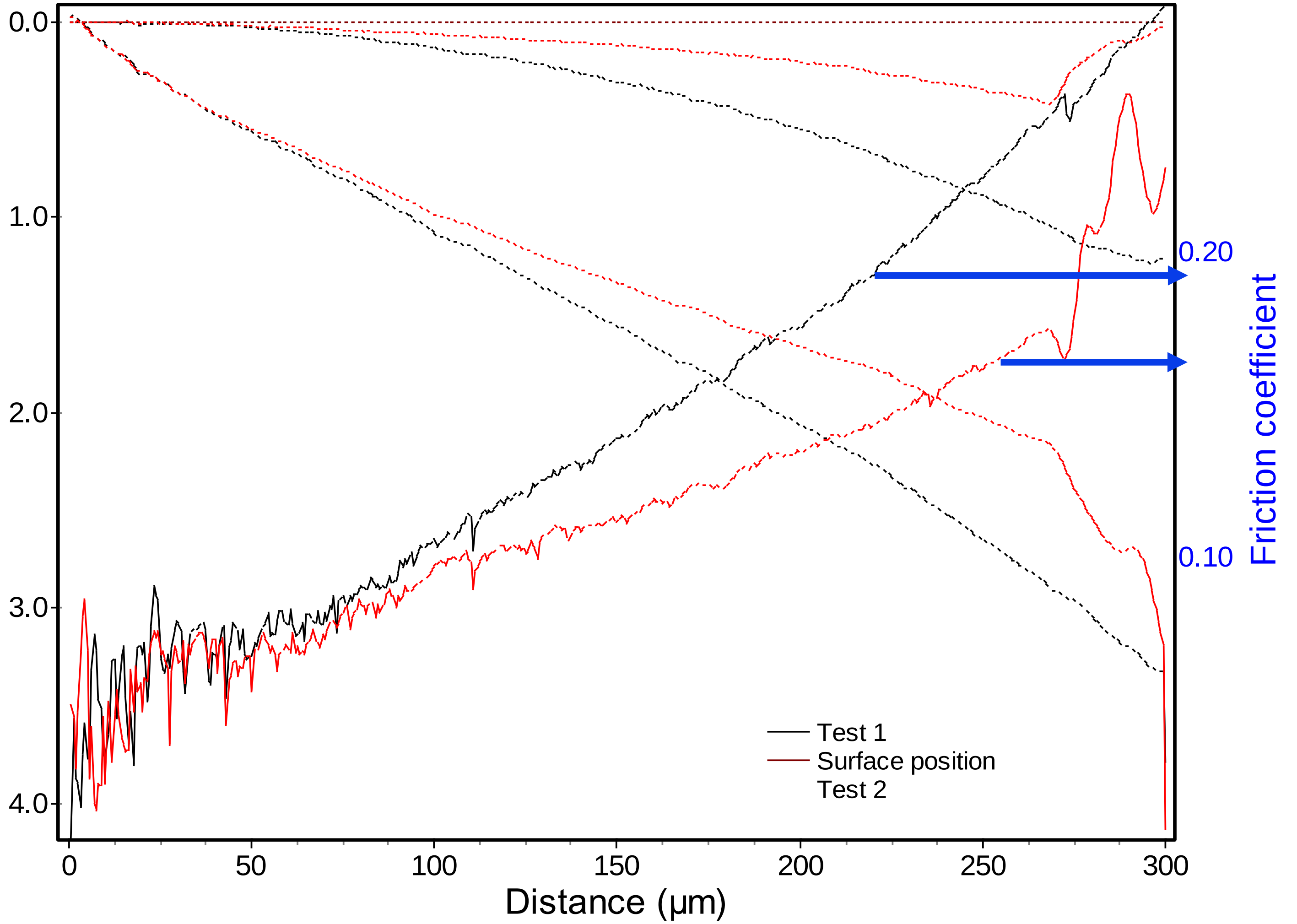
Failure type: fracture

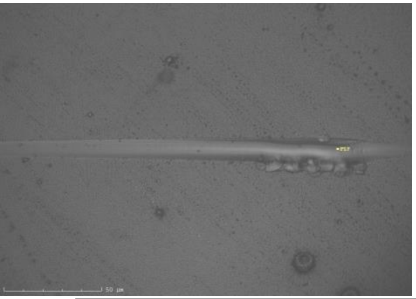
asmec ADVANCED SURFACE MECHANICS

Test 1+2

1500mN

