

View Inside Material Systems with Tribological Contact Experiments

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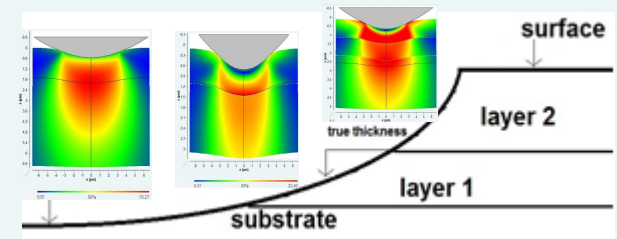
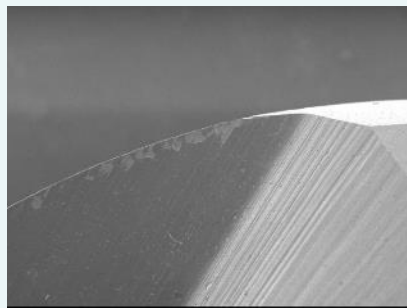
Tankow 2
18569 Ummanz / Rügen

Am Lauchberg 2
04838 Eilenburg

Germany



- Gather material parameters
 - Base idea and theory
 - Existing software modules
 - New software module for created extension





**Developed for project with
technical university of munich**



www.spp2013.tum.de

Indentation device from MicroMaterials



Theory

Extended Hertzian stress distribution

Effective indenter concept

Exponentially shaped indenter

Analytical models for stress-strain-field calculations

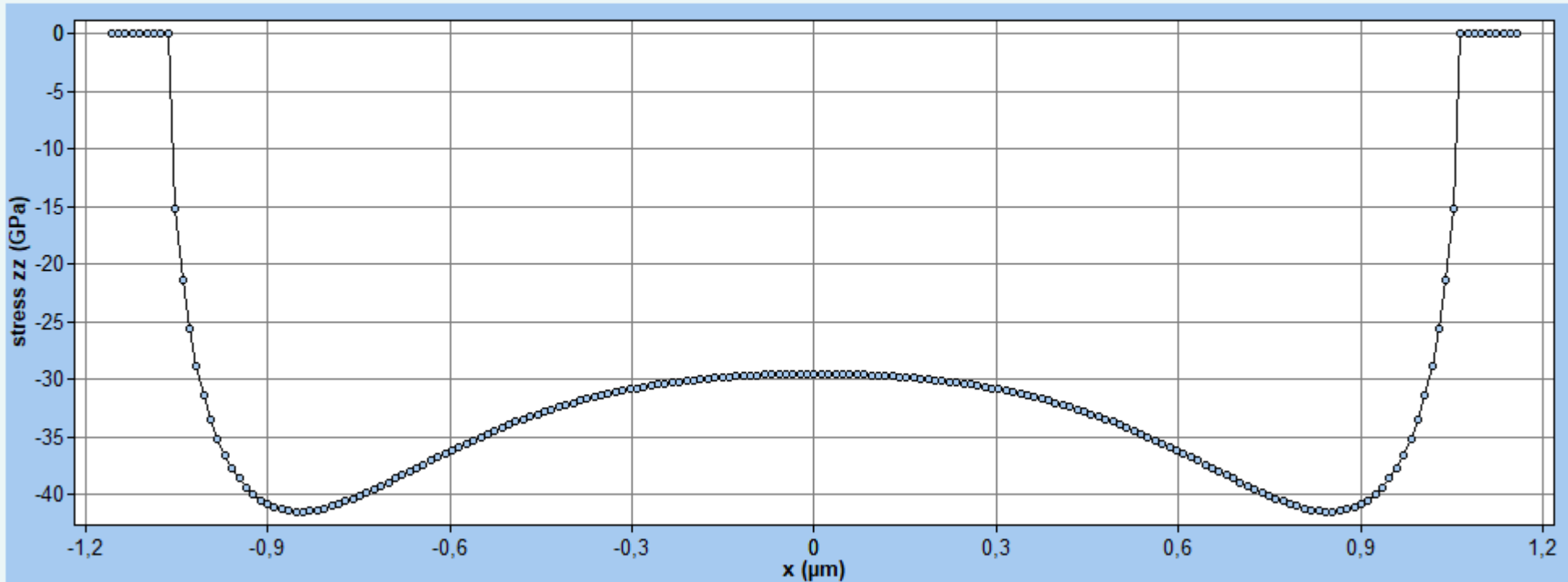
Extended Hertzian stress distribution

normal surface stress:

$$\sigma_{zz0}(r) = norm * \sum_{n=0}^N c_n r^n \sqrt{a^2 - r^2}, \quad n = 0, 2, 4, 6$$



F: mN μm



penetration depth: 176,172 nm

rigid indenter

c_0 :

c_2 :

c_4 :

c_6 :

OK

Extended Hertzian stress distribution

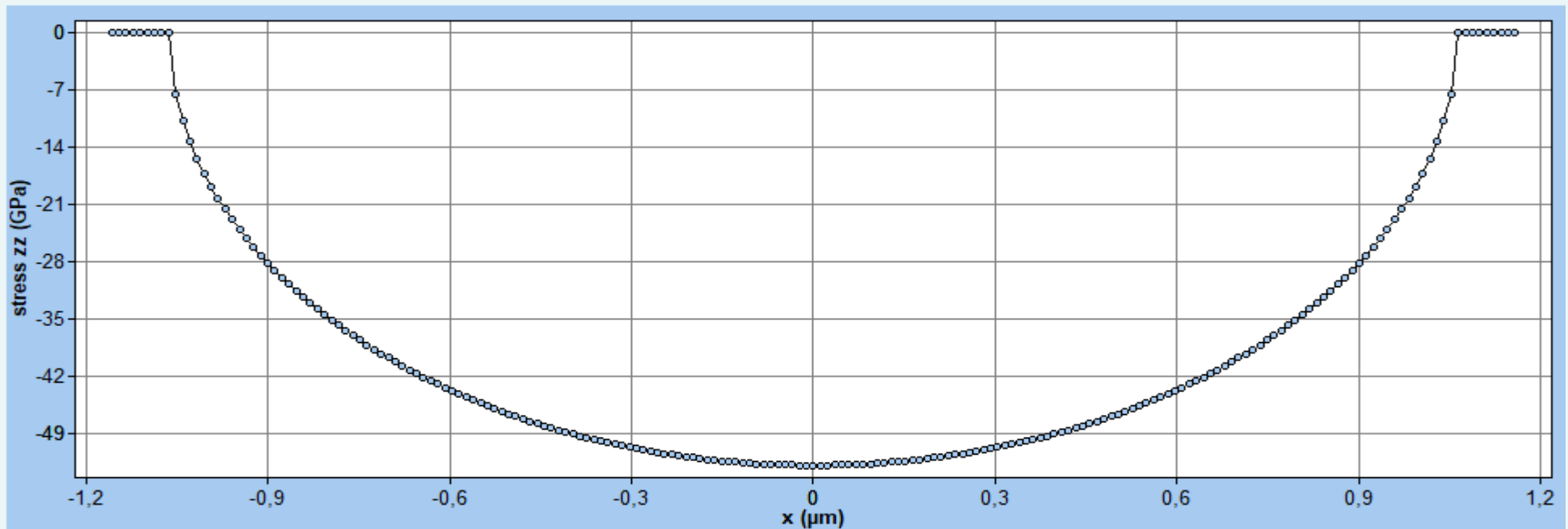
$C_0=1, C_2=C_4=C_6=0 \rightarrow$ Hertzian stress distribution

normal surface stress:

$$\sigma_{zz0}(r) = norm * \sum_{n=0}^N c_n r^n \sqrt{a^2 - r^2}, \quad n = 0, 2, 4, 6$$



F: mN



penetration depth: 202,789 nm

rigid indenter

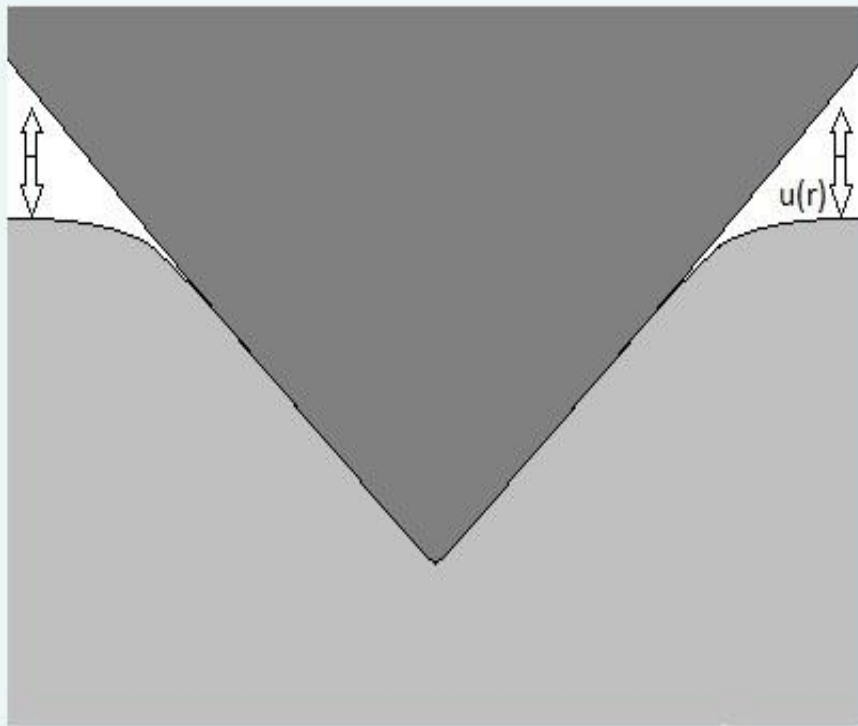
c_0 :

c_2 :

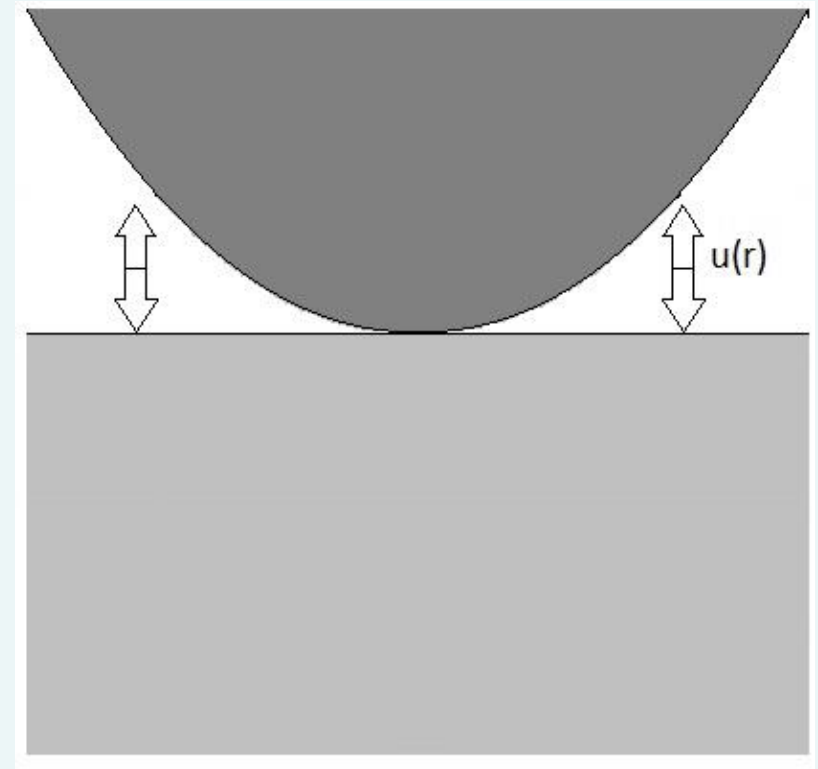
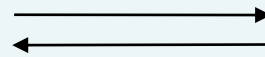
c_4 :

c_6 :

