Evaluating true coating properties from an indentation measurement series – A new combination of calotte grinding and indentation tests

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Movitation

Goal

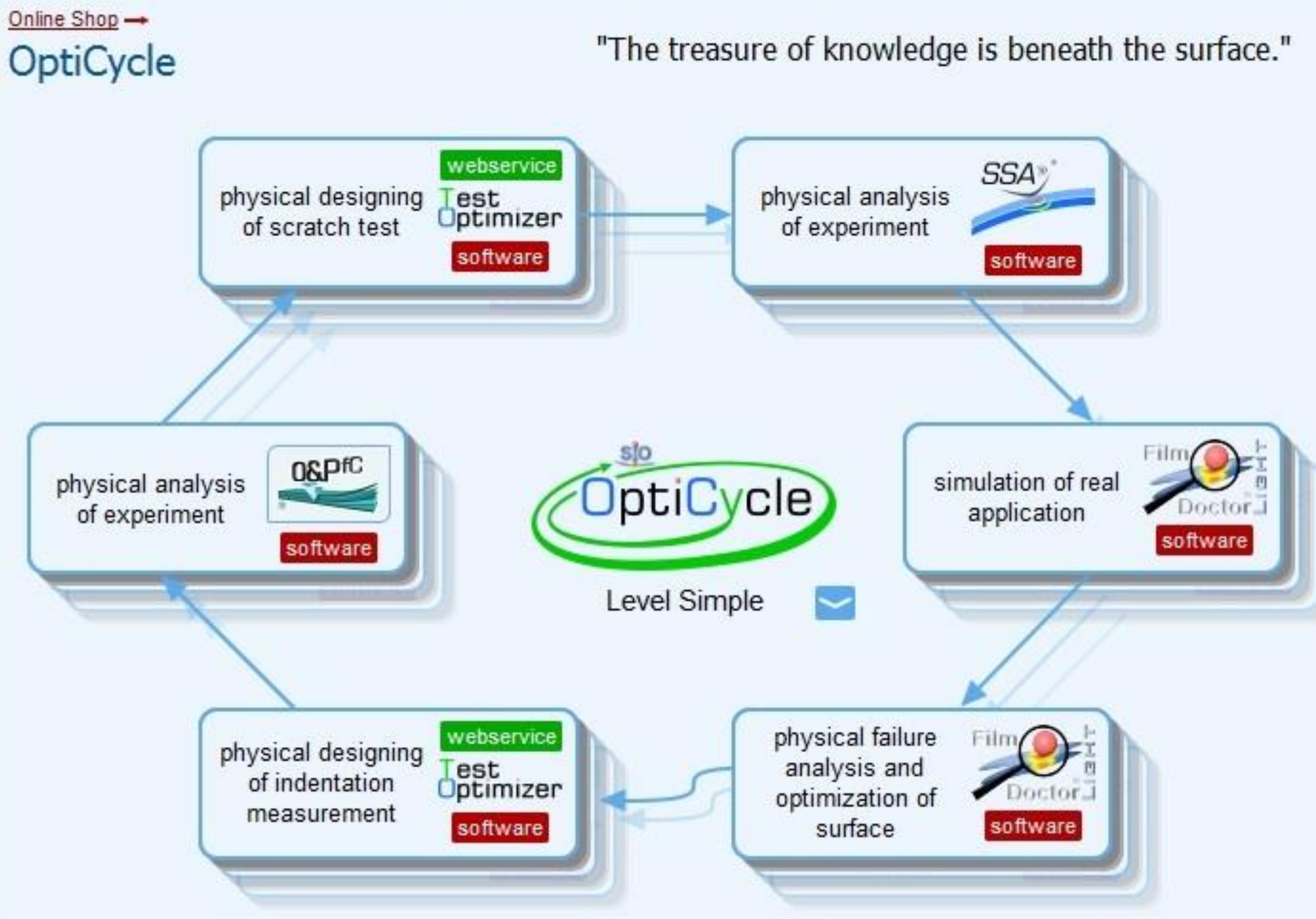
Optimize the materials and material combinations to increase application performance to reach certain goals (e.g. reduced fuel consumption or carbon dioxide emission)

- but structures are more and more complex nowadays

- need computers and models, personal experience and rules of thumb are no longer enough

- generic material parameters needed

The flow chart of the Plan



Online Shop →

OptiCycle

"The treasure of knowledge is beneath the surface."

physical designing of scratch test

physical analysis of experiment

simulation of real application

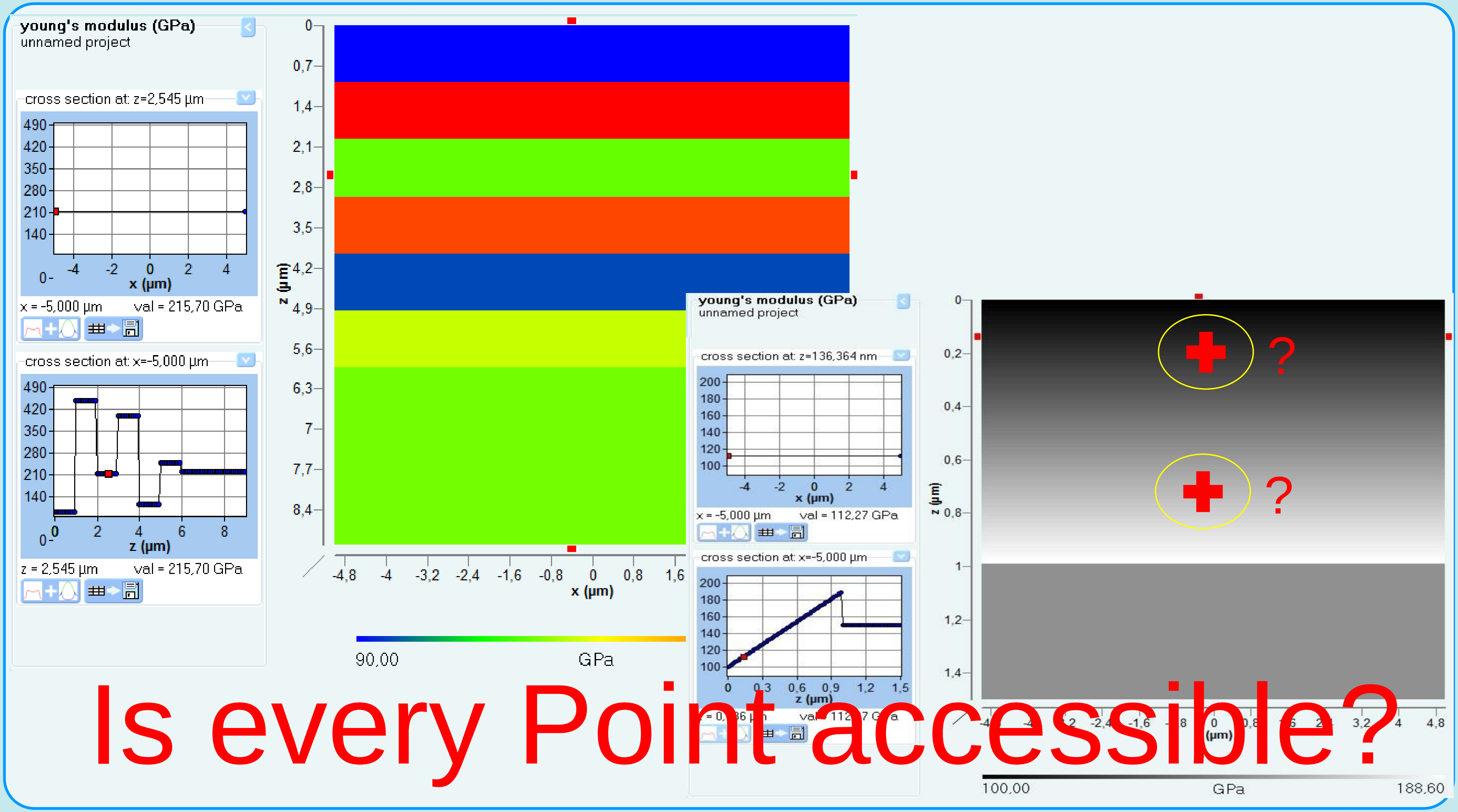
physical failure analysis and optimization of surface

physical designing of indentation measurement

physical analysis of experiment

http://siomec.de/en/119/OptiCycle

Complex structures – multilayers & gradients



young's modulus (GPa)
unnamed project

cross section at: z=2,545 µm

x = -5,000 µm val = 215,70 GPa

cross section at: x=-5,000 µm

z = 2,545 µm val = 215,70 GPa

young's modulus (GPa)
unnamed project

cross section at: z=136,364 nm

x = -5,000 µm val = 112,27 GPa

cross section at: x=-5,000 µm

Is every Point accessible?

Information at every point accessible?

No!