#60



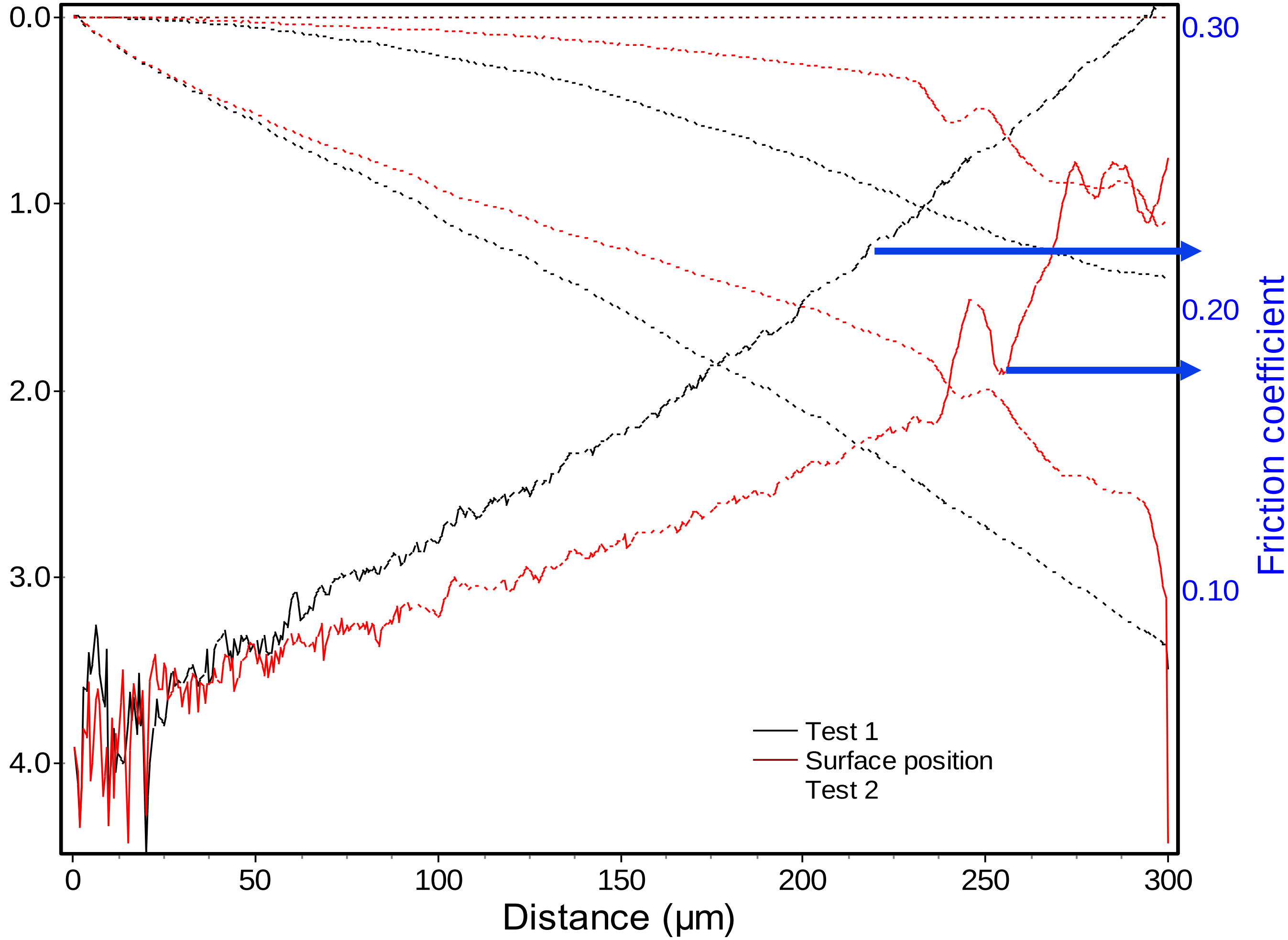


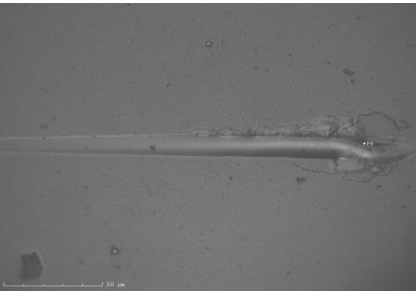
Comparison of test 1 and 2
Fracture starts in test 2 at about 1341mN