Effective indenter concept

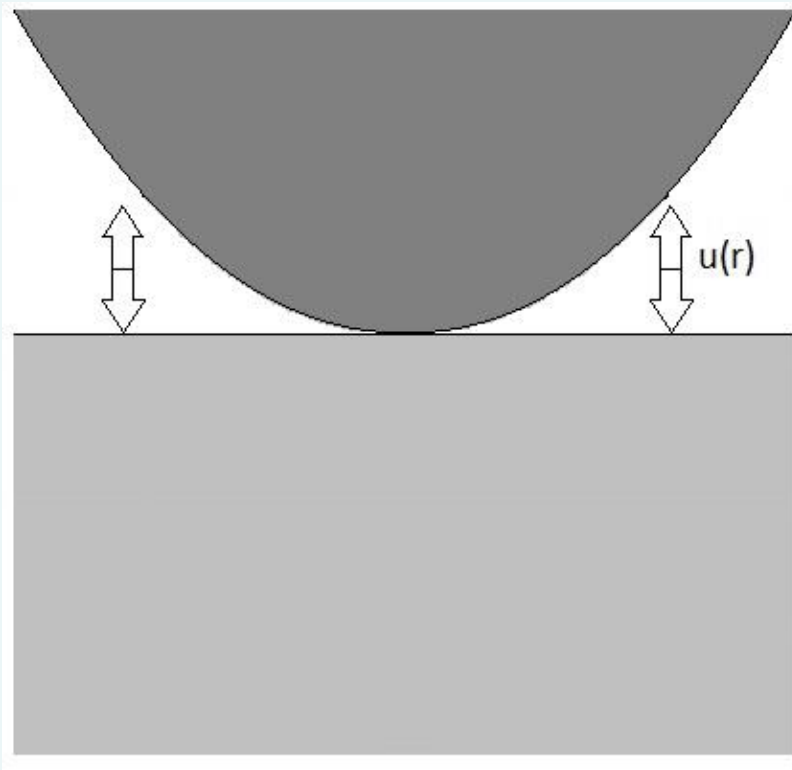


real surface

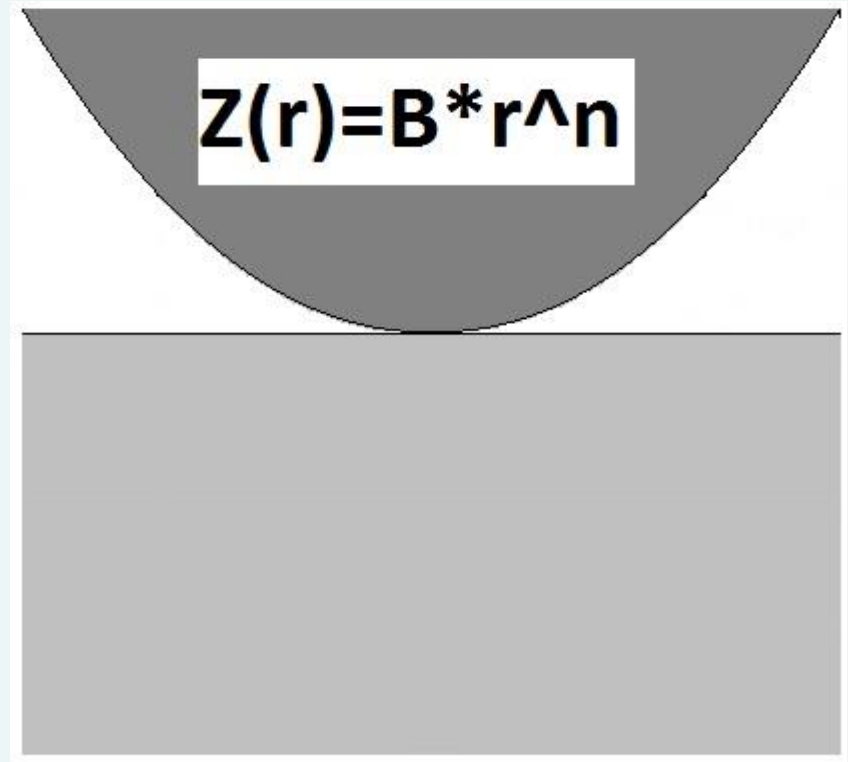
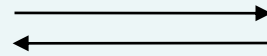


transformed surface

Exponentially shaped indenter



transformed surface



effective indenter

FilmDoctor Studio TUM v 0.9.5.0 - time-limited edition - 14 days left - project: intrinsic stress reference 9 indents

Project Tools Help

material curves **load** fit results close residual stress reference module

step 3: define the load parameters

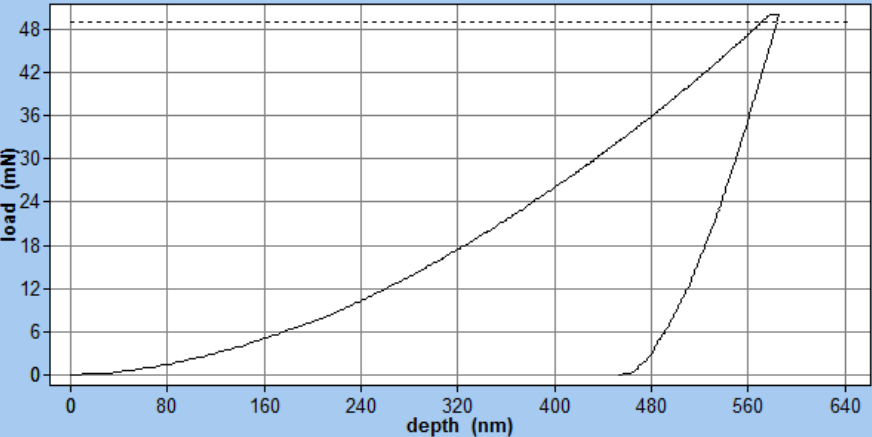
choose load definition method: fit load-depth curve

kind of indenter: exponentially shaped indenter

use effective half space accuracy: 10

indenter: ν : 0,07 E: 1,141 TPa user defined B: 1 n: 1

effective: ν : 0,3 Er: 189,482 GPa F: 50,0076903 mN H: 6,57649 GPa



load (mN)

depth (nm)

load-depth curve load curve save curve clear curve edit curve draw points

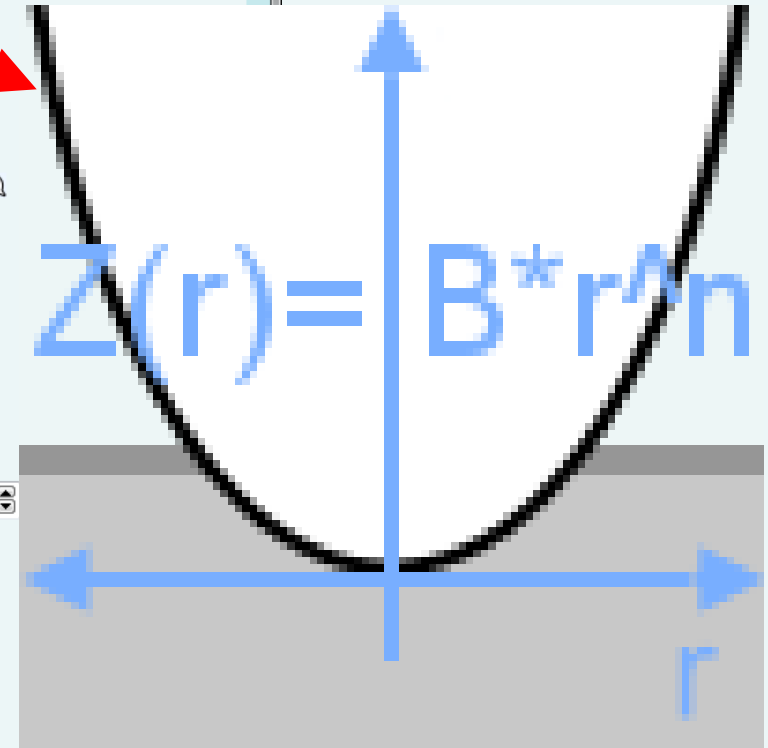
fit curve save graph fit: 10 pts. 40 to 98 % of curve rms-dev.:

$F = C(h - h_0)^m$ m

show values for curve 1 start series analysis

h_0 : 0 nm

OK



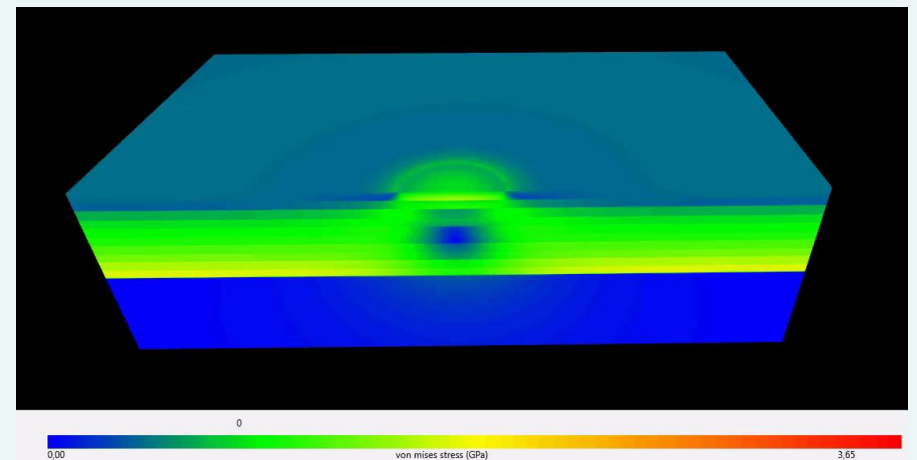
Analytical models

Optimize the materials and material combinations to increase application performance to reach certain goals (e.g. longer application life time of our composite structures)