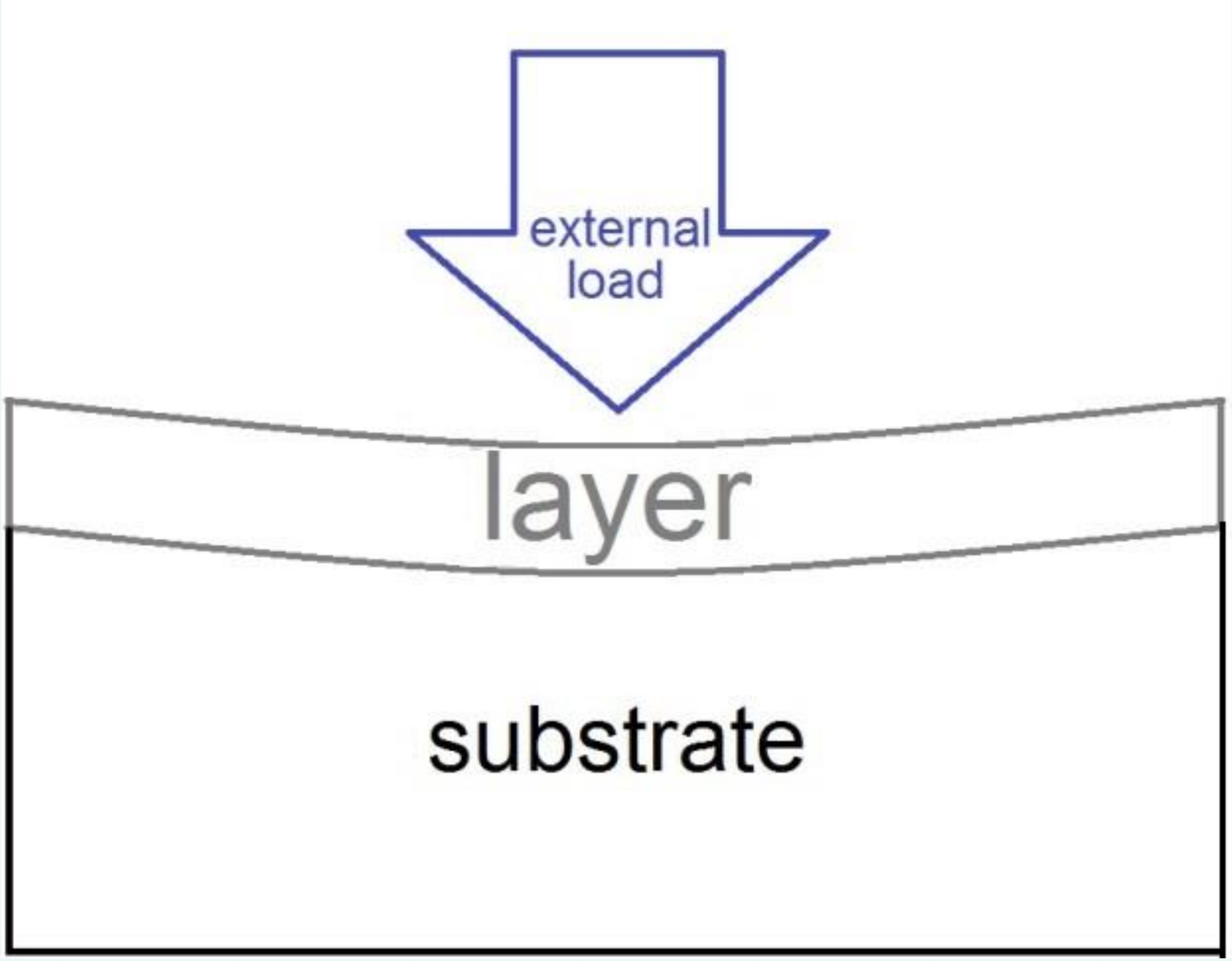
Because of shadow effects

Eggshell shadowing

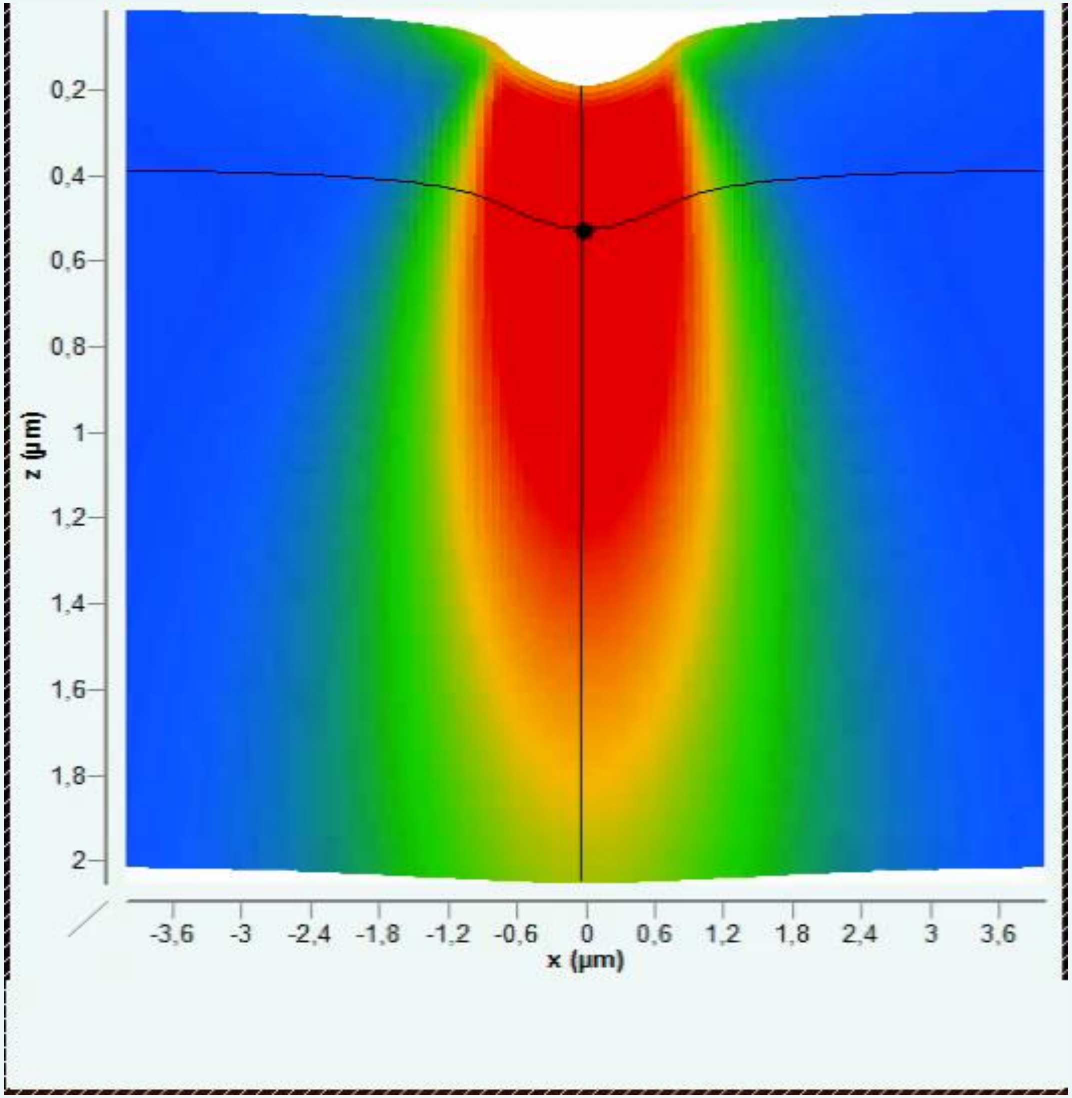
The eggshell shadowing.

Example: Simple 1 layer system $\frac{E_{layer}}{E_{substrate}}$

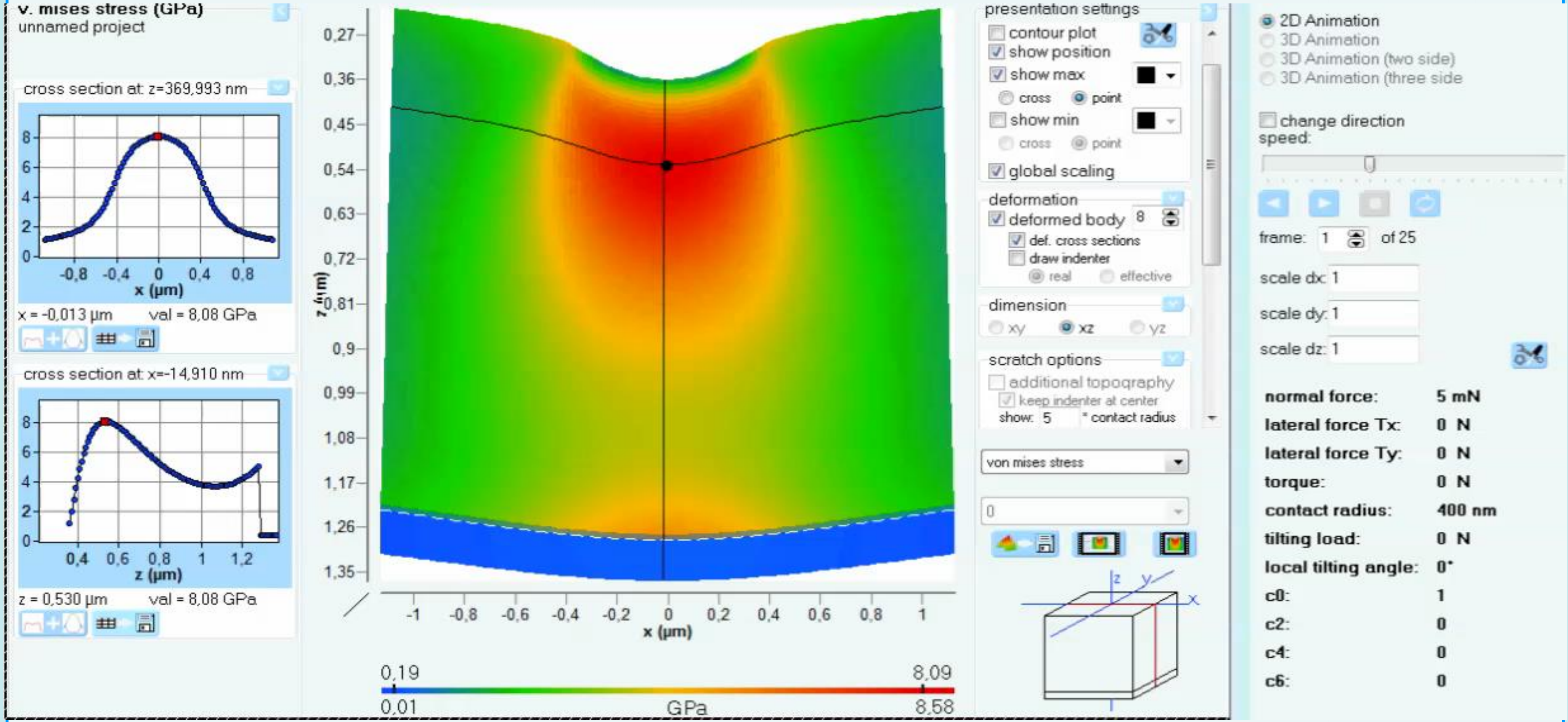
Eggshell shadowing



Homogeneous material



Eggshell effect: E=70 GPa on 4 GPa



x = -0,013 µm val = 8,08 GPa

z = 0,530 µm val = 8,08 GPa

presentation settings

- ☐ contour plot
- ☑ show position
- ☑ show max
  - ○ cross ● point
- ☐ show min
  - ○ cross ● point
- ☑ global scaling

deformation

- ☑ deformed body 8
  - ☑ def. cross sections
  - ☐ draw indenter
    - ● real ○ effective

dimension

○ xy ● xz ○ yz

scratch options

- ☐ additional topography
  - ☑ keep indenter at center
  - show: 5 * contact radius

von mises stress

0

- ● 2D Animation
- ○ 3D Animation
- ○ 3D Animation (two side)
- ○ 3D Animation (three side

☐ change direction

speed:

frame: 1 of 25

scale dx: 1

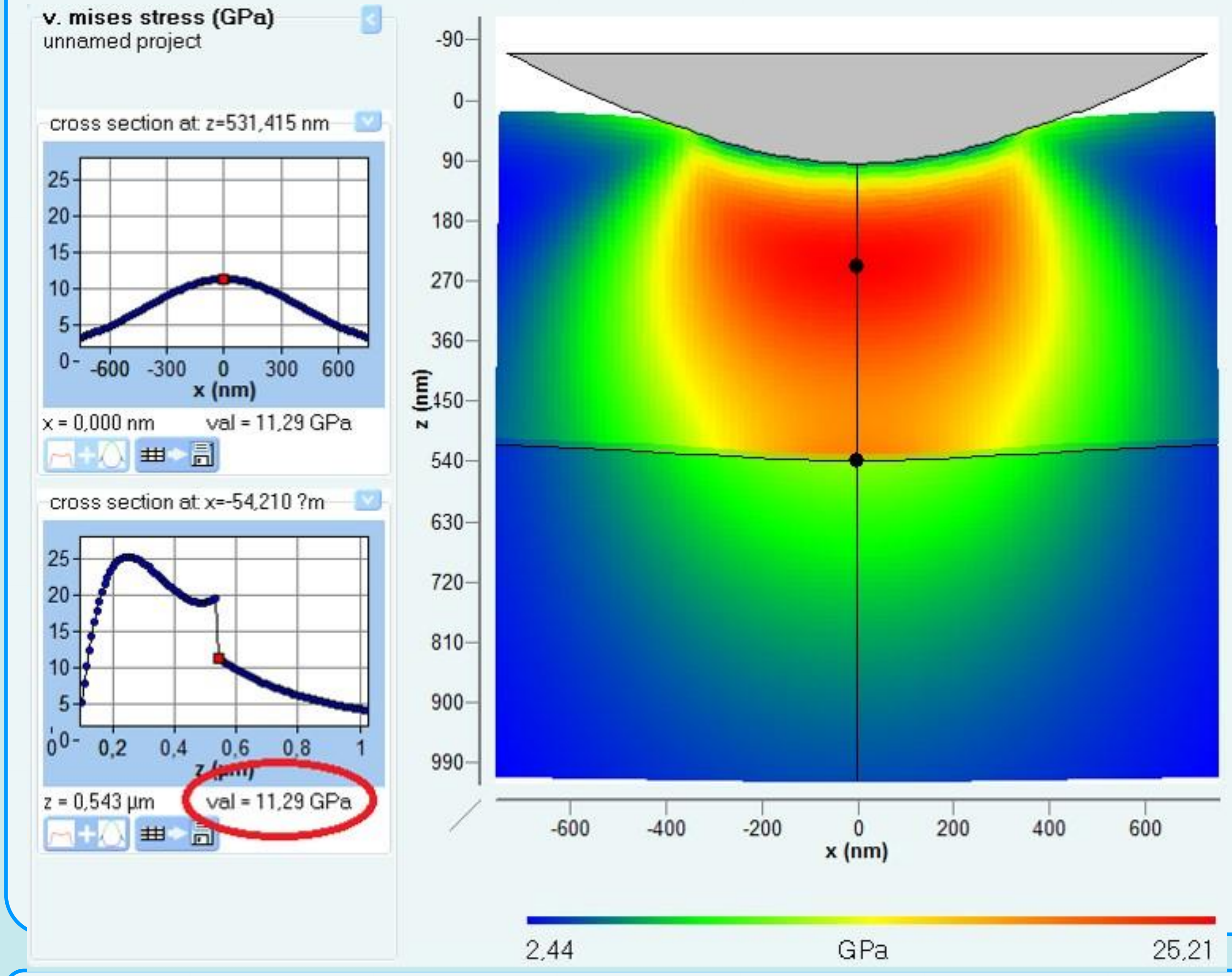
scale dy: 1

scale dz: 1

normal force:	**5 mN**
lateral force Tx:	**0 N**
lateral force Ty:	**0 N**
torque:	**0 N**
contact radius:	**400 nm**
tilting load:	**0 N**
local tilting angle:	**0°**
c0:	**1**
c2:	**0**
c4:	**0**
c6:	**0**

Yield ratio shadowing

Shadowing by high ratio $\frac{Y_{layer}}{Y_{substrate}}$

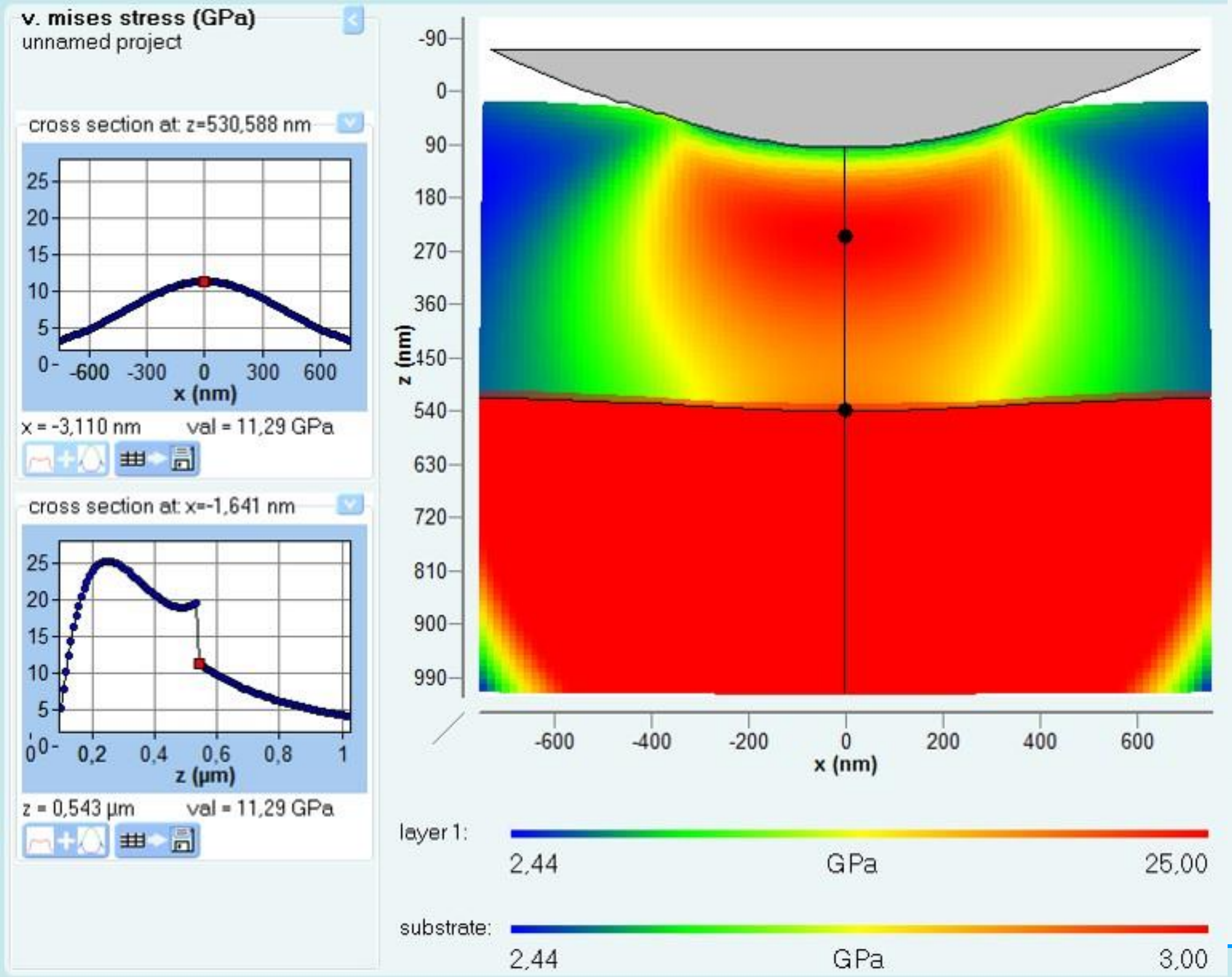


Example TiN on Steel
->$Y_{substrate} \ll Y_{layer}$

(E=590 on 221 GPa,
Y=25 on 3 GPa)