Test 1+2

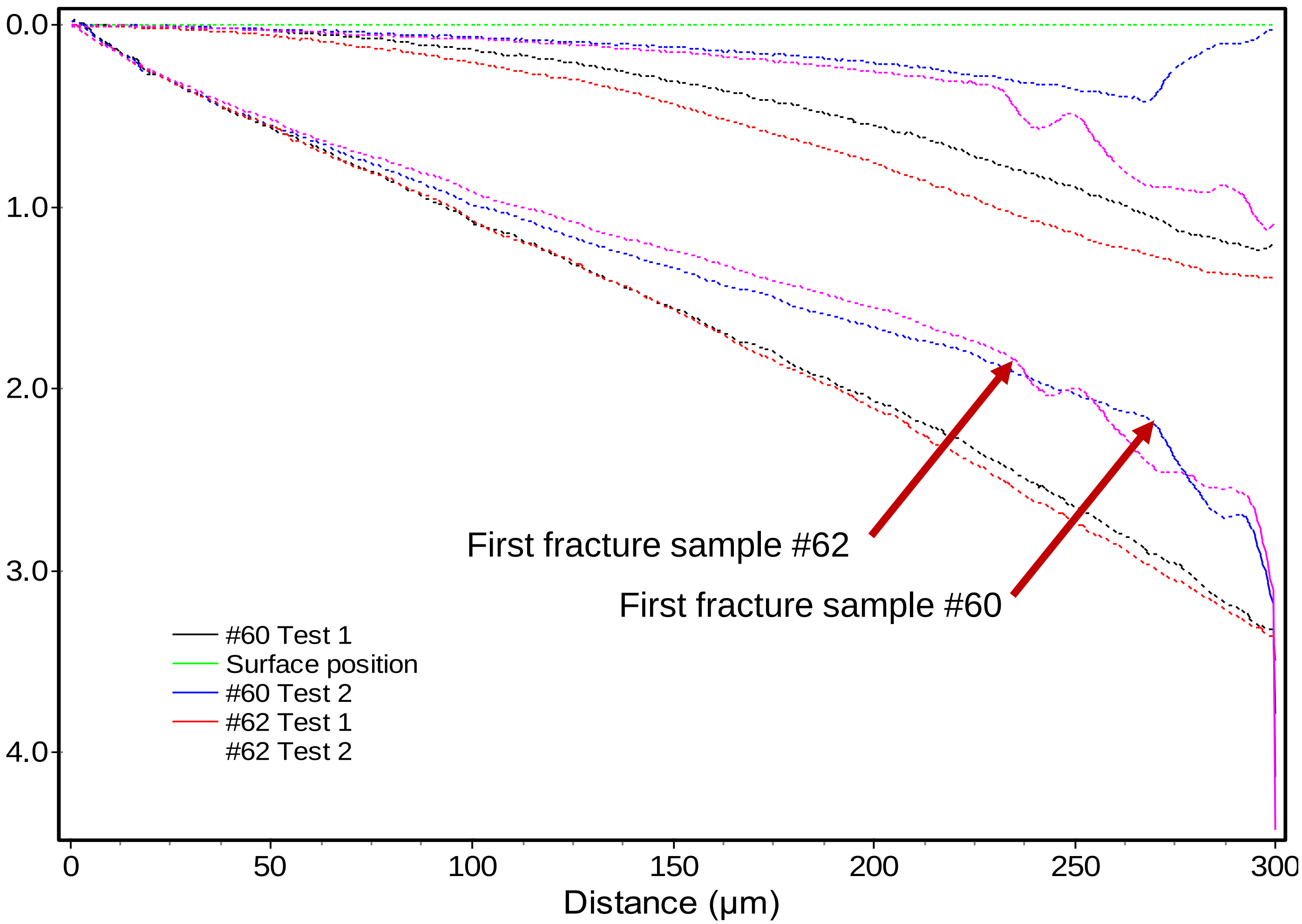
1500mN

#62