No FEM system – closed formula calculations

but structures are more and more complex nowadays

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



Analytical models

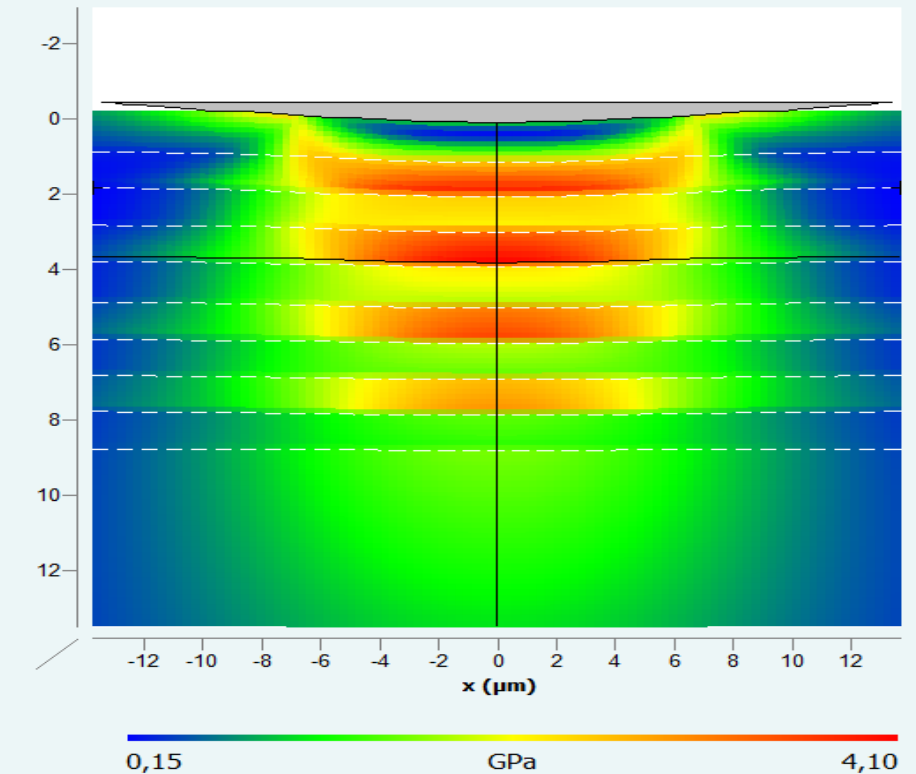
Much faster calculations possible

→ thus allows software for

experimental analysis

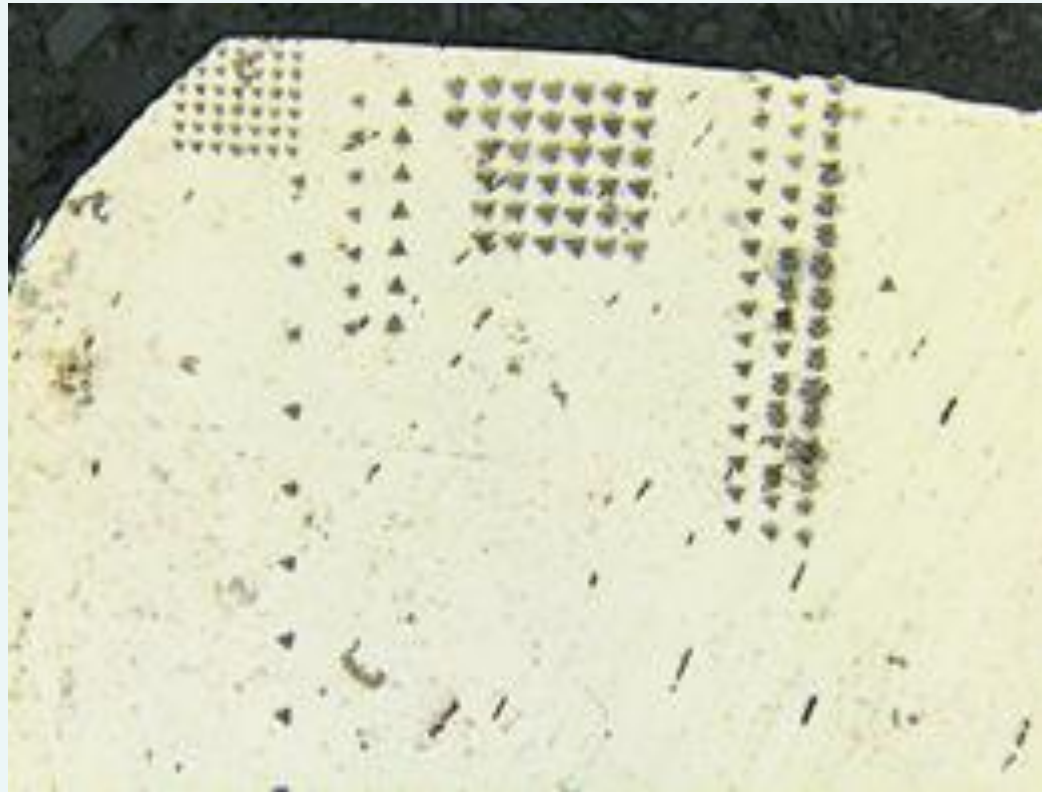
simulation

optimization



Analyze a series of indentation measurements

Calculate relative residual stress values for each indent





Reference module

FilmDoctor Studio TUM v 0.9.5.0 - time-limited edition - 14 days left - project: intrinsic stress reference 9 indents

Project Tools Help

material **curves** load fit results close residual stress reference module

step 2: load measured data

load curves use Contact Area use Hardness

filename	Reduced modulus	Hardness	x	y
tum 9 indents referenz.fdop	189,50	6,58	0 μm	0 μm
tum 9 indents s1.fdop	189,30	6,56	0 μm	9 μm
tum 9 indents s2.fdop	181,70	6,38	0 μm	18 μm
tum 9 indents s3.fdop	187,40	5,81	9 μm	0 μm
tum 9 indents s4.fdop	187,90	6,60	9 μm	9 μm
tum 9 indents s5.fdop	188,00	6,63	9 μm	18 μm
tum 9 indents s6.fdop	186,90	6,42	18 μm	0 μm
tum 9 indents s7.fdop	192,00	5,76	18 μm	9 μm
tum 9 indents s8.fdop	193,80	5,64	18 μm	18 μm

select reference measurement: tum 9 indents referer set reference residual stress: 0 GPa start stress determination OK

FilmDoctor Studio TUM v 0.9.5.0 - time-limited edition - 14 days left - project: intrinsic stress reference 9 indents

Project Tools Help

material curves **load** fit results close residual stress reference module

step 3: define the load parameters

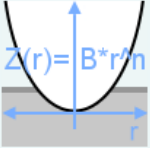
choose load definition method: fit load-depth curve

kind of indenter: exponentially shaped indenter

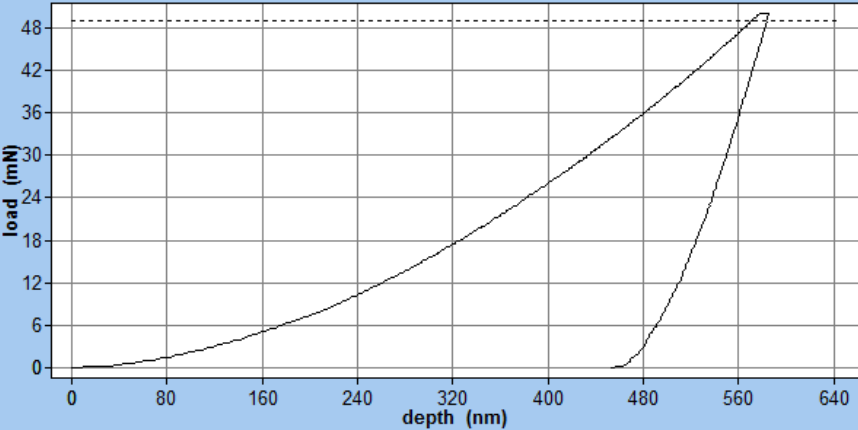
use effective half space accuracy: 10

indenter: ν : 0,07 E: 1,141 TPa user defined B: 1 n: 1

effective: ν : 0,3 E_r : 189,482 GPa F: 50,0076903 mN H: 6,57649 GPa



load (mN)



depth (nm)

load-depth curve load curve save curve clear curve edit curve draw points

fit curve save graph fit: 10 pts. 40 to 98 % of curve rms-dev.:

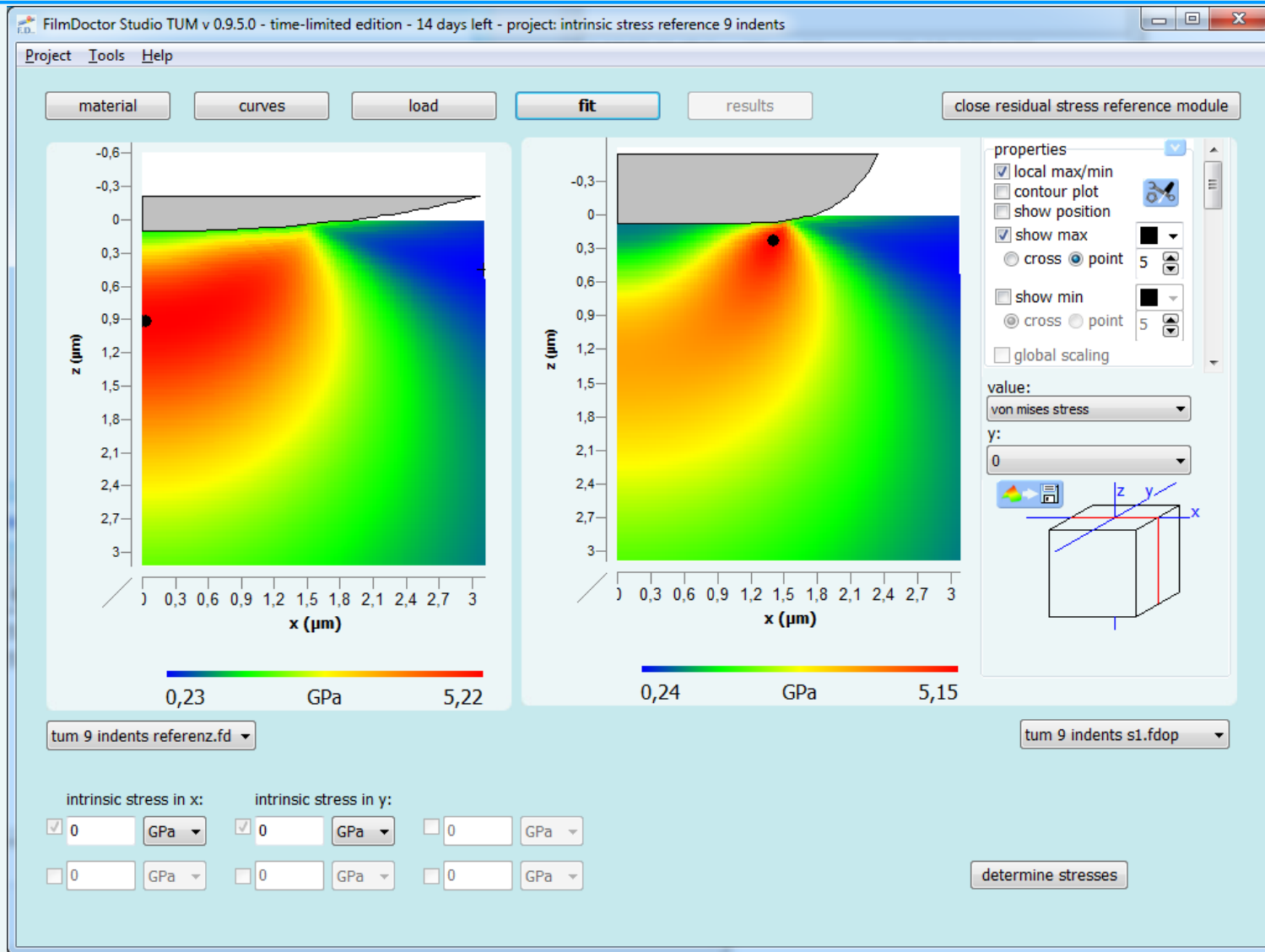
$F = C(h - h_0)^m$ h_0 : 0 nm

show values for curve 1 of 9

start series analysis

OK

Reference module





Reference module

FilmDoctor Studio TUM v 0.9.5.0 - time-limited edition - 14 days left - project: intrinsic stress reference 9 indents