Yield ratio shadowing

Shadowing by high ratio $\frac{Y_{layer}}{Y_{substrate}}$



x = -3,110 nm val = 11,29 GPa

z = 0,543 µm val = 11,29 GPa

layer 1: 2,44 GPa 25,00

substrate: 2,44 GPa 3,00

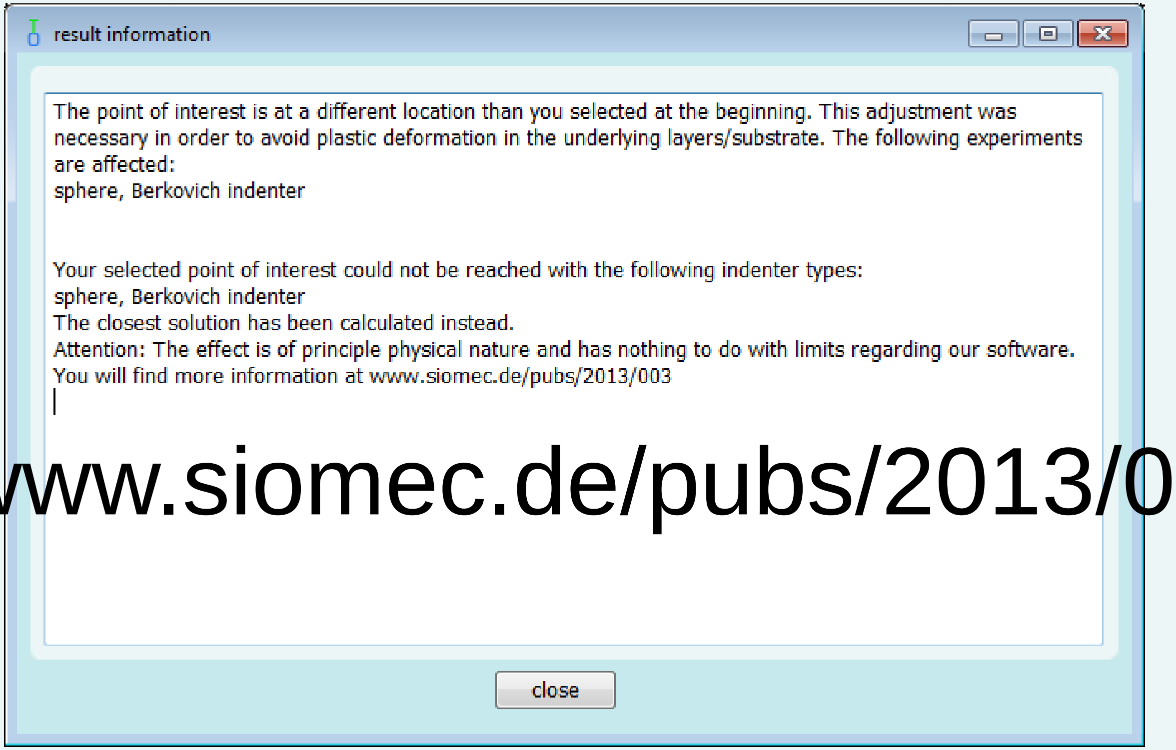
Example TiN on Steel
->$Y_{substrate} \ll Y_{layer}$

(E=590 on 221 GPa,
Y=25 on 3 GPa)

Shadow effects

TestOptimizer (www.siomec.de/testoptimizer)

- calculates optimum values for load and indenter parameter
- shows hints about shadow effects.

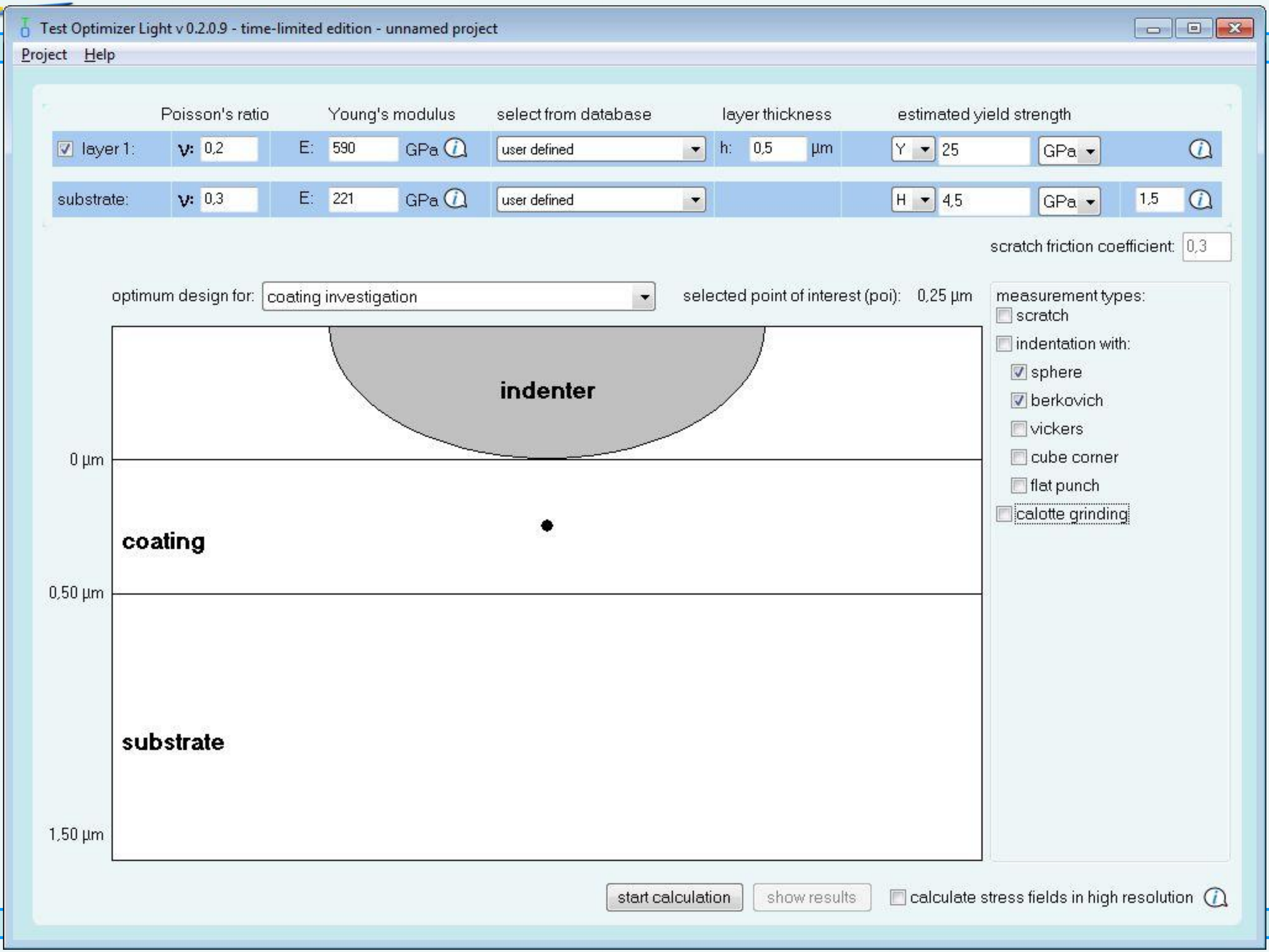


The point of interest is at a different location than you selected at the beginning. This adjustment was necessary in order to avoid plastic deformation in the underlying layers/substrate. The following experiments are affected:
sphere, Berkovich indenter

Your selected point of interest could not be reached with the following indenter types:
sphere, Berkovich indenter
The closest solution has been calculated instead.
Attention: The effect is of principle physical nature and has nothing to do with limits regarding our software.
You will find more information at www.siomec.de/pubs/2013/003

www.siomec.de/pubs/2013/003

Test Optimizer



Test Optimizer Light v 0.2.0.9 - time-limited edition - unnamed project

	Poisson's ratio	Young's modulus	select from database	layer thickness	estimated yield strength
☑ layer 1:	ν: 0,2	E: 590 GPa	user defined	h: 0,5 µm	Y 25 GPa
substrate:	ν: 0,3	E: 221 GPa	user defined		H 4,5 GPa 1,5

scratch friction coefficient: 0,3

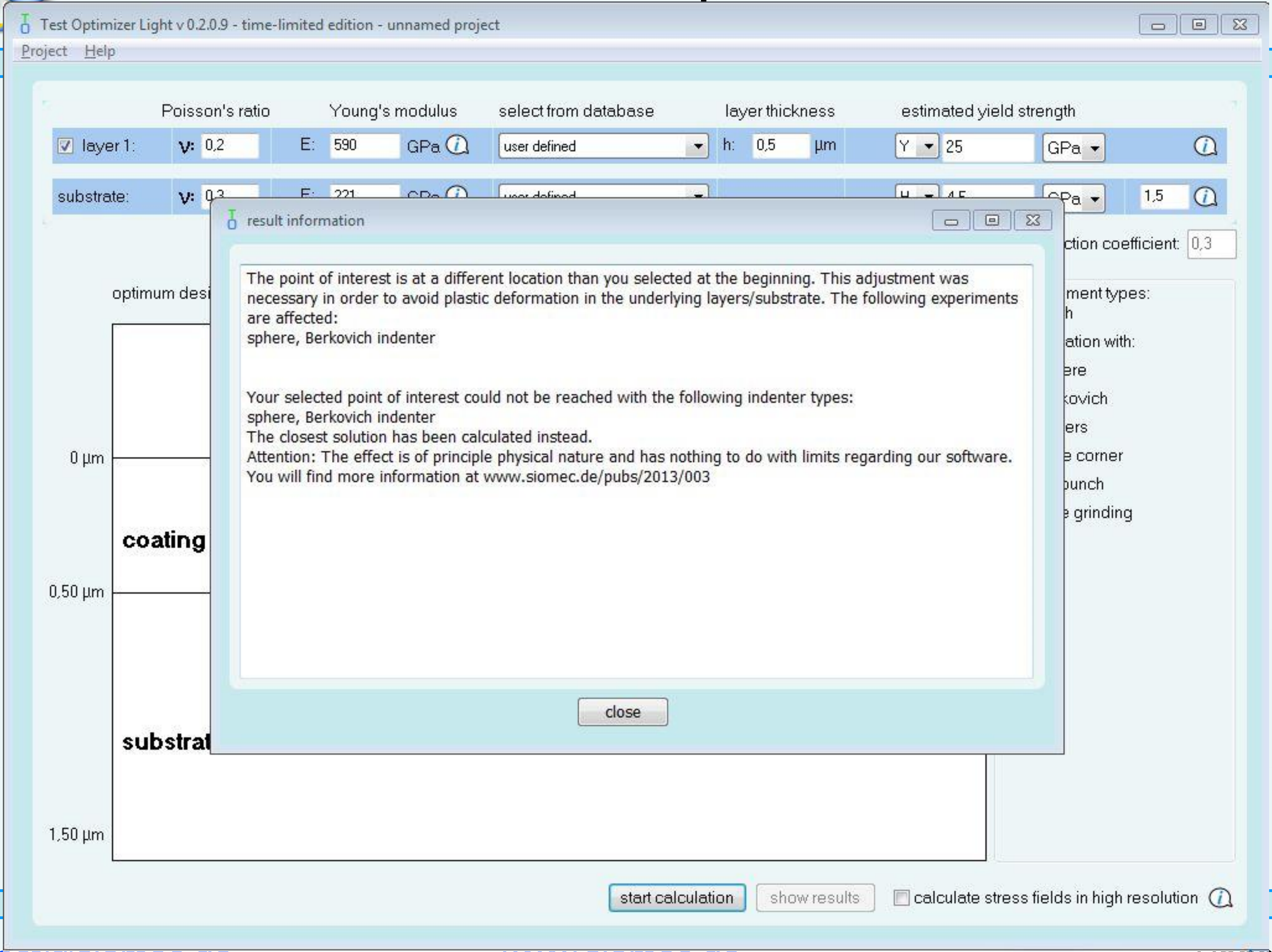
optimum design for: coating investigation

selected point of interest (poi): 0,25 µm

measurement types:
- ☐ scratch
- ☐ indentation with:
  - ☑ sphere
  - ☑ berkovich
  - ☐ vickers
  - ☐ cube corner
  - ☐ flat punch
- ☐ calotte grinding