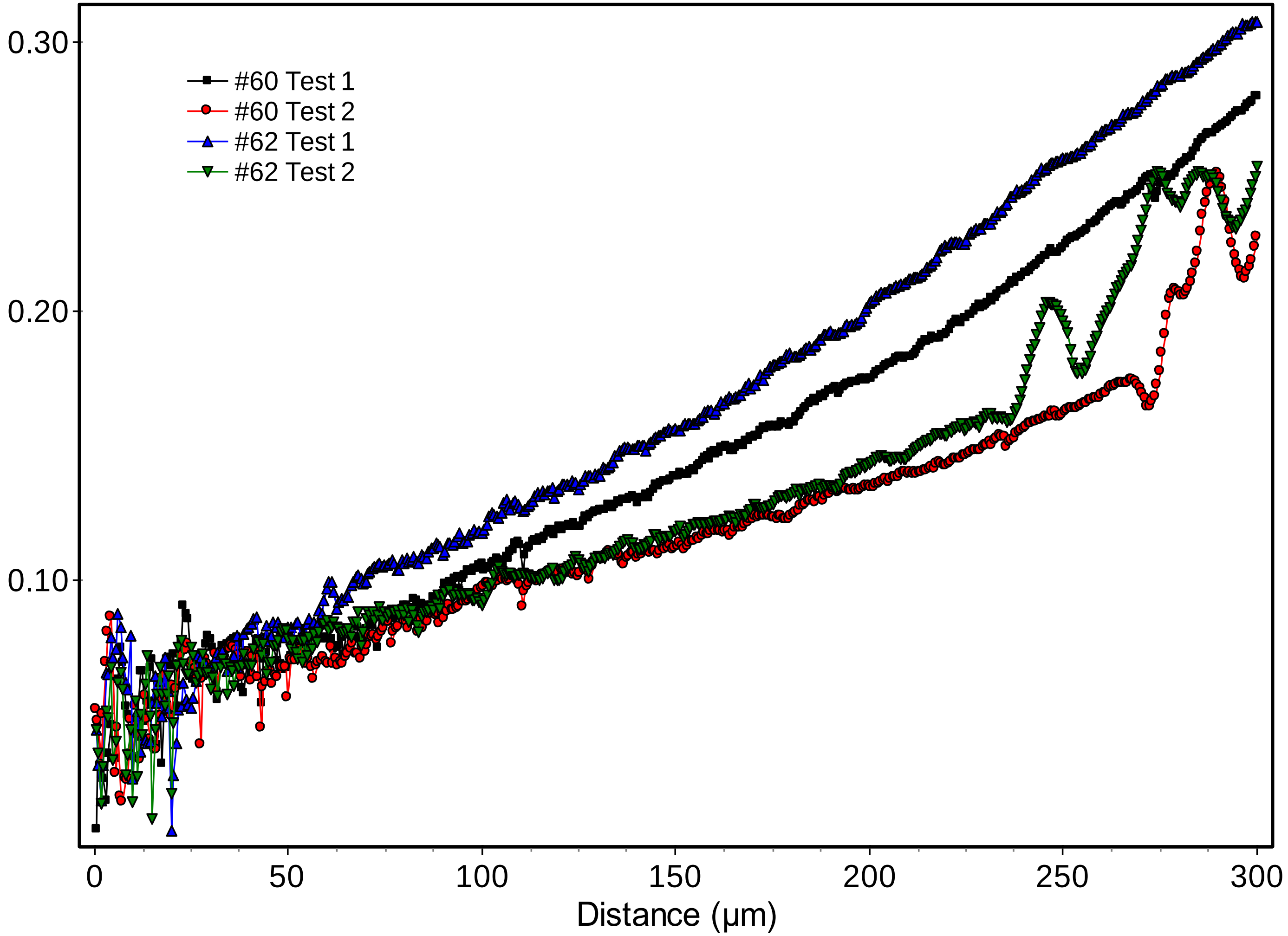




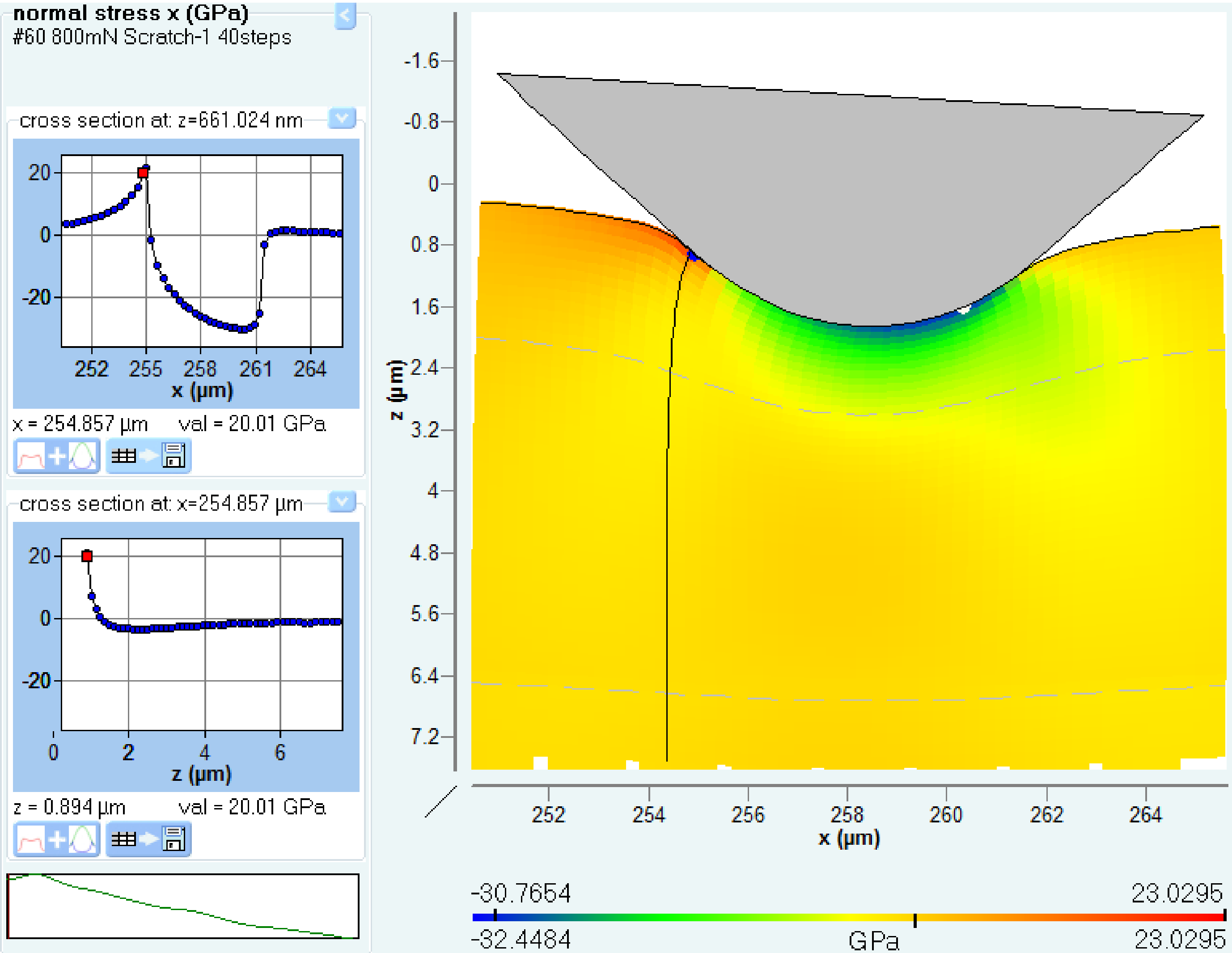
Comparison of test 1 and 2 with
Fracture starts in test 2 at about 1180mN