Project Tools Help

material curves load fit results close residual stress reference module

values value table

measurement	x (μm)	y (μm)	biaxial stress (GPa)	depth of von Mises max. (μm)
tum 9 indents referenz.f dop	0	0	0	0,924518307809584
tum 9 indents s1.f dop	0	9	0,0703023808129127	0,220626780716599
tum 9 indents s2.f dop	0	18	0,711100921486317	0,675848811174404
tum 9 indents s3.f dop	9	0	0,43031566235328	0,919983481676788
tum 9 indents s4.f dop	9	9	-0,195164116647758	0,892272921999284
tum 9 indents s5.f dop	9	18	-0,240287055011461	0,893261809855548
tum 9 indents s6.f dop	18	0	-0,276843111031189	0,87238158760392
tum 9 indents s7.f dop	18	9	1,32690885036188	0,507276785723011
tum 9 indents s8.f dop	18	18	1,25729292077593	0,684454282693959
average stress			0,342625161455546	
standard deviation			0,591907659685718	

Reference module

FilmDoctor Studio TUM v 0.9.5.0 - time-limited edition - 14 days left - project: intrinsic stress reference 9 indents

Project Tools Help

material curves load fit results close residual stress reference module

values value table

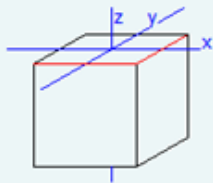
biaxial intrinsic stress (GPa)		x (μm)	
	0,00	9,00	18,00
0,00	0,00	0,43	-0,28
9,00	0,07	-0,20	1,33
18,00	0,71	-0,24	1,26

presentation settings

dimension: xy xz yz

value: biaxial intrinsic stress

depth: 0





Reference module extension

Extend the analysis by using the third dimension

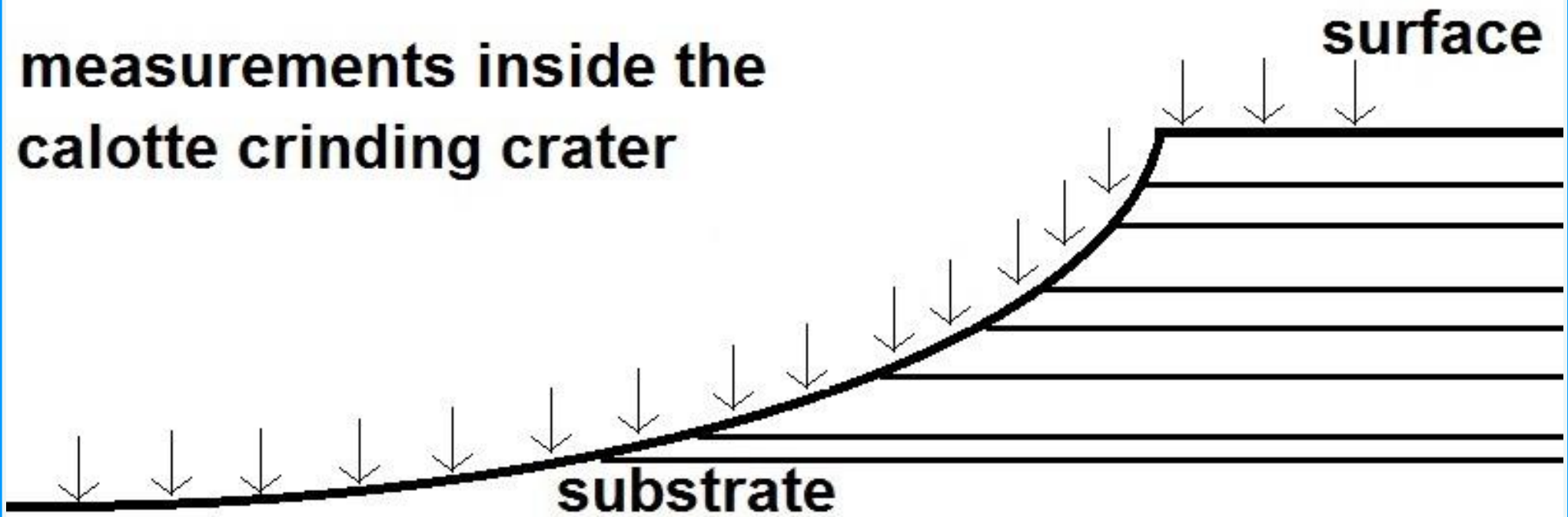
Get profile information along the z-axis (into the material)

How?

**Combine with another existing method
-> calotte grinding**

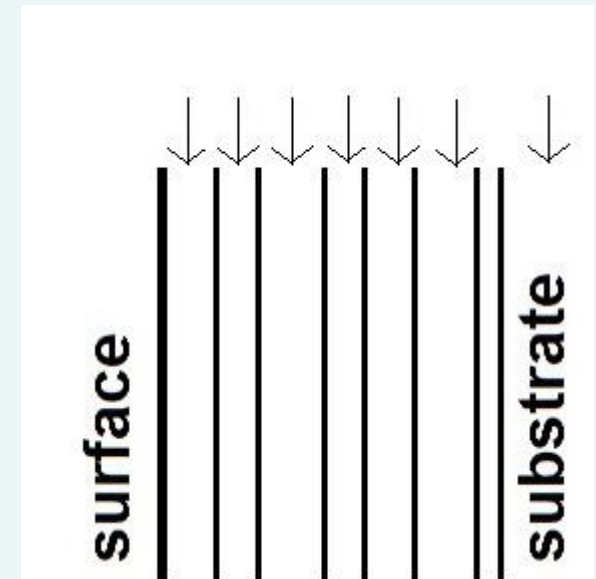
Now perform a series of indentation measurements inside and outside of the crater

**measurements inside the
calotte crinding crater**



Why calotte grinding?

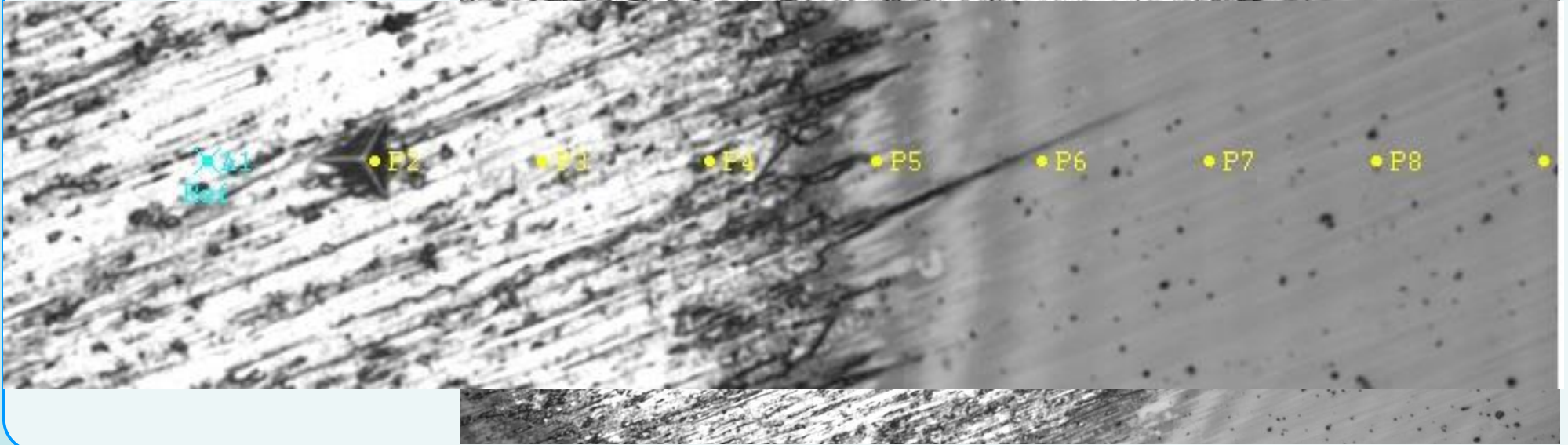
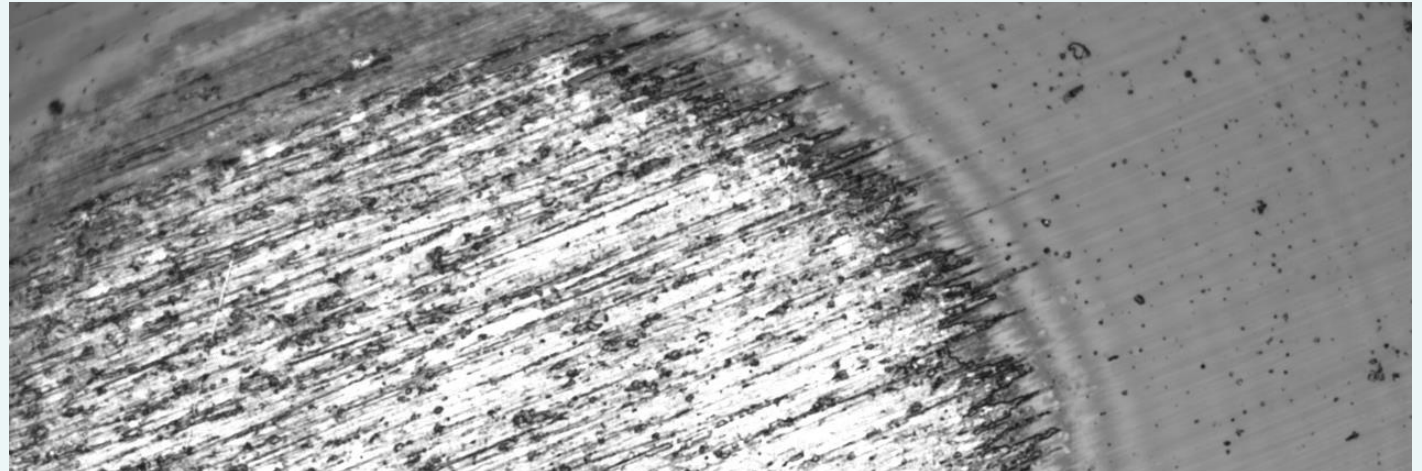
- easy and cheap, already available in most labs
- creates a huge area which could be used for indentation measurements compared to vertical cuts