☐ calculate stress fields in high resolution

Test Optimizer



Test Optimizer Light v 0.2.0.9 - time-limited edition - unnamed project

	Poisson's ratio	Young's modulus	select from database	layer thickness	estimated yield strength	
☑ layer 1:	ν: 0,2	E: 590 GPa	user defined	h: 0,5 µm	Y 25 GPa	
substrate:	ν: 0,3	E: 221 GPa	user defined		H 4,5 GPa	1,5

ction coefficient: 0,3

result information

The point of interest is at a different location than you selected at the beginning. This adjustment was necessary in order to avoid plastic deformation in the underlying layers/substrate. The following experiments are affected:
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Your selected point of interest could not be reached with the following indenter types:
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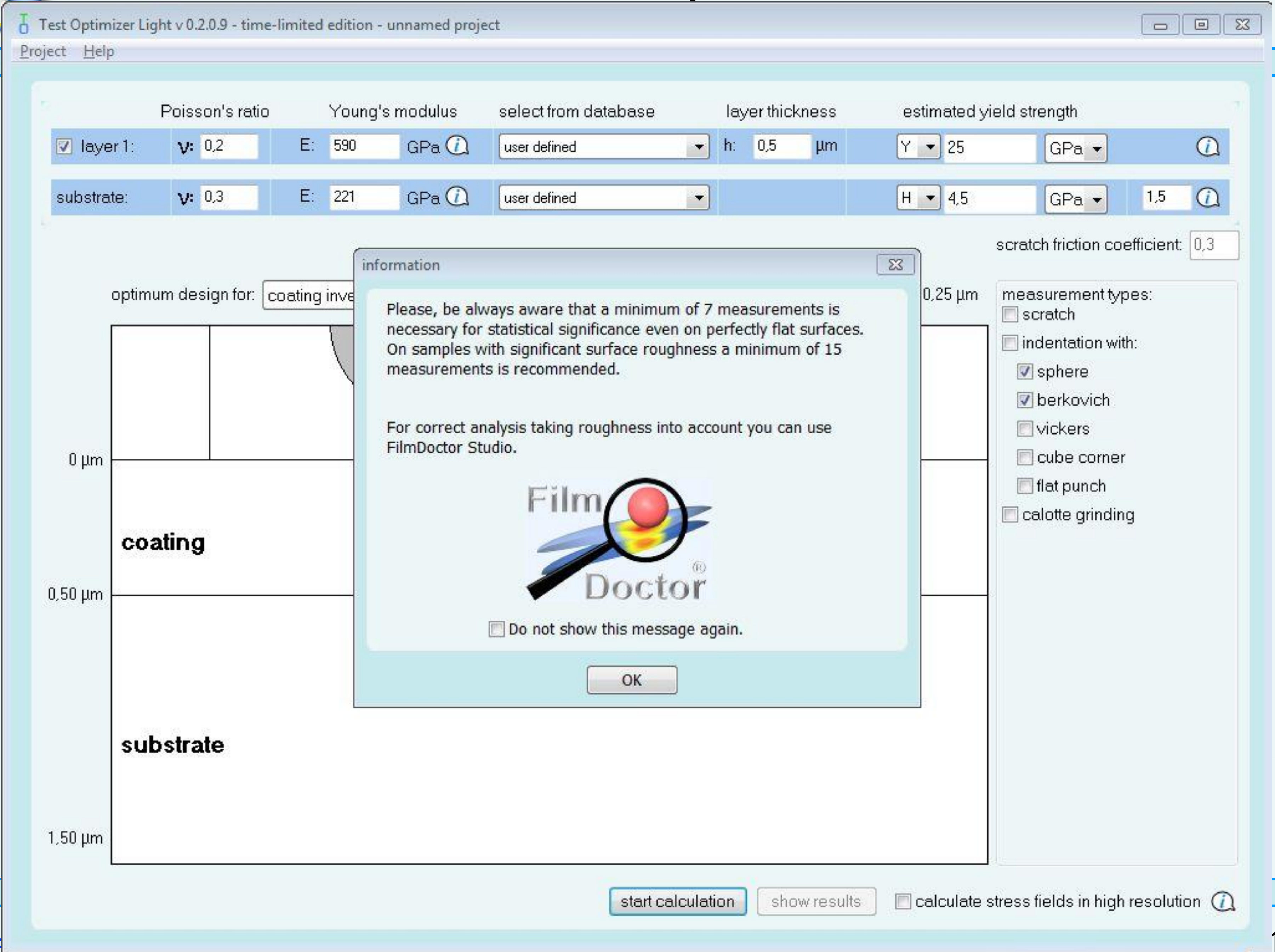
☐ calculate stress fields in high resolution

Test Optimizer



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	Poisson's ratio	Young's modulus	select from database	layer thickness	estimated yield strength	
☑ layer 1:	ν: 0,2	E: 590 GPa	user defined	h: 0,5 µm	Y 25 GPa	
substrate:	ν: 0,3	E: 221 GPa	user defined		H 4,5 GPa	1,5

scratch friction coefficient: 0,3

optimum design for: coating inve

0,25 µm

measurement types:
☐ scratch
☐ indentation with:
  ☑ sphere
  ☑ berkovich
  ☐ vickers
  ☐ cube corner
  ☐ flat punch
☐ calotte grinding

information

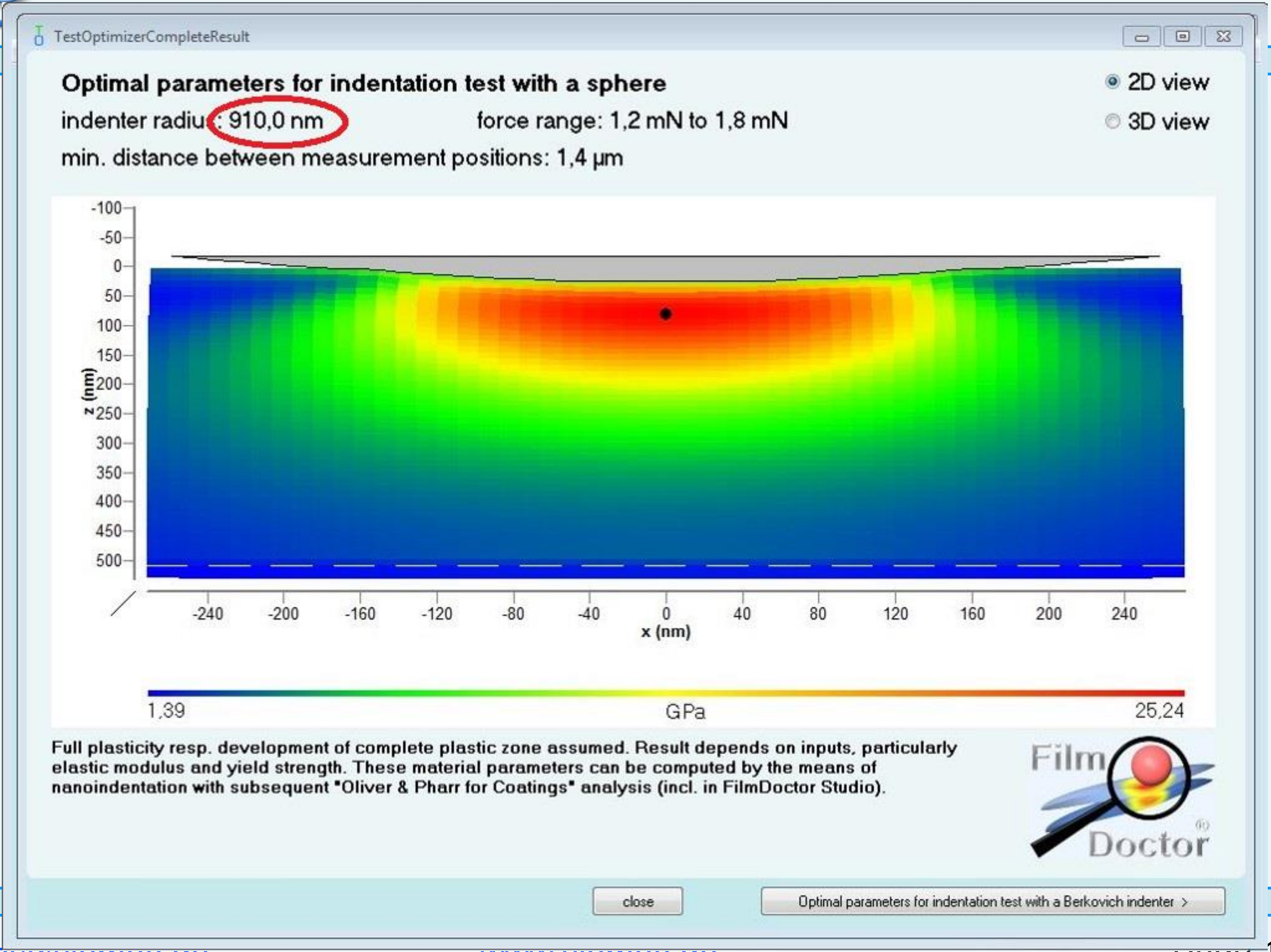
Please, be always aware that a minimum of 7 measurements is necessary for statistical significance even on perfectly flat surfaces. On samples with significant surface roughness a minimum of 15 measurements is recommended.

For correct analysis taking roughness into account you can use FilmDoctor Studio.

☐ Do not show this message again.

☐ calculate stress fields in high resolution

Test Optimizer



TestOptimizerCompleteResult

Optimal parameters for indentation test with a sphere

indenter radius: 910,0 nm force range: 1,2 mN to 1,8 mN

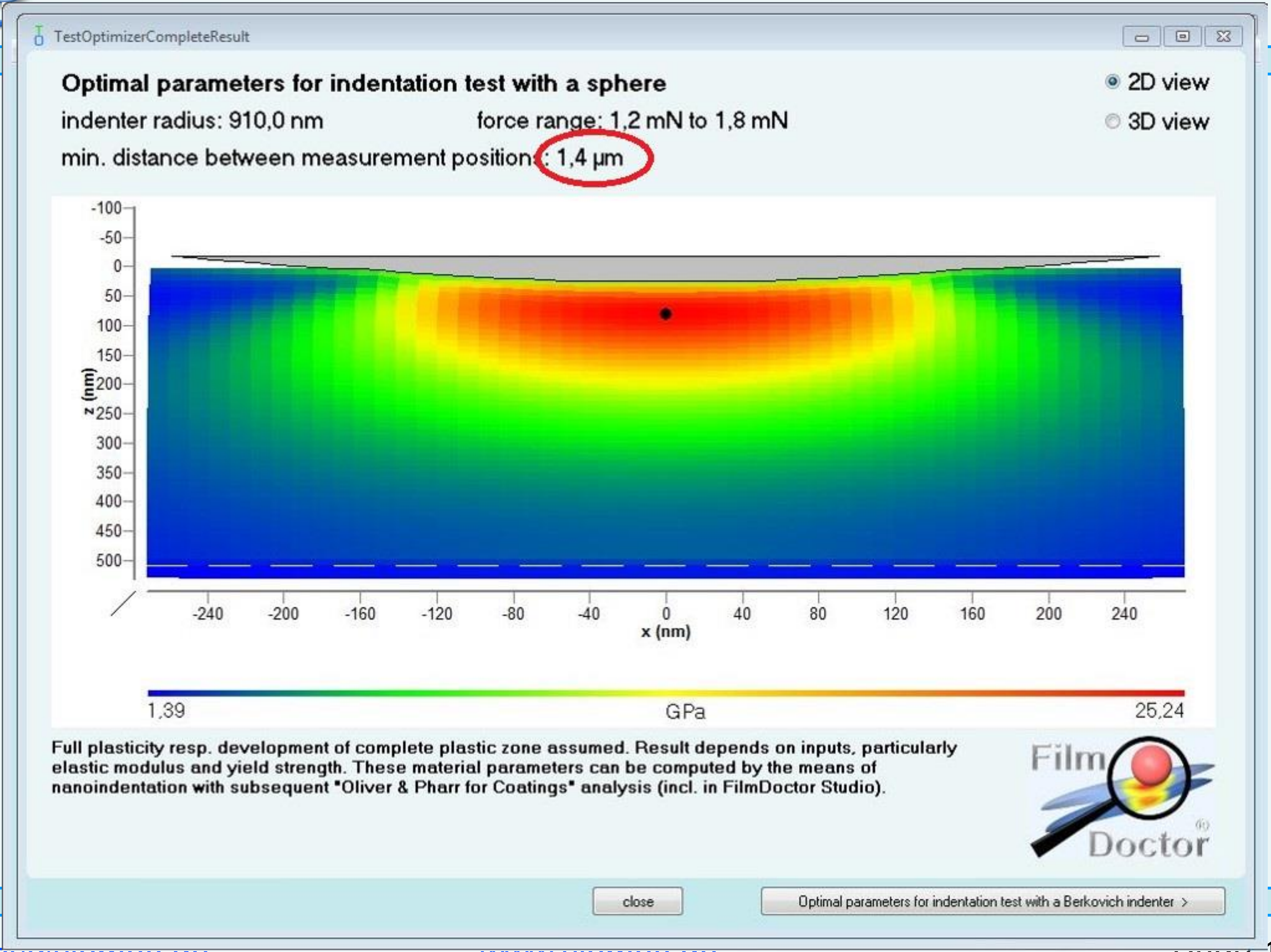
min. distance between measurement positions: 1,4 µm

● 2D view

○ 3D view

Full plasticity resp. development of complete plastic zone assumed. Result depends on inputs, particularly elastic modulus and yield strength. These material parameters can be computed by the means of nanoindentation with subsequent "Oliver & Pharr for Coatings" analysis (incl. in FilmDoctor Studio).