Comparison of depth under load and remaining depth from 1500mN tests on sample #60 and 62



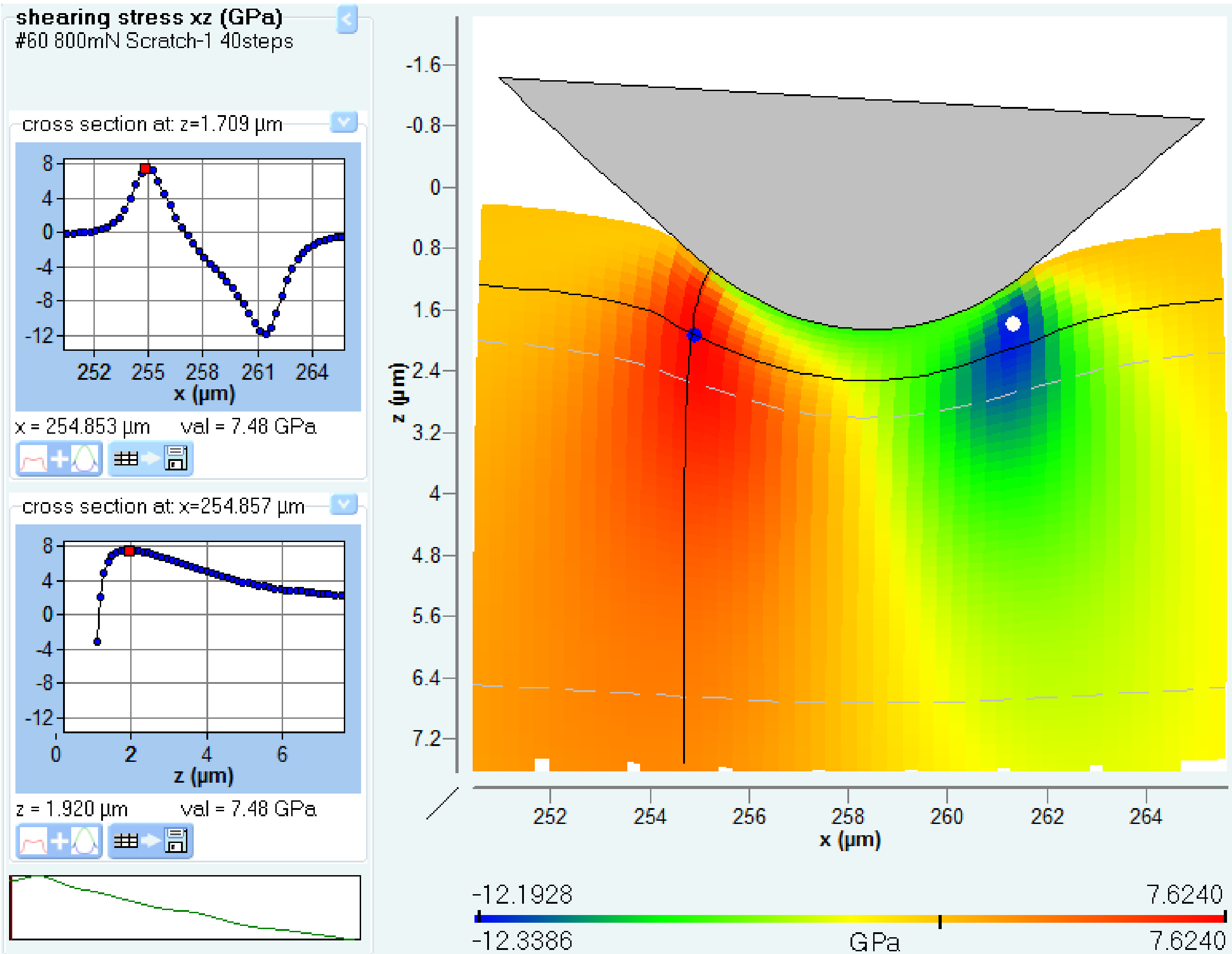
Comparison of friction coefficient from 1500mN tests on sample #60 and 62