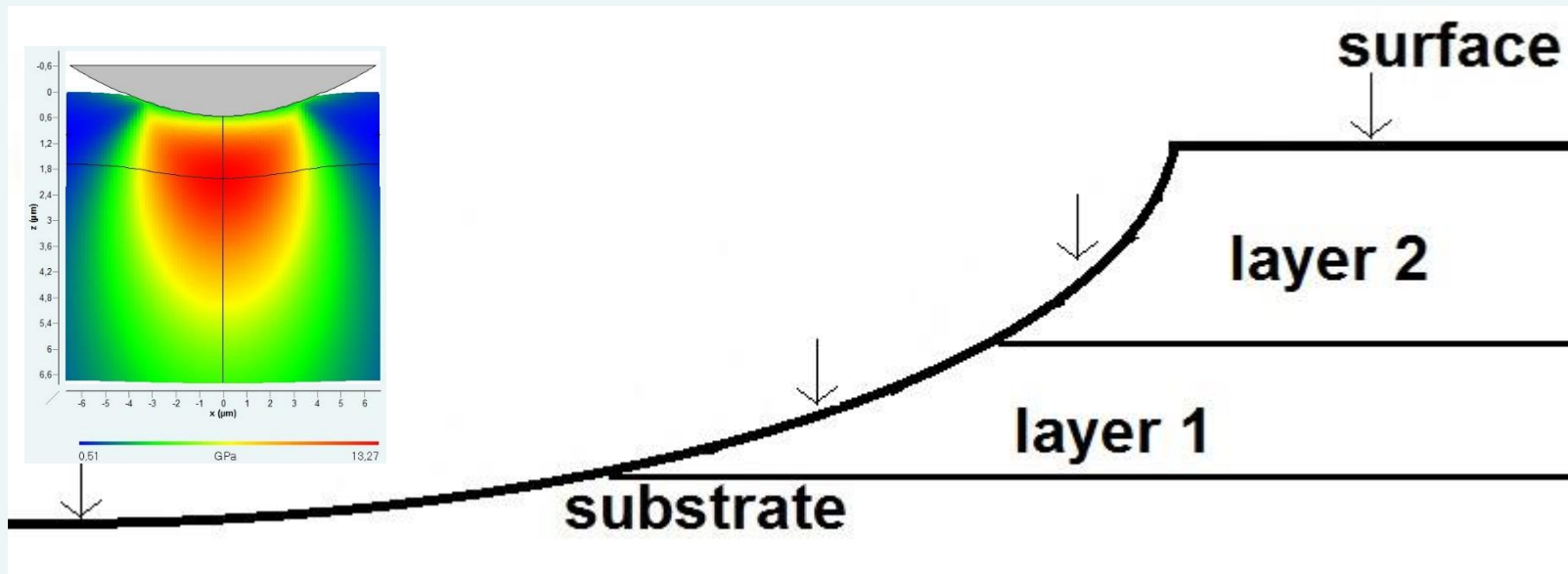
SIO Calotte Module

Can be easily programmed in modern indentation devices



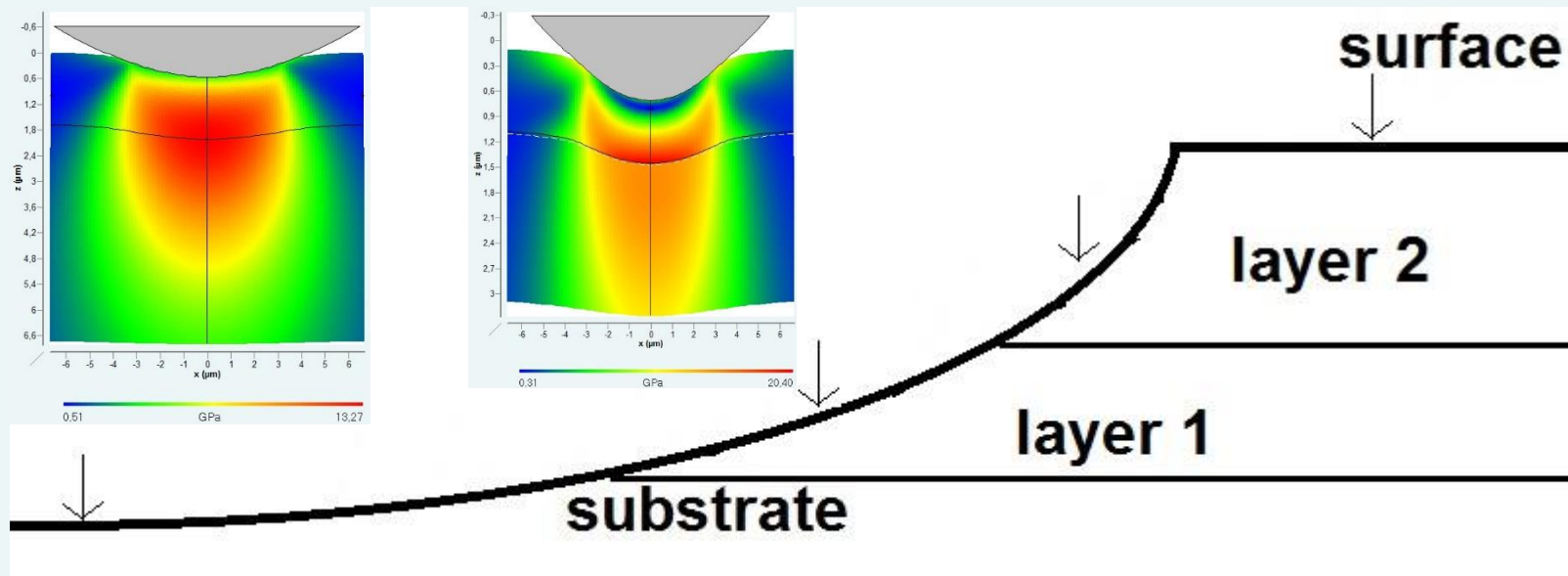
How it works in detail

- Analysis I: substrate measurement → E_s



How it works in detail

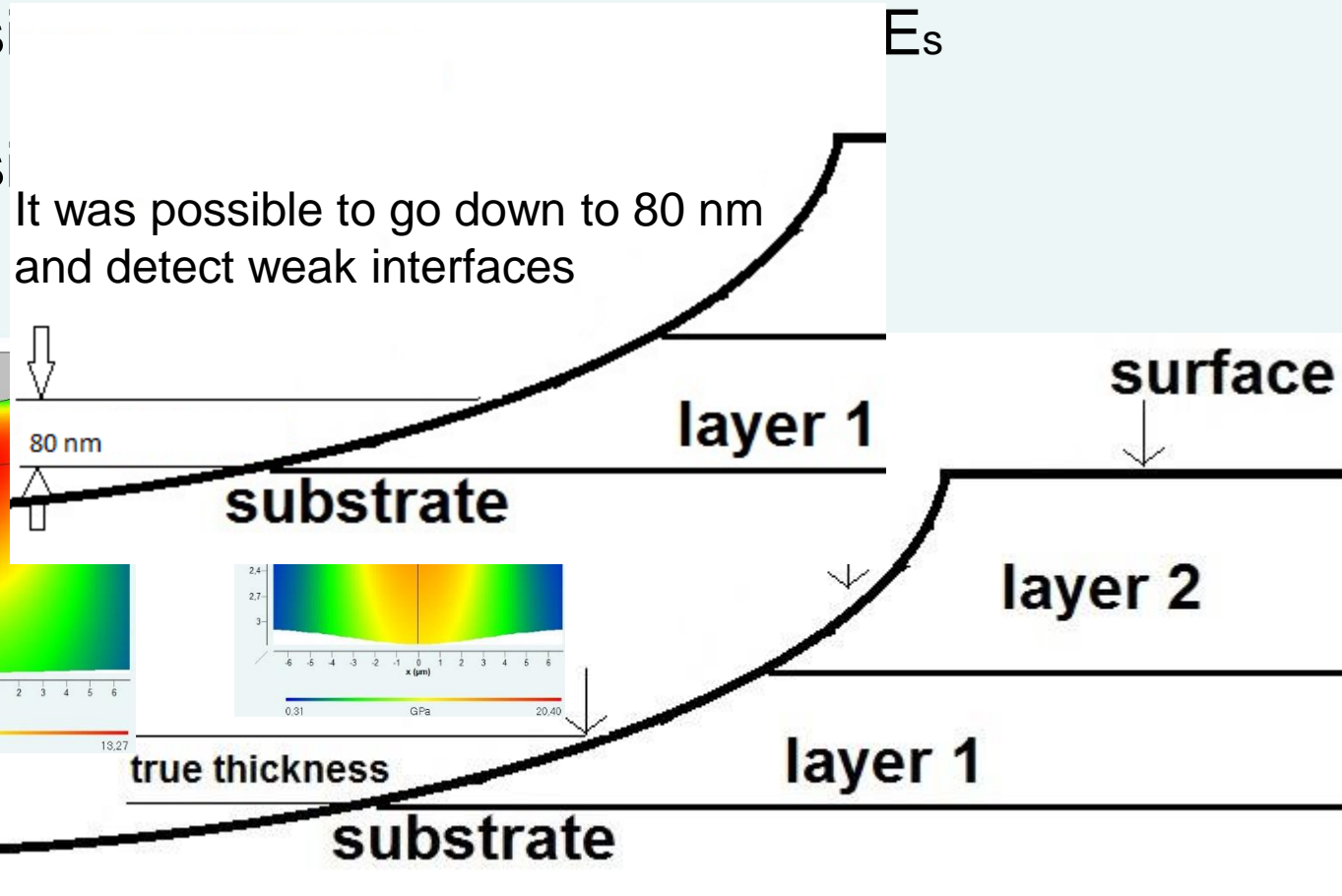
- Analysis I: substrate measurement $\rightarrow E_s$
- Analysis II: 1 μm thick layer 1 $\rightarrow E_1$