Test Optimizer



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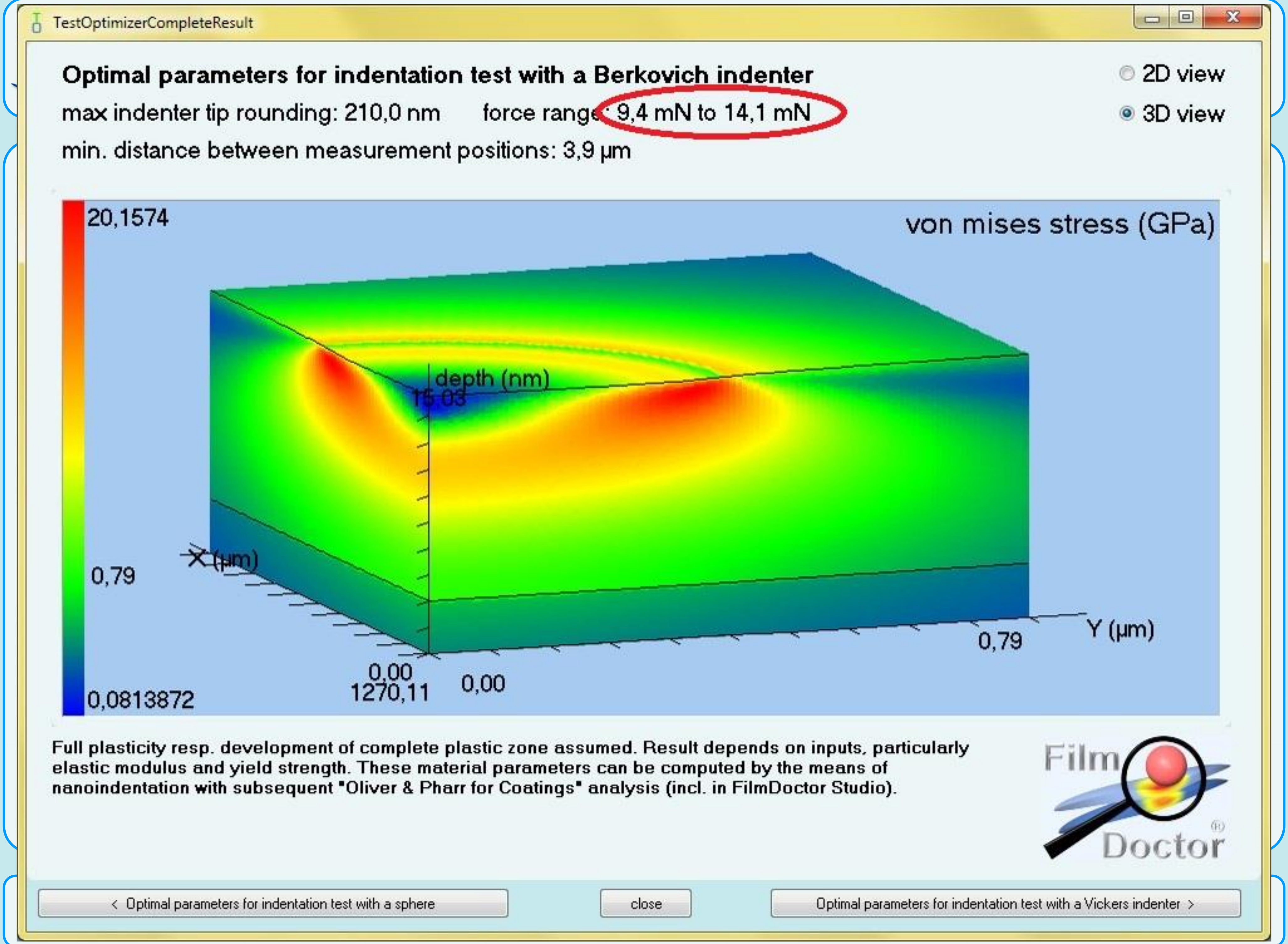


Optimal parameters for indentation test with a Berkovich indenter

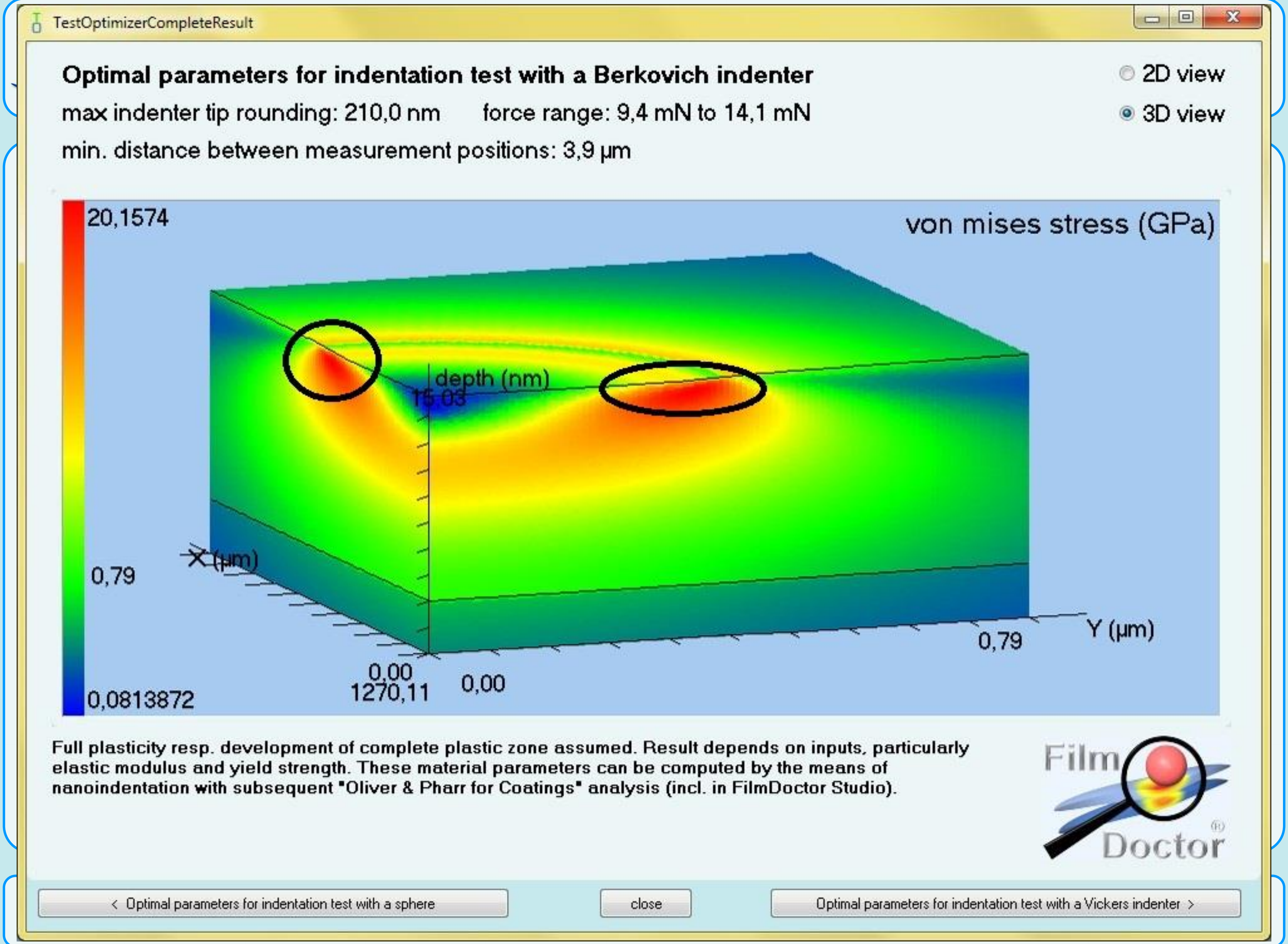
max indenter tip rounding: 210,0 nm force range: 9,4 mN to 14,1 mN

min. distance between measurement positions: 3,9 µm

○ 2D view

● 3D view

Full plasticity resp. development of complete plastic zone assumed. Result depends on inputs, particularly elastic modulus and yield strength. These material parameters can be computed by the means of nanoindentation with subsequent "Oliver & Pharr for Coatings" analysis (incl. in FilmDoctor Studio).



TestOptimizerCompleteResult

Optimal parameters for indentation test with a Berkovich indenter

max indenter tip rounding: 210,0 nm force range: 9,4 mN to 14,1 mN

min. distance between measurement positions: 3,9 µm

○ 2D view

● 3D view

Full plasticity resp. development of complete plastic zone assumed. Result depends on inputs, particularly elastic modulus and yield strength. These material parameters can be computed by the means of nanoindentation with subsequent "Oliver & Pharr for Coatings" analysis (incl. in FilmDoctor Studio).

Determination of material parameters

Determination of physical parameters by nanoindentation

Standard analysis method (Oliver & Pharr) gives you only effective values for Young's modulus and Hardness

For measurement analysis needed:

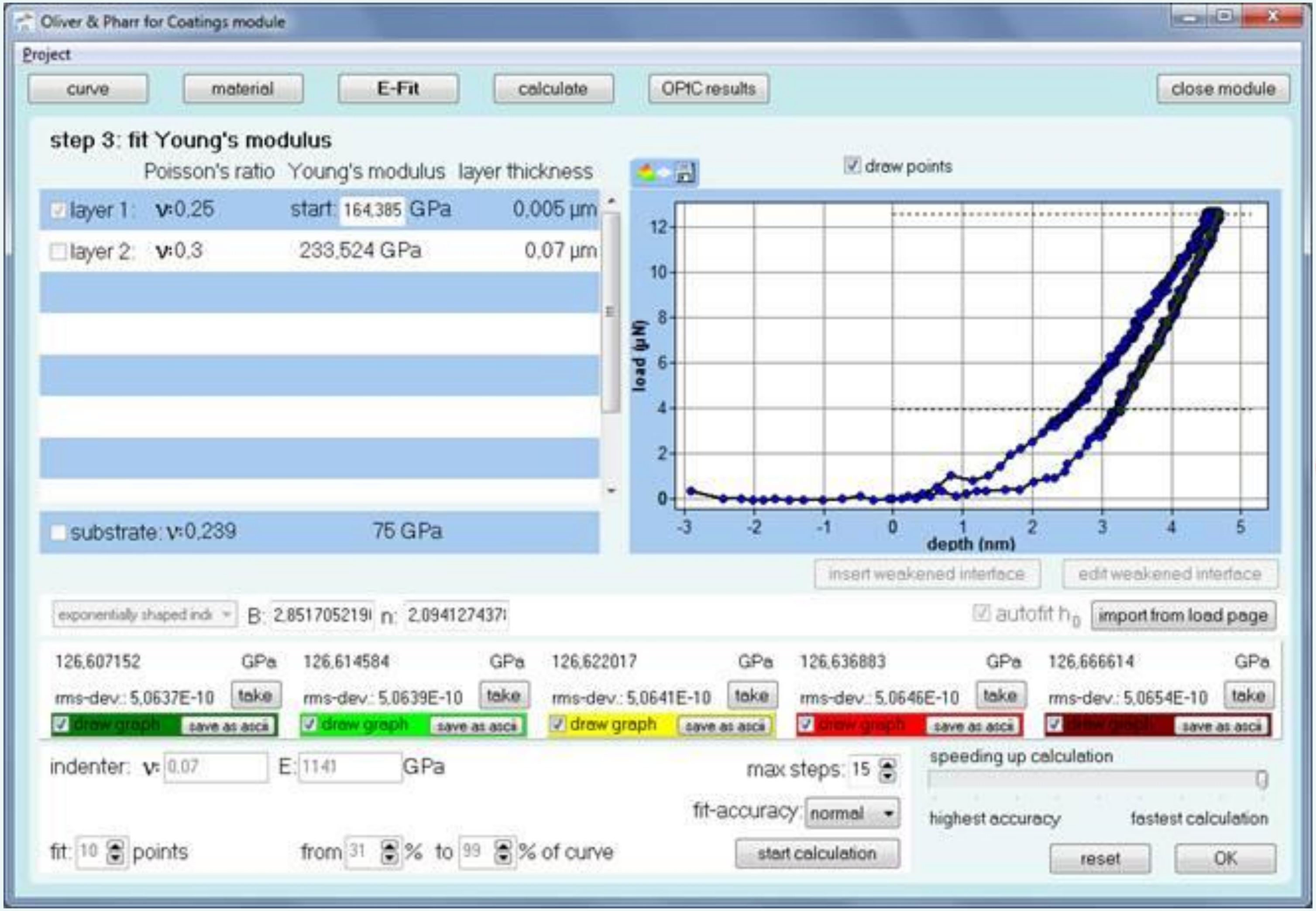
- physical parameters like e.g. Yield Strength
- true material parameters for inhomogeneous materials