Maximum tensile stress behind indenter:

20 GPa

Sample# 60, radial XX-stress just before fracture

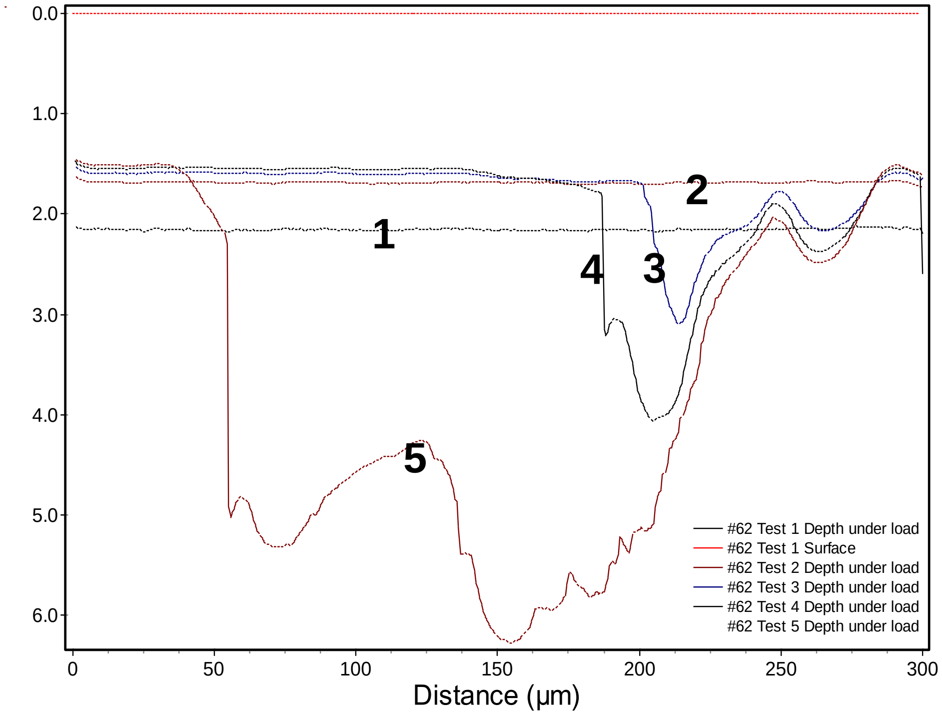


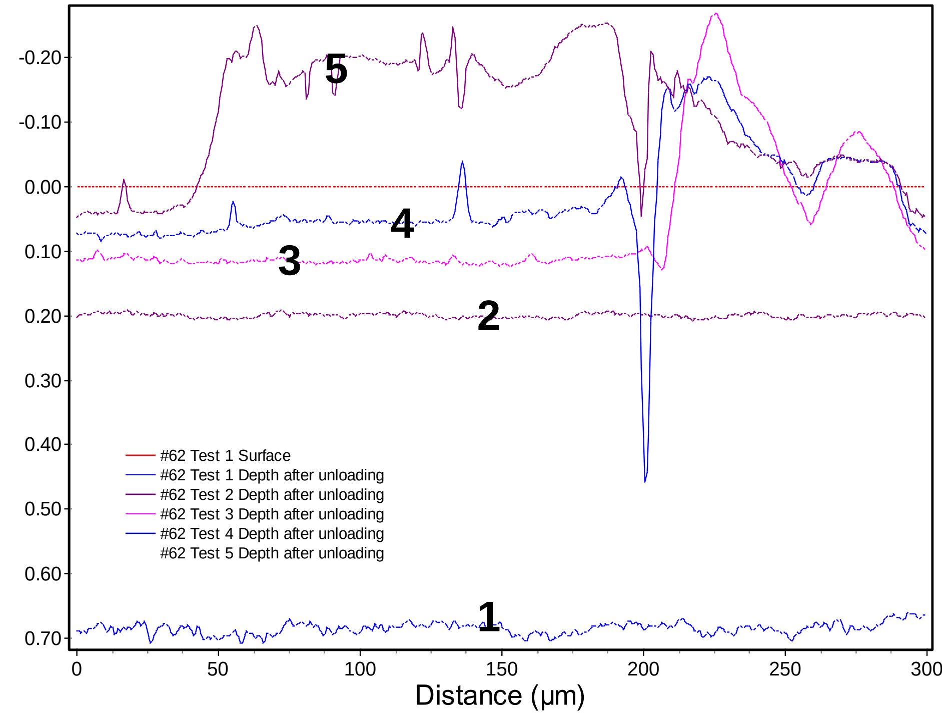
Maximum shear stress behind indenter:

8 GPa

Sample# 60, XZ shear stress just before fracture

Comparison of 5 successive scratch tests with constant load on sample #62
distance 300µm, force 1000mN





Development of the depth under load from test to test

Development of the depth change from test to test (profile after-before test)
The surface after the test is higher than before the test

Summary

Sample	#60 gradient	#62 homogeneous
Hardness	11.5 GPa	14.5 GPa
Modulus at surface	120 GPa	150 GPa
Elastic-plastic transition	207 mN	173 mN
Max. von Mises stress	14.5 GPa	15.8 GPa
First fracture in test 2	1341 mN	1180 mN
Max. tensile stress at fracture	20 GPa	
Max. shear stress at fracture	8 GPa	

The stress calculation did not consider the real 3D surface profile and the multilayer structure of the coating Therefore the stresses are overestimated here.

Conclusions

- In micro scratch tests it is possible to detect the elastic – plastic transition and to follow a peak reduction in repeated tests at same position
- Roughness is reduced but not removed after plastic deformation in a scratch track
- The stress is not steadily increasing with increasing force but depends on surface profile
- At peaks the von Mises stress is considerably increased
- At increasing flanks of peaks the tensile stress is considerably increased
- Severe coating failure may occur after repeated loading of the same position → a single scratch my not be enough to evaluate the failure probability of coatings
- The gradient layer is more failure resistant than a homogeneous layer
- Detailed stress calculations help to understand coating failures and to optimize coatings. This requires an accurate determination of indenter radius.

Thank you for your attention !

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