How it works in detail

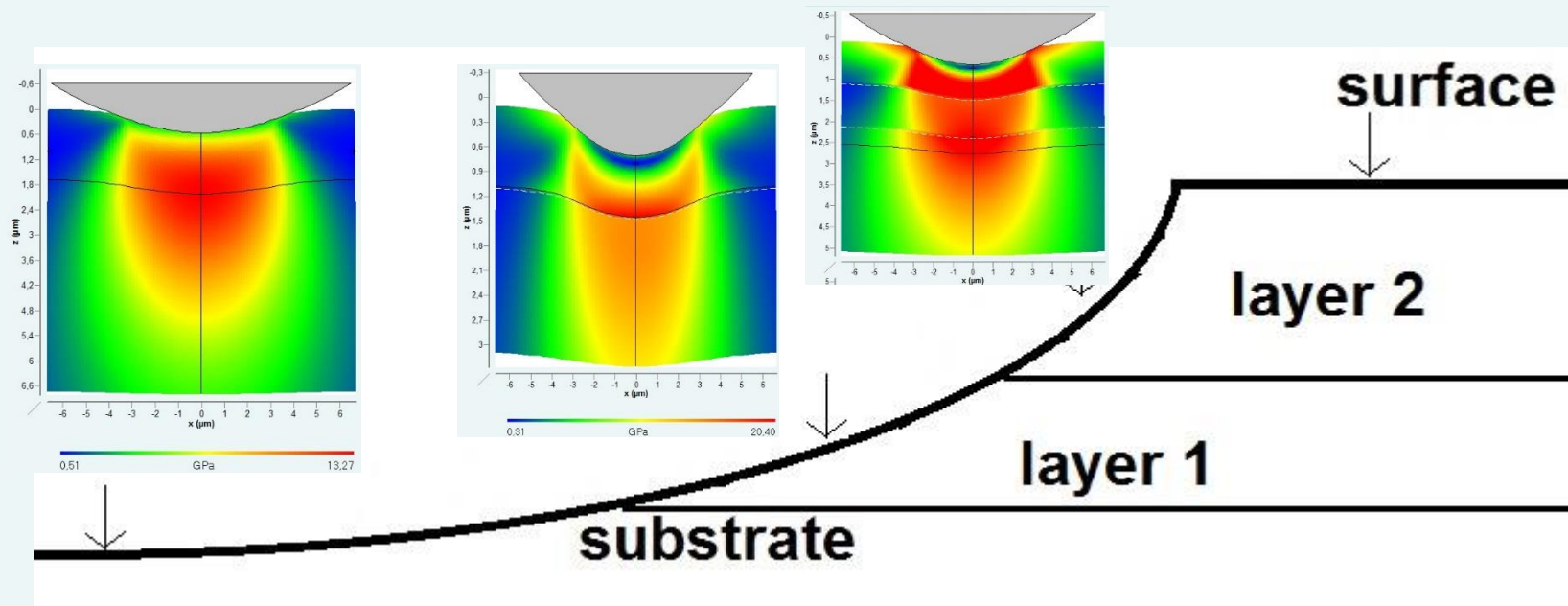
➤ Analysis

➤ Analysis



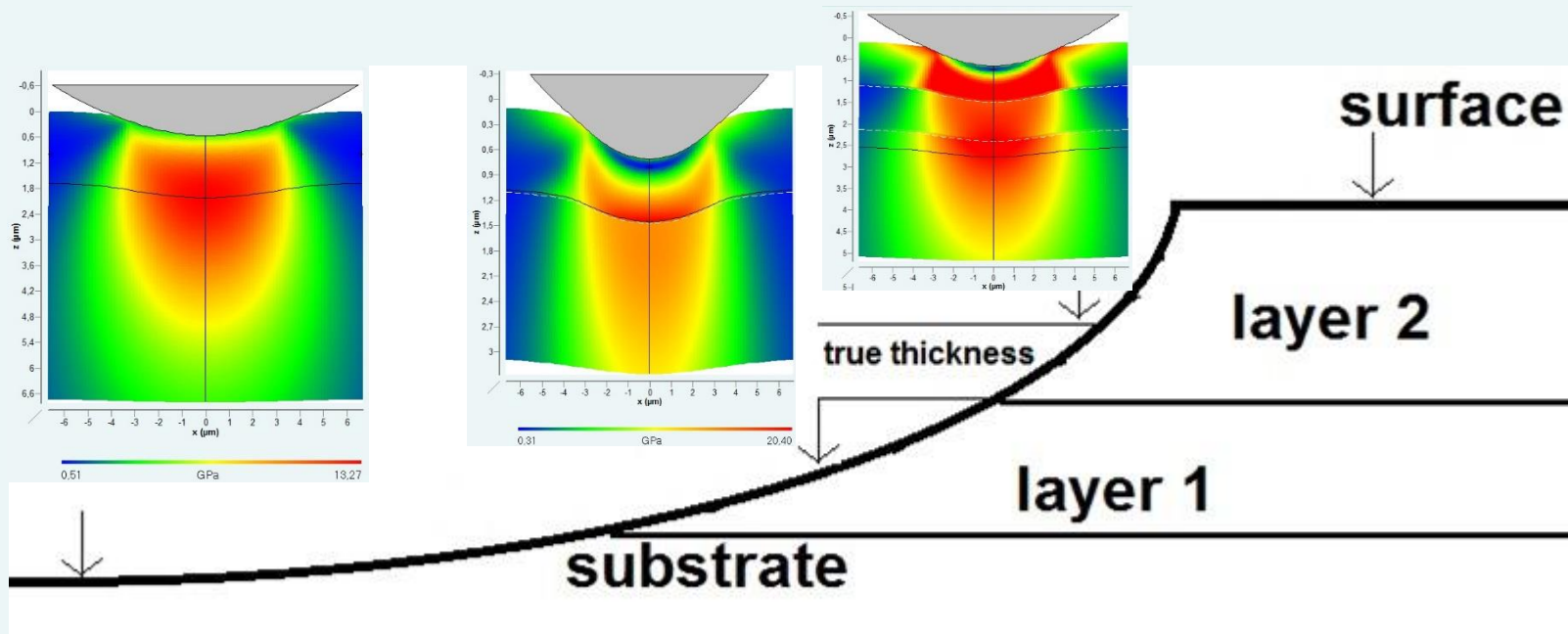
How it works in detail

- Analysis III: 1 μm thick layer 2 $\rightarrow E_{21}$



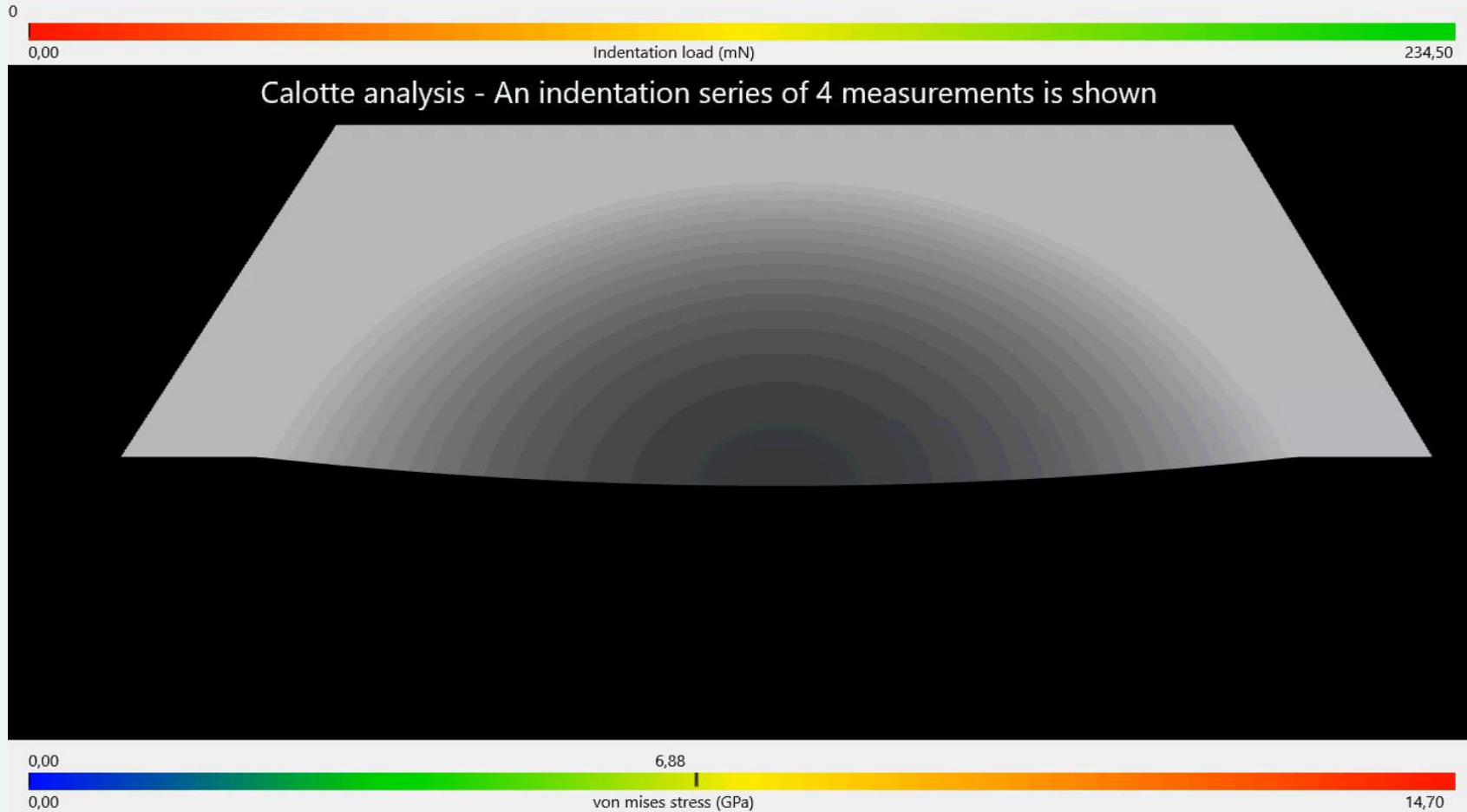
How it works in detail

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





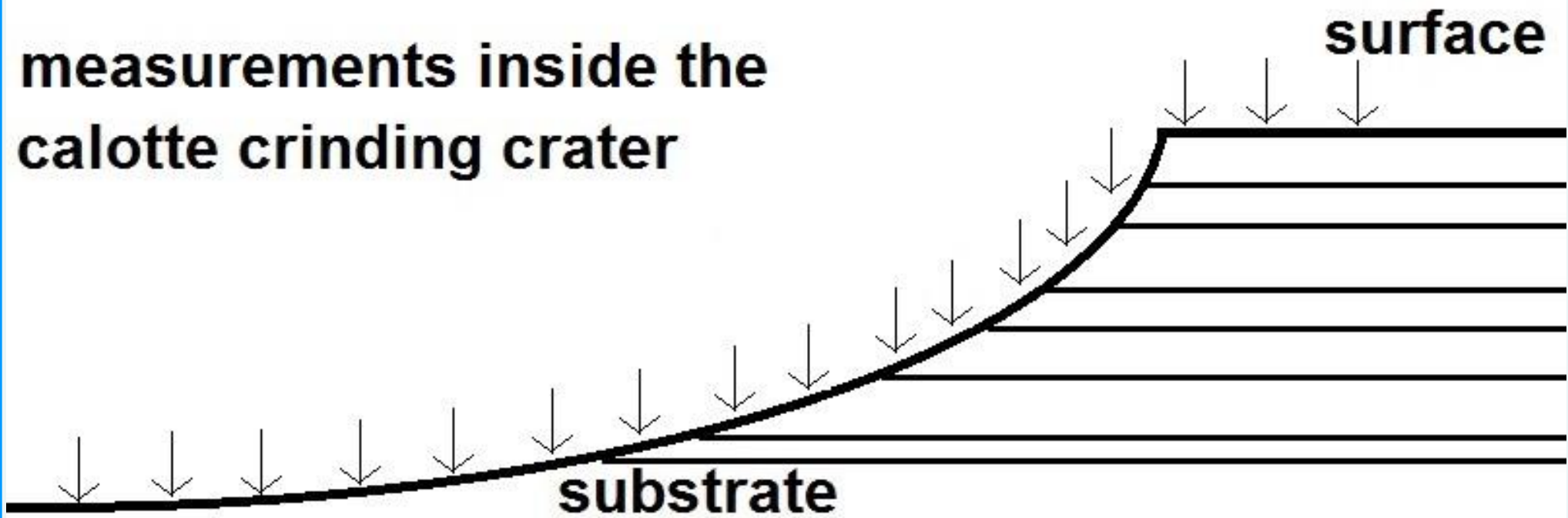
SIO Calotte Module



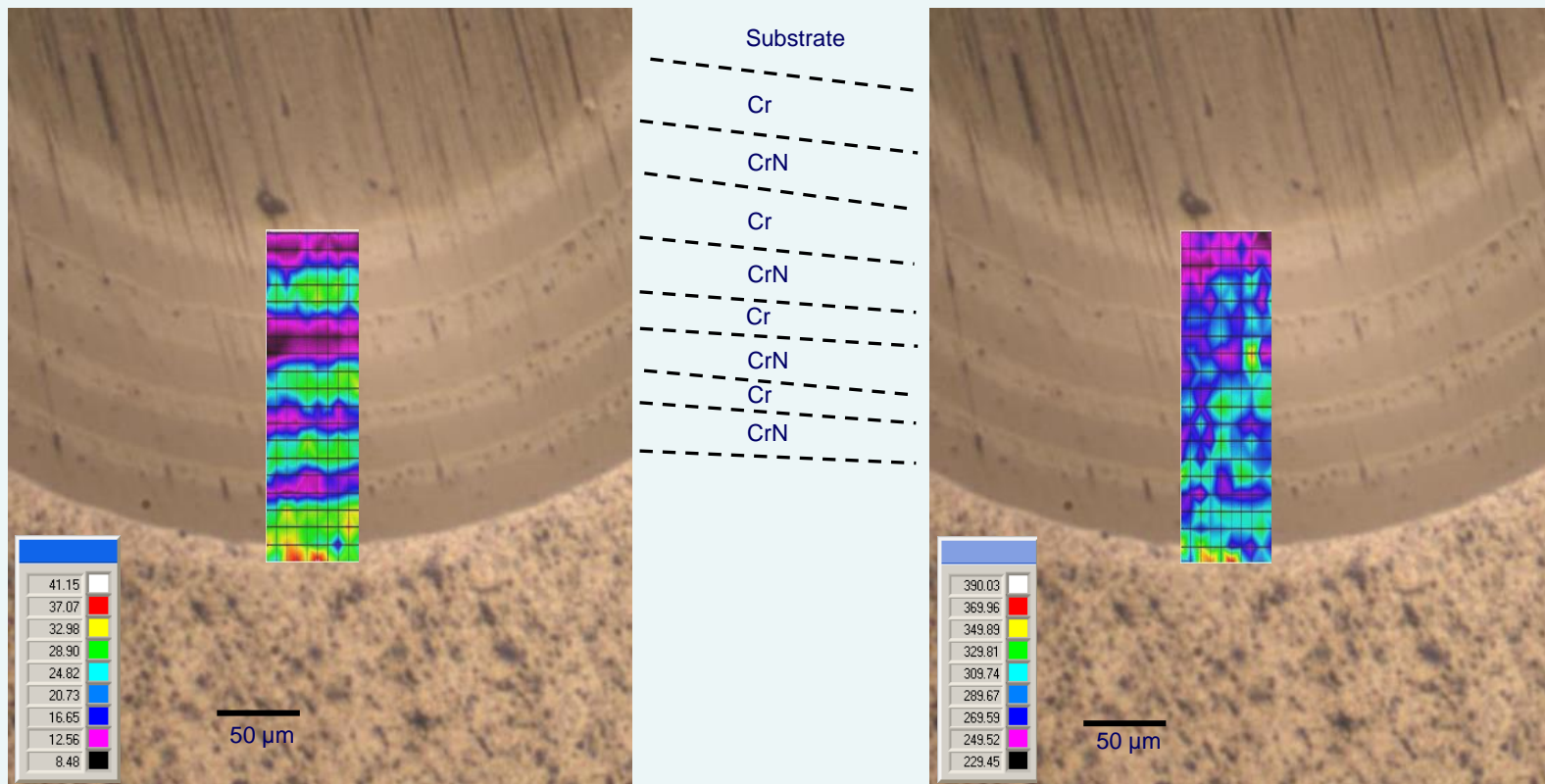
<https://worldformulaapps.com/portfolio/calotte>
[youtube](#)

Now perform a series of indentation measurements inside and outside of the crater
Reference indent could be anywhere

**measurements inside the
calotte crinding crater**



Hardness and modulus maps are created automatically in most actual indentation device software packages. The manufacturers put a lot of effort in faster measurement procedures.





Stress profiles

Example: 4.5 μm dlc coating on a metal substrate

material curves load fit results close residual stress reference module

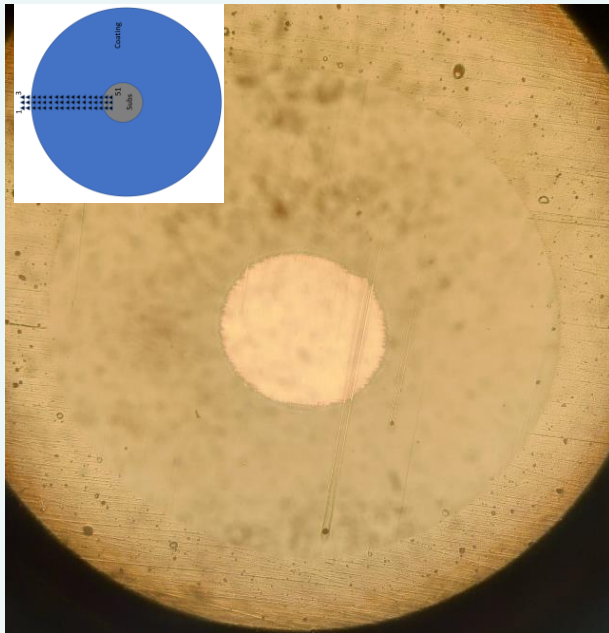
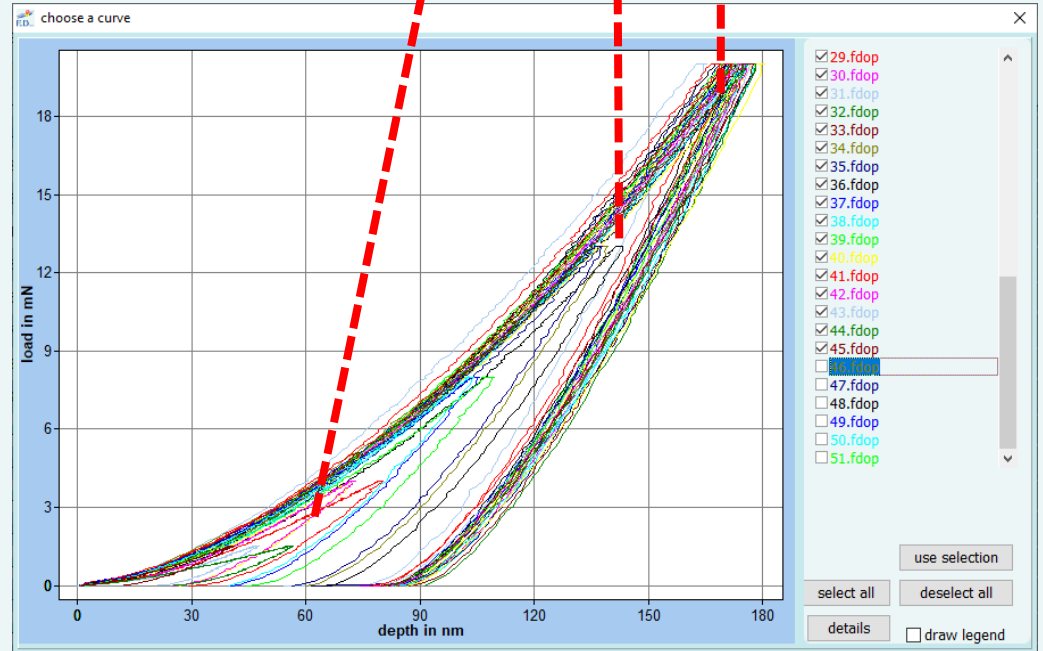
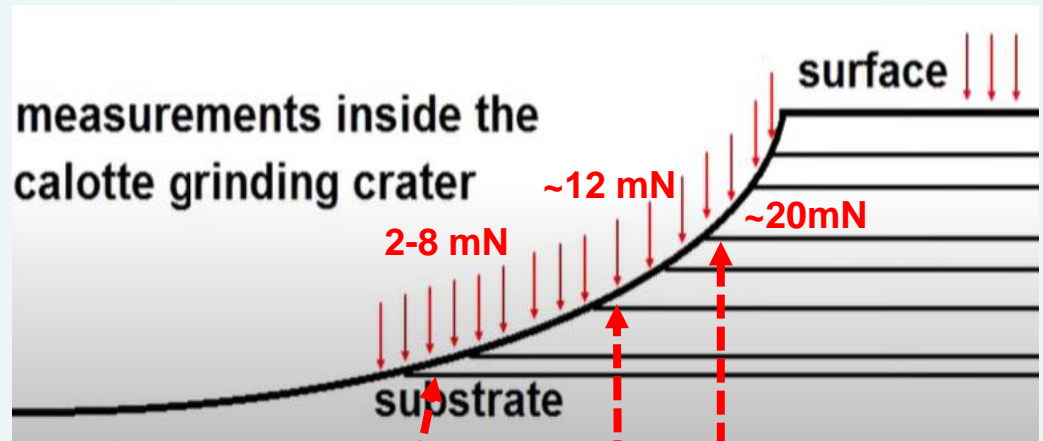
step 1: select your material

select all Poisson's ratio Young's modulus select from database layer thickness intrinsic stresses

gradient viscous gradient gradient

<input checked="" type="checkbox"/> layer 1:	ν : 0,25	E: 440 GPa	user defined	h: 4,5 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 2:	ν : 0,22	E: 450 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 3:	ν : 0,321	E: 115,7 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 4:	ν : 0,22	E: 450 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 5:	ν : 0,321	E: 115,7 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 6:	ν : 0,22	E: 450 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 7:	ν : 0,321	E: 115,7 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 8:	ν : 0,22	E: 450 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 9:	ν : 0,321	E: 115,7 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 10:	ν : 0,22	E: 450 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 11:	ν : 0,321	E: 115,7 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 12:	ν : 0,22	E: 450 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 13:	ν : 0,321	E: 115,7 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 14:	ν : 0,22	E: 450 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="checkbox"/> layer 15:	ν : 0,321	E: 115,7 GPa	user defined	h: 1 μm	in x: 0 GPa	in y: 0 GPa	more
<input type="button" value="upgrade to more layers"/>							
substrate:	ν : 0,3	E: 240 GPa	user defined		in x: 0 GPa	in y: 0 GPa	more