•Therefore SIO has extended the standard method → Oliver & Pharr for Coatings

- for inhomogeneous materials (layered, gradients, …)
- for time depending material behaviour (viscose, creep (HT))

O&PfC

Getting basic generic parameters example 1



step 3: fit Young's modulus

	Poisson's ratio	Young's modulus	layer thickness
☑ layer 1:	ν:0,25	start: 164,385 GPa	0,005 µm
☐ layer 2:	ν:0,3	233,524 GPa	0,07 µm
☐ substrate:	ν:0,239	75 GPa	

exponentially shaped ind. B: 2,851705219 n: 2,094127437

☑ autofit h_0

126,607152 GPa	126,614584 GPa	126,622017 GPa	126,636883 GPa	126,666614 GPa
rms-dev.: 5,0637E-10	rms-dev.: 5,0639E-10	rms-dev.: 5,0641E-10	rms-dev.: 5,0646E-10	rms-dev.: 5,0654E-10
☑ draw graph	☑ draw graph	☑ draw graph	☑ draw graph	☑ draw graph

indenter: ν: 0,07 E: 1141 GPa

max steps: 15

fit-accuracy: normal

speeding up calculation: highest accuracy – fastest calculation

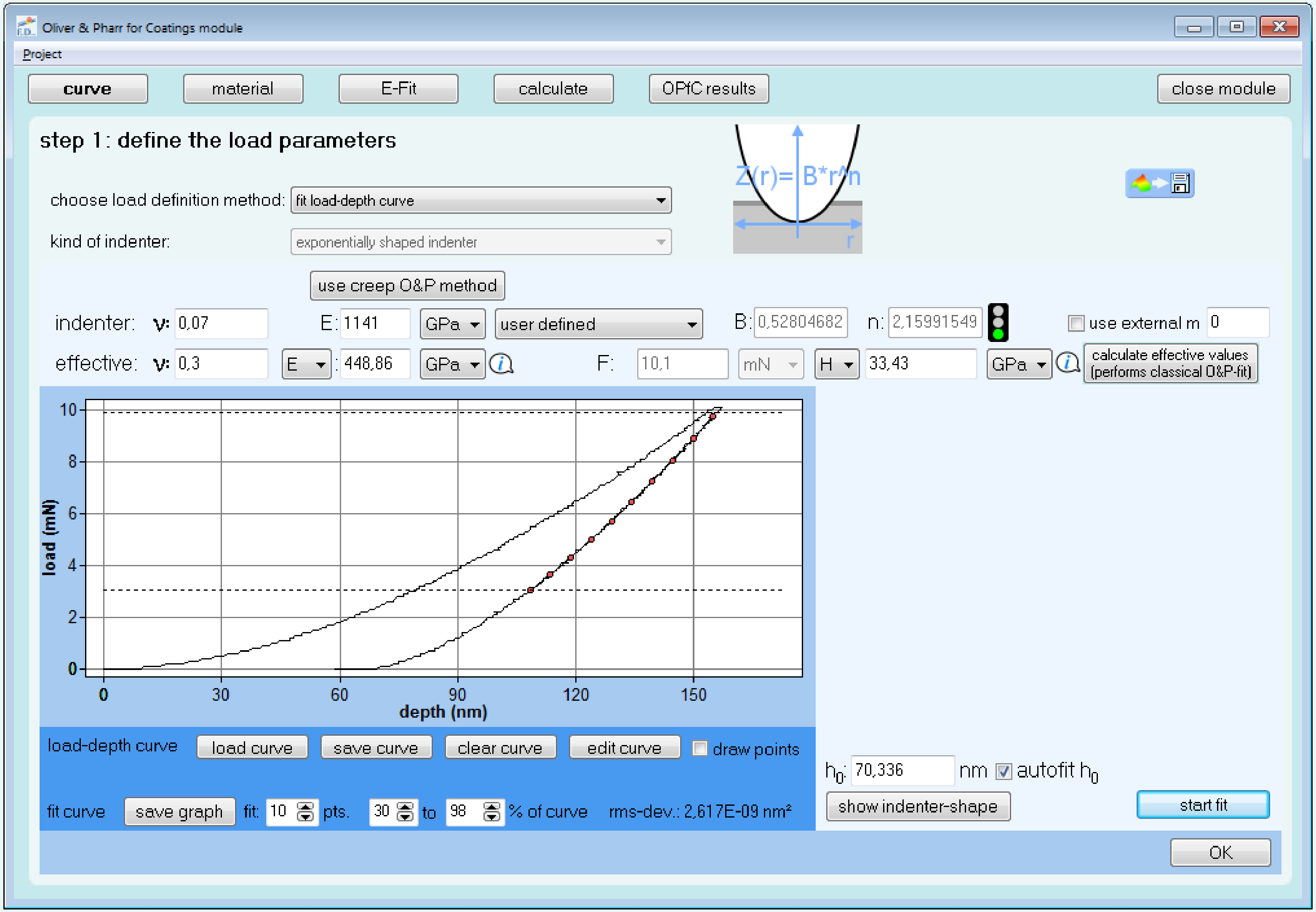
fit: 10 points from 31 % to 99 % of curve

O&P method for Coatings, gradients etc.

5nm DLC
70nm IL
SI

1/10 rule can‘t be applied

Getting basic generic parameters example 2



step 1: define the load parameters

choose load definition method: fit load-depth curve

kind of indenter: exponentially shaped indenter

indenter: ν: 0,07 E: 1141 GPa user defined B: 0,52804682 n: 2,15991549 ☐ use external m 0

effective: ν: 0,3 E: 448,86 GPa F: 10,1 mN H 33,43 GPa

load-depth curve ☐ draw points

fit curve fit: 10 pts. 30 to 98 % of curve rms-dev.: 2,617E-09 nm²

h_0: 70,336 nm ☑ autofit h_0

Indentation depth:

ca. 160 nm

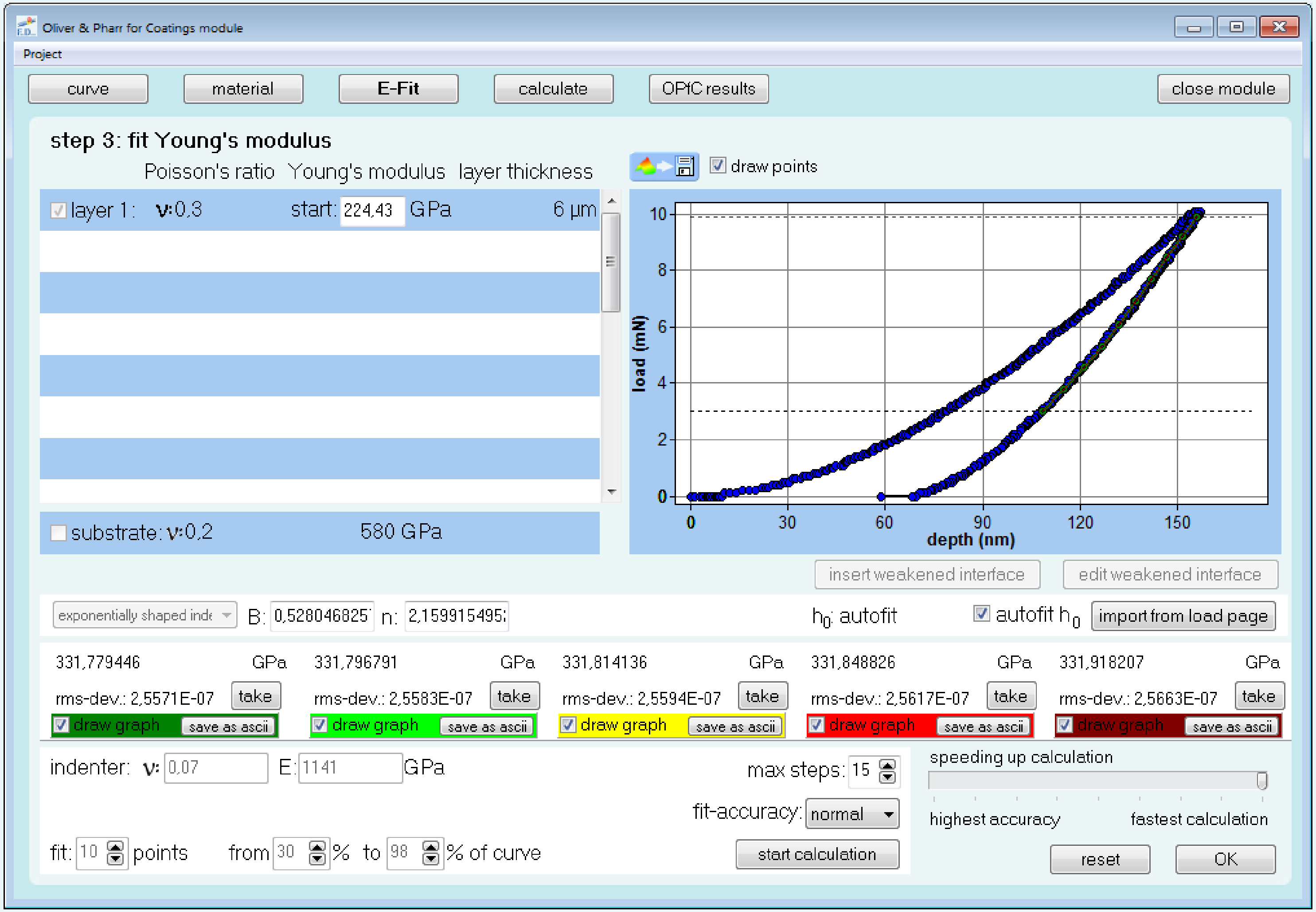
Thickness: 6 µm

Substrate: WC
E=580 GPa

Classic O&P result:

E=448,6 GPa

Getting basic generic parameters example 2



step 3: fit Young's modulus

	Poisson's ratio	Young's modulus	layer thickness
☑ layer 1:	ν:0,3	start: 224,43 GPa	6 µm
☐ substrate:	ν:0,2	580 GPa	

exponentially shaped inde B: 0,528046825 n: 2,159915495 h₀: autofit ☑ autofit h_0

331,779446 GPa	331,796791 GPa	331,814136 GPa	331,848826 GPa	331,918207 GPa
rms-dev.: 2,5571E-07	rms-dev.: 2,5583E-07	rms-dev.: 2,5594E-07	rms-dev.: 2,5617E-07	rms-dev.: 2,5663E-07

indenter: ν: 0,07 E: 1141 GPa

max steps: 15

fit-accuracy: normal

speeding up calculation: highest accuracy – fastest calculation

fit: 10 points from 30 % to 98 % of curve

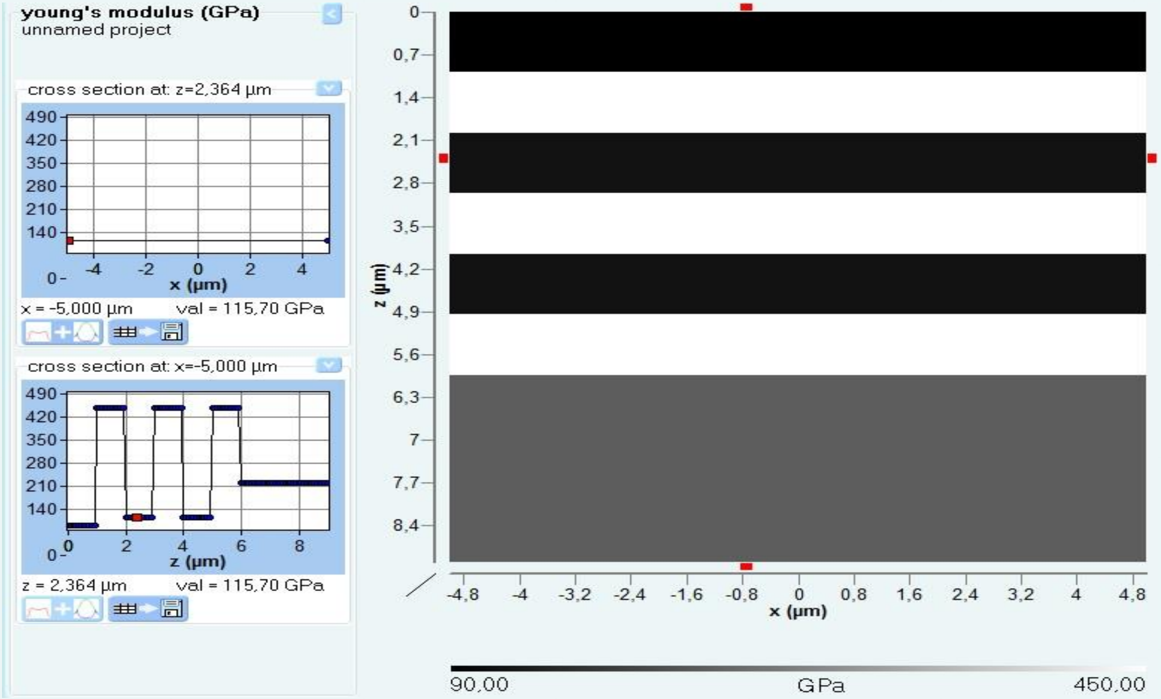
O&P method for Coatings, gradients etc.:

Fitting correct coating Young's modulus:

E=331,78 GPa

New idea -> SIO Calotte Module

Standard OPfC often not applicable to complex structures



young's modulus (GPa)
unnamed project

cross section at: z=2,364 µm

x = -5,000 µm val = 115,70 GPa

cross section at: x=-5,000 µm

z = 2,364 µm val = 115,70 GPa

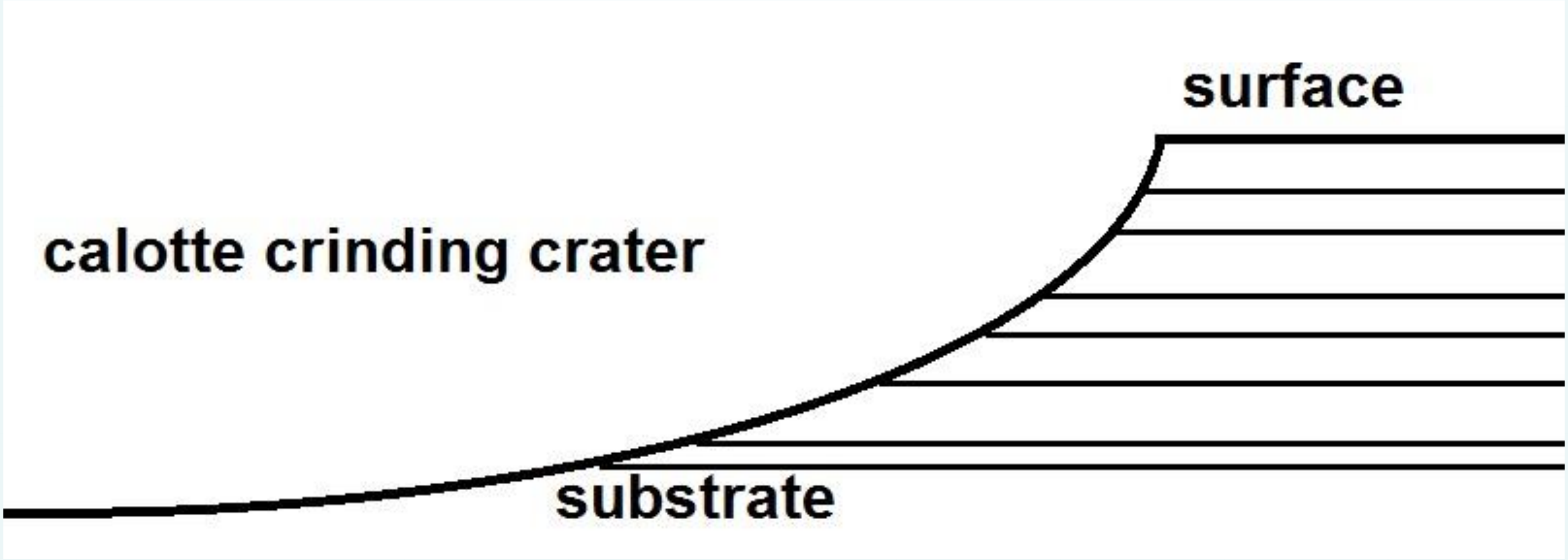
Need to stop the production process after each layer to perform the measurement

Creating a new layer could change the properties of existing layers
(e.g. introducing stresses, creating a mixed zone)

Calotte grinding

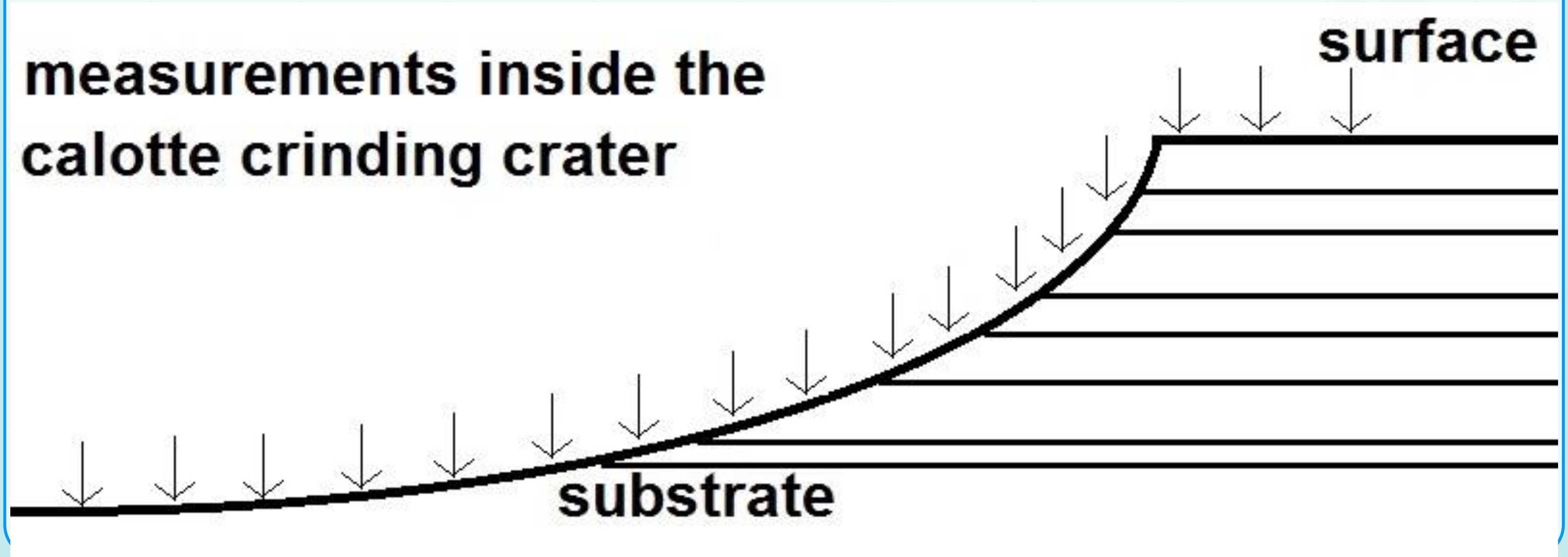
Calotte grinding is an easy and cheap technology

Normally used to determine the thicknesses of all layers

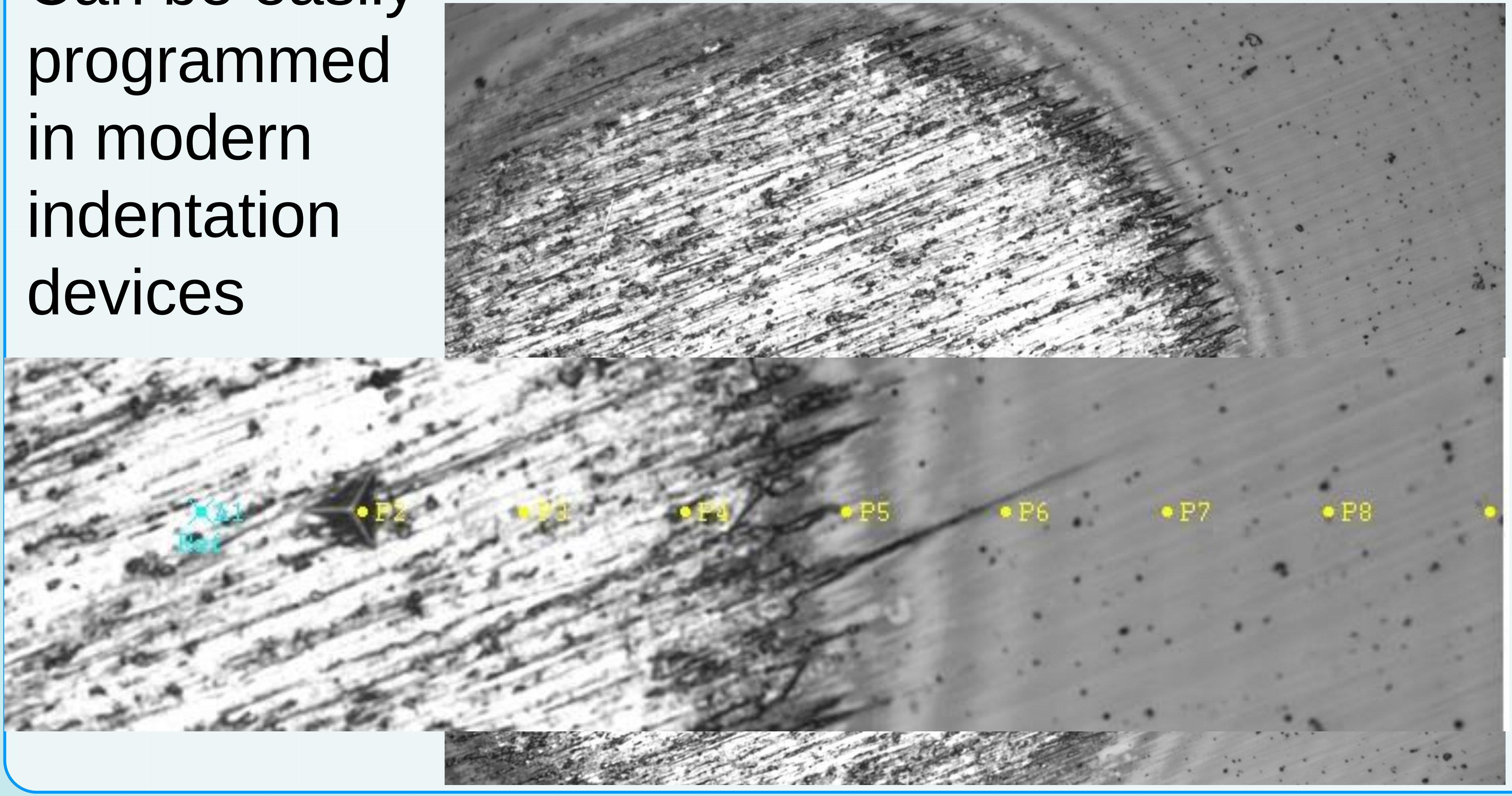


SIO Calotte Module

Now perform a series of indentation measurements inside and outside of the crater

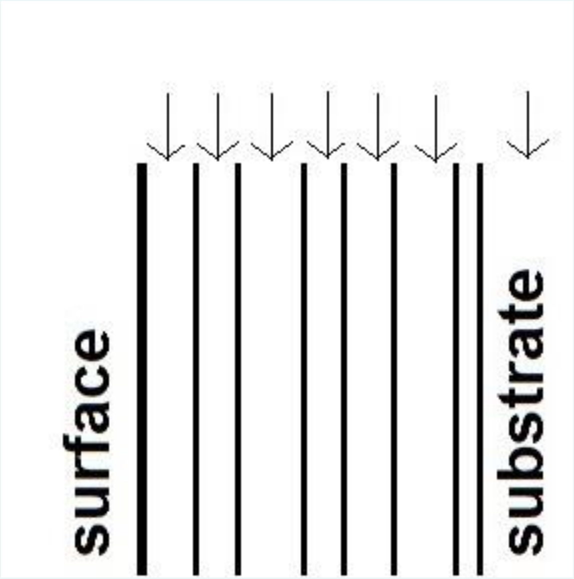


Can be easily programmed in modern indentation devices



Why calotte grinding?

- easy and cheap, already available in most labs
- creates a huge area which could be used for indentation measurements compared to vertical cuts



How it works in the software

- First define the material structure

step 1: select your material

select all

☐ gradient ☐ viscous

	Poisson's ratio	Young's modulus		select from database	layer thickness	
☑ layer 1:	ν: 0,25	E: 590	GPa	TiN (E:590)	h: 1	µm
☑ layer 2:	ν: 0,22	E: 450	GPa	Titanium Nitride (TiN) (E:450)	h: 0,5	µm
☑ layer 3:	ν: 0,21	E: 248	GPa	Chromium (Cr) (E:248)	h: 3	µm
☑ layer 4:	ν: 0,24	E: 691	GPa	Tungsten carbide (WC) (E:691	h: 0,1	µm
☑ layer 5:	ν: 0,343	E: 129,8	GPa	Copper (Cu), hard (E:129,8)	h: 2	µm
☑ layer 6:	ν: 0,22	E: 450	GPa	Titanium Nitride (TiN) (E:450)	h: 1,1	µm

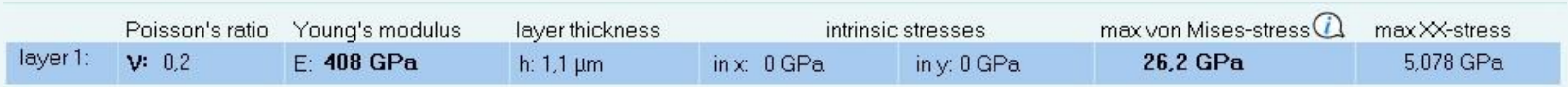
How it works in the software

- First define the material structure
- Secondly load the measurement data and define the x or z position of each measurement

step 2: load measured data

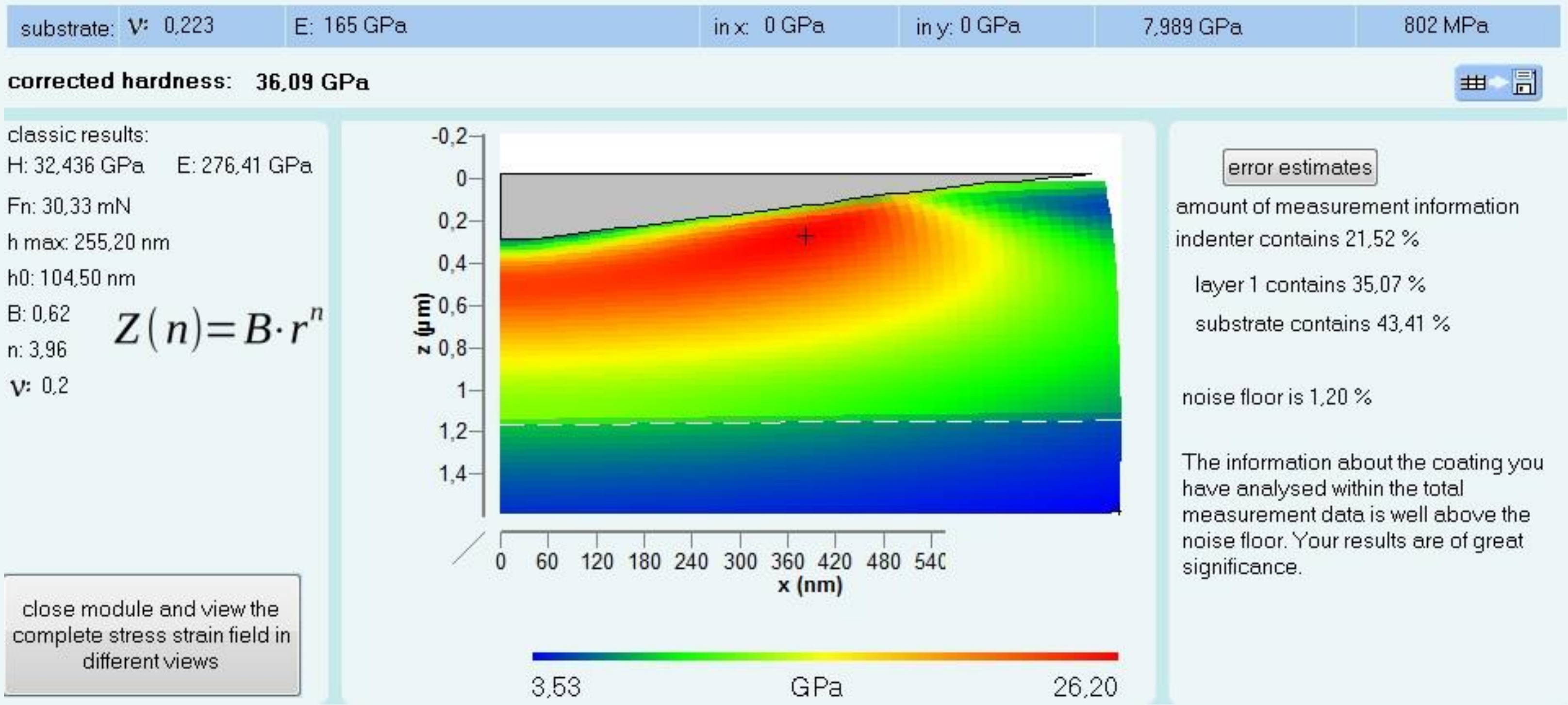
filename	Reduced modulus	C
distance_10mue.fdop	212,20	1,03
distance_20mue.fdop	217,50	0,89
distance_30mue.fdop	238,40	0,77
distance_40mue.fdop	255,30	0,62
distance_50mue.fdop	246,00	0,62

Start the evaluation and look at each result



	Poisson's ratio	Young's modulus	layer thickness	intrinsic stresses		max von Mises-stress	max XX-stress
layer 1:	**ν:** 0,2	E: **408 GPa**	h: 1,1 µm	in x: 0 GPa	in y: 0 GPa	**26,2 GPa**	5,078 GPa

uses Oliver & Pharr for Coatings procedure



substrate:	**ν:** 0,223	E: 165 GPa		in x: 0 GPa	in y: 0 GPa	7,989 GPa	802 MPa

corrected hardness: 36,09 GPa

classic results:
H: 32,436 GPa E: 276,41 GPa
Fn: 30,33 mN
h max: 255,20 nm
h0: 104,50 nm
B: 0,62
n: 3,96
ν: 0,2

$$Z(n)=B\cdot r^{n}$$

amount of measurement information
indenter contains 21,52 %
layer 1 contains 35,07 %
substrate contains 43,41 %

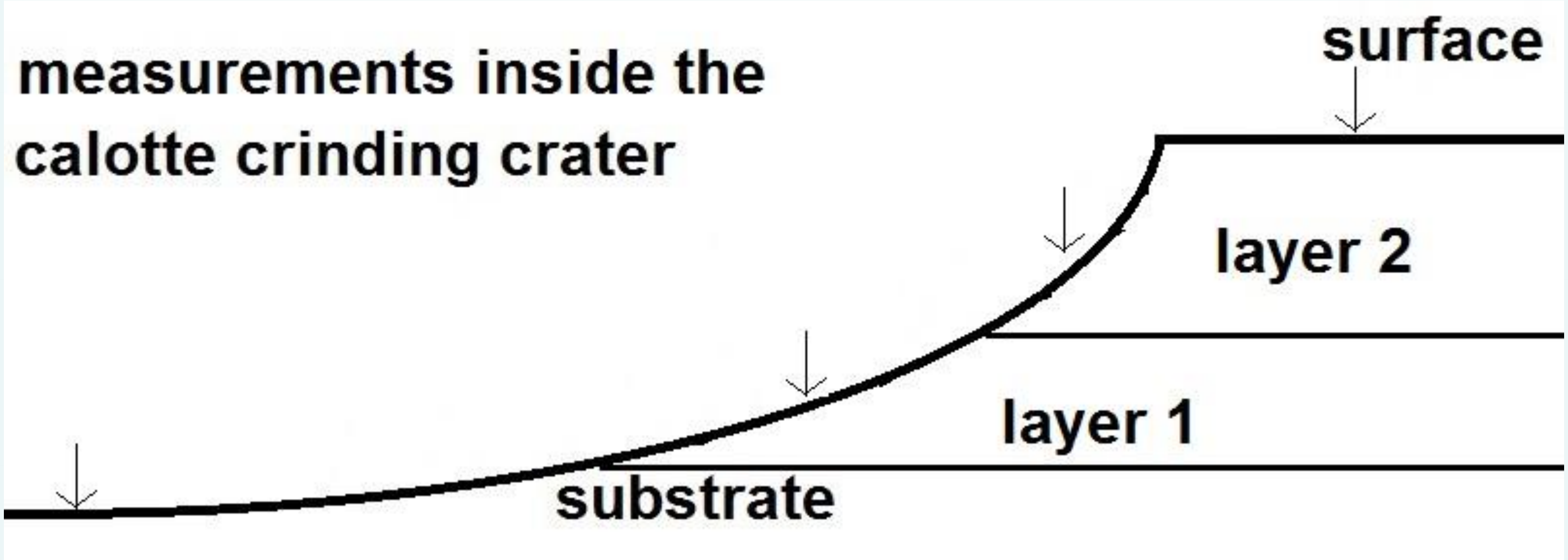
noise floor is 1,20 %

The information about the coating you have analysed within the total measurement data is well above the noise floor. Your results are of great significance.

SIO Calotte Module

How it works in detail

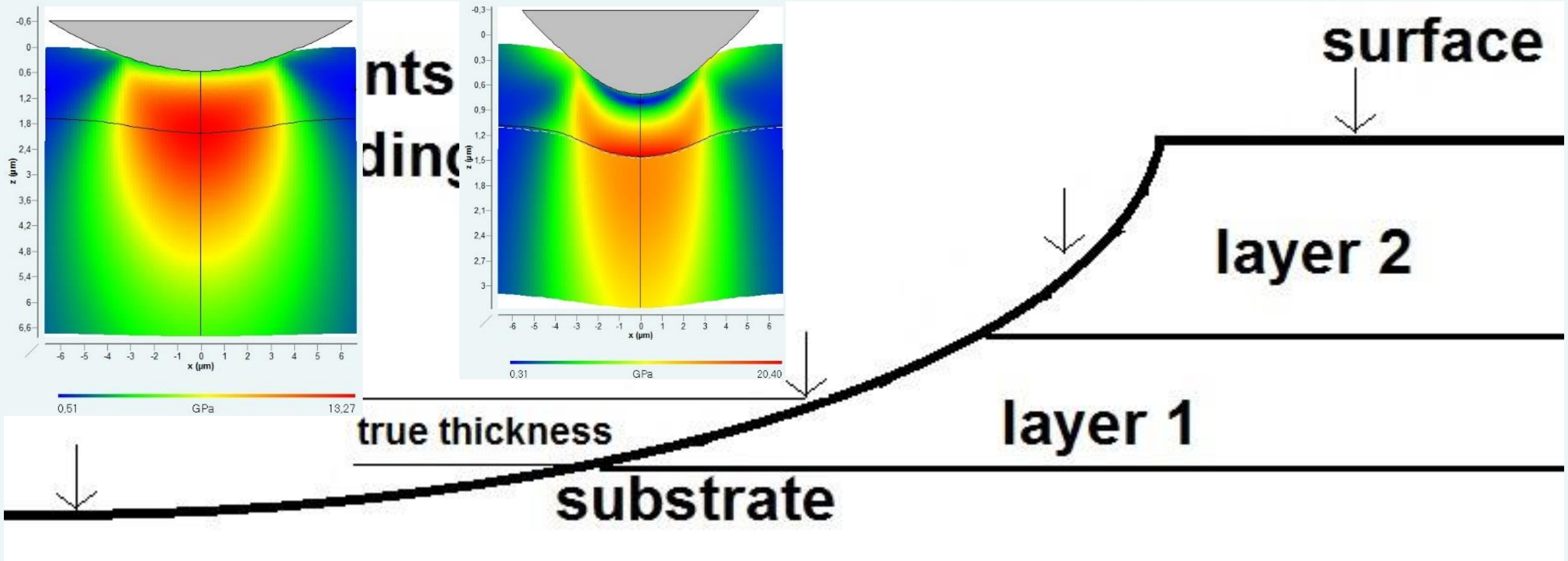
- The analysis is performed from the inside to the outside
- Simple example: 2 layer system with 2 µm interlayer (layer 1) and 3 µm top layer (layer 2)
- 4 measurements (one in substrate, one in layer 1 and two in layer 2)



SIO Calotte Module

How it works in detail

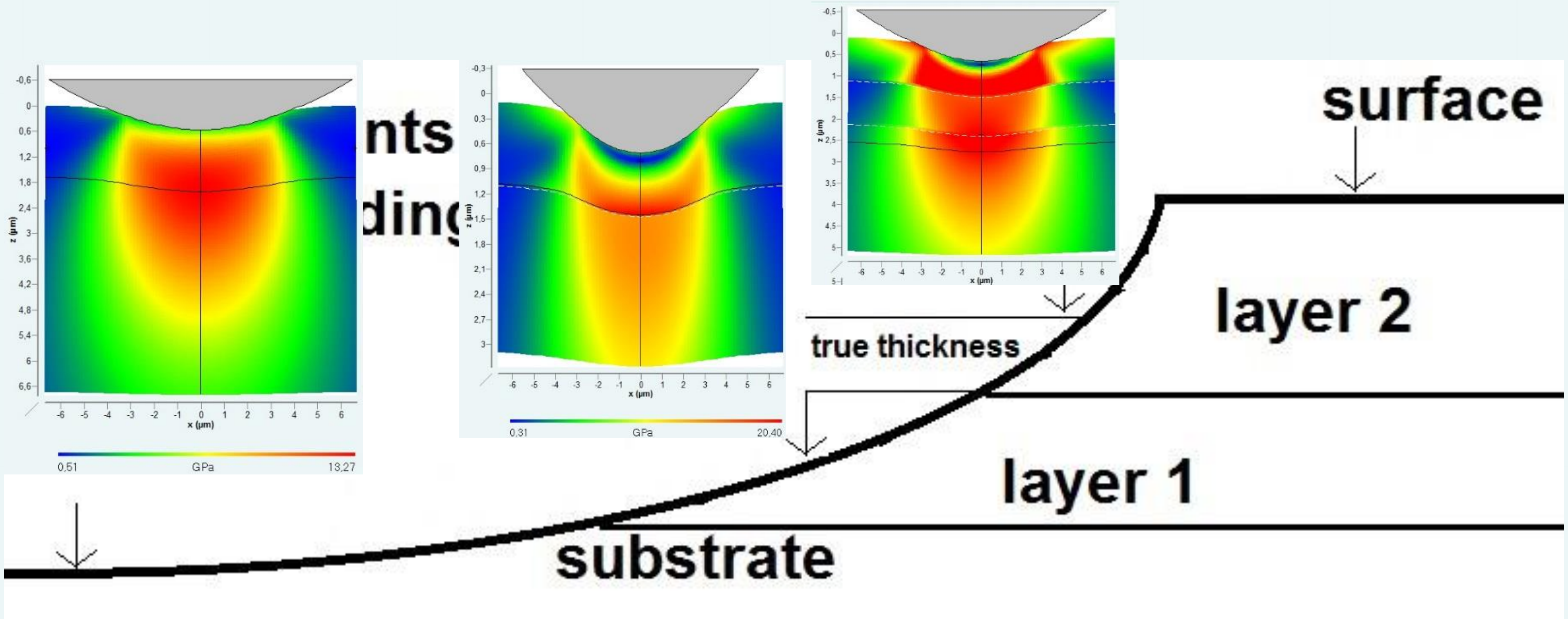
- Analysis I: substrate measurement → E_s
- Analysis II: 1 µm thick layer 1 → E_1



SIO Calotte Module

How it works in detail

- Analysis III: 1 µm thick layer 2 → E_{21}
- Analysis IV: 3 µm thick layer 2 → E_{22}
- If E_{21} and E_{22} differs significantly check homogeneity!



Conclusions

Combination of Calotte Grinding and Indentation Testing offers a new way to extract the material parameters of multi-layer stacks

Gives you generic material parameters of each part and hints about the structure of each part

Both technologies are relatively cheap and easy to use

Visit booth #402 for more information

Thanks for your attention.