Load a series of
indentation
measurements

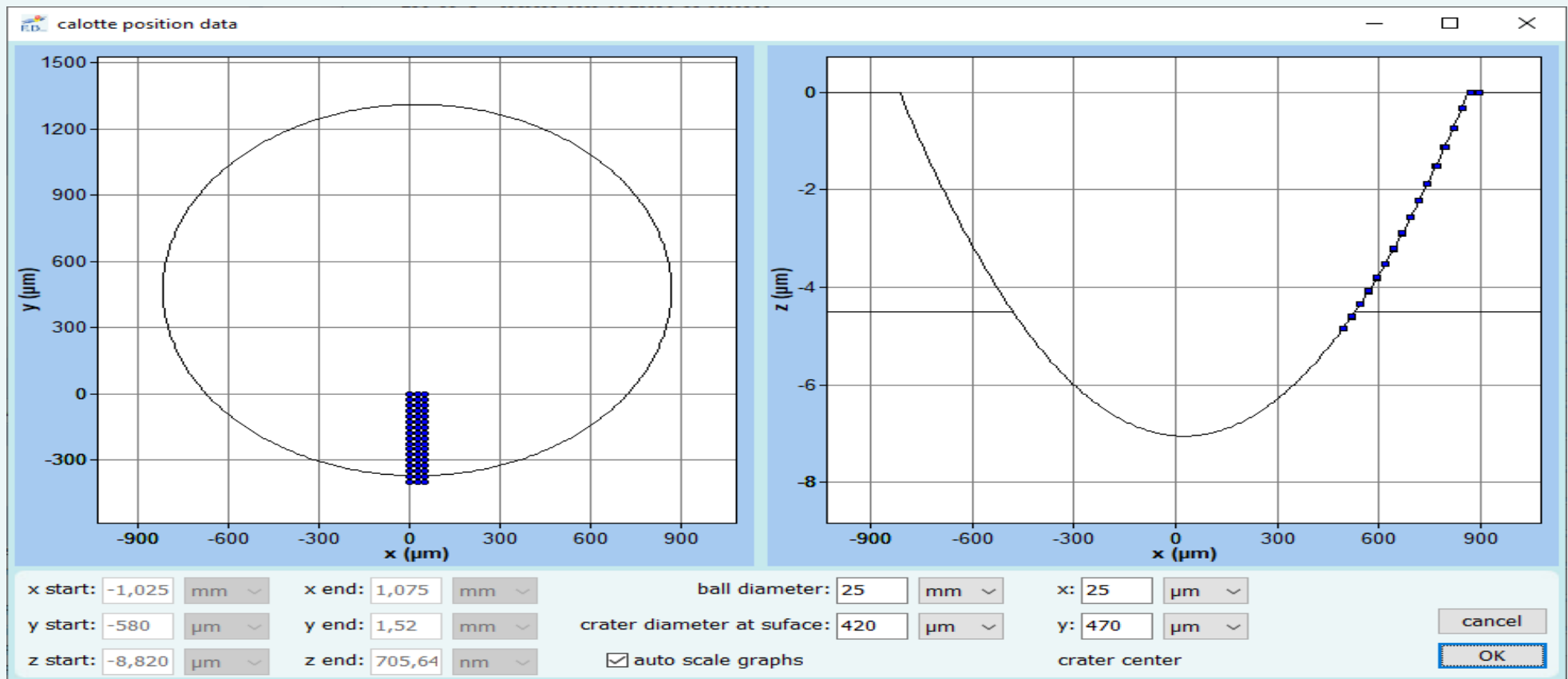




Stress profiles

Define the position of each measurement

Define the calotte crater parameters

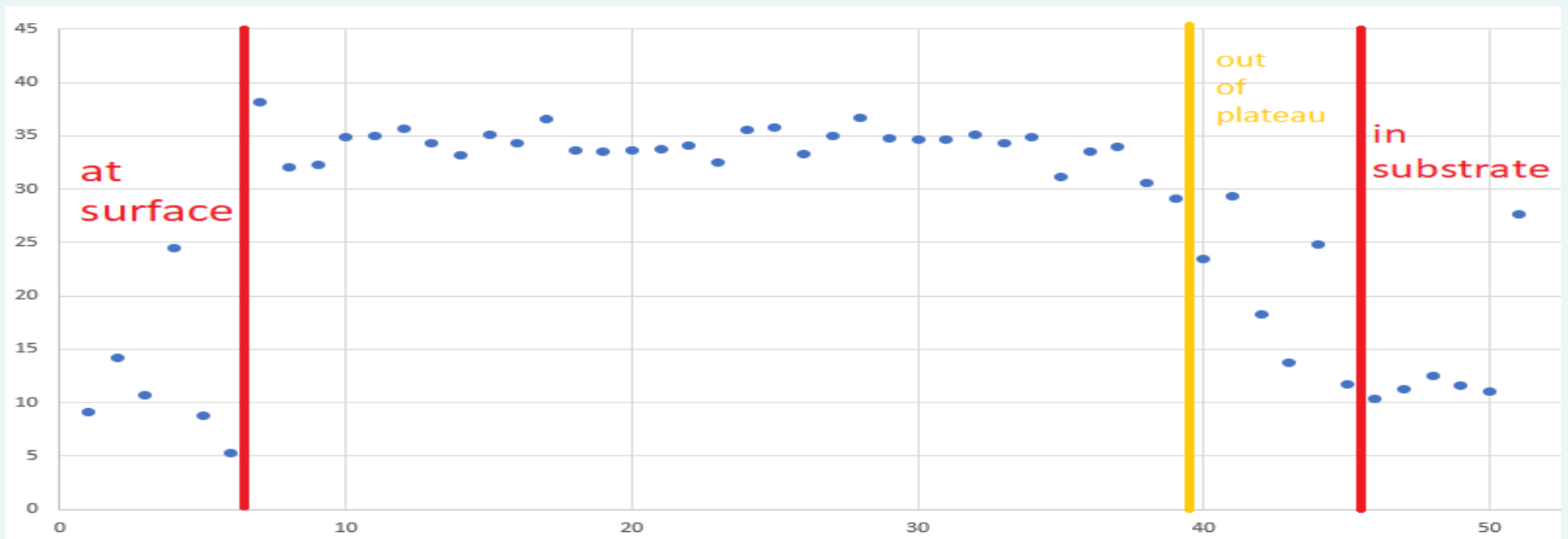




Stress profiles

Measurements were performed from the surface to the substrate

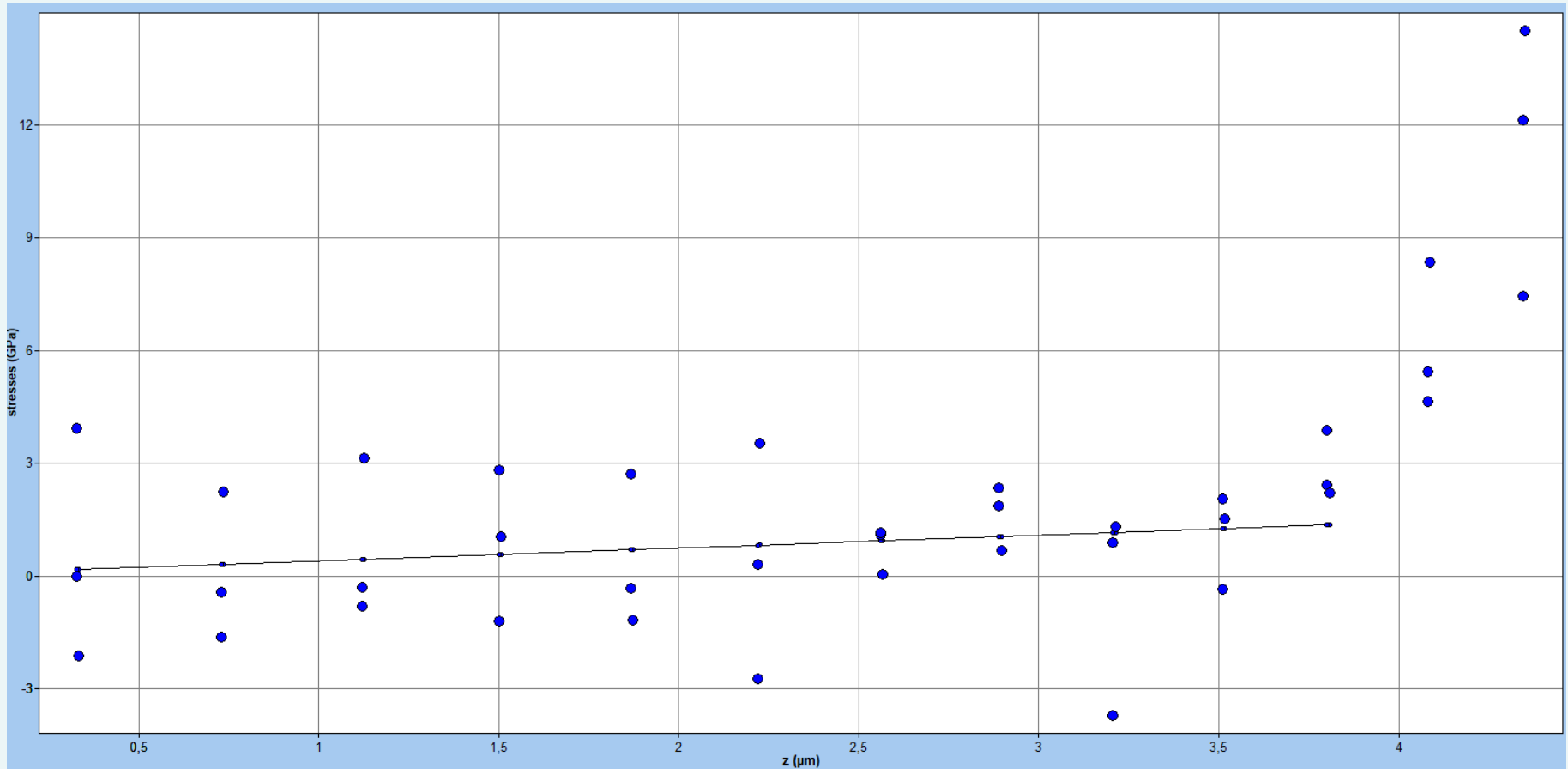
Nice hardness plateau in most of the coating





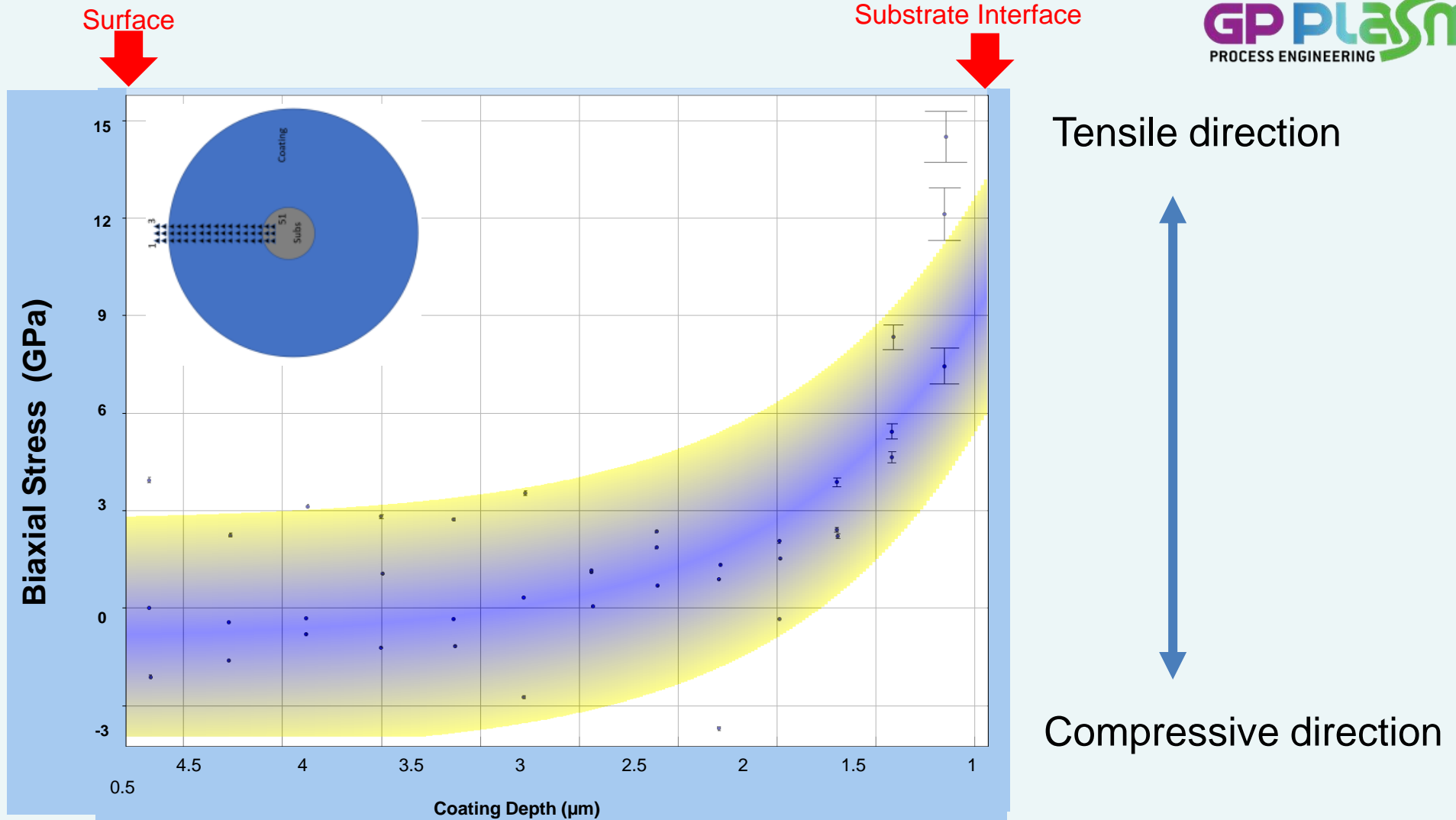
Stress profiles

Nice residual stress plateau in most of the coating was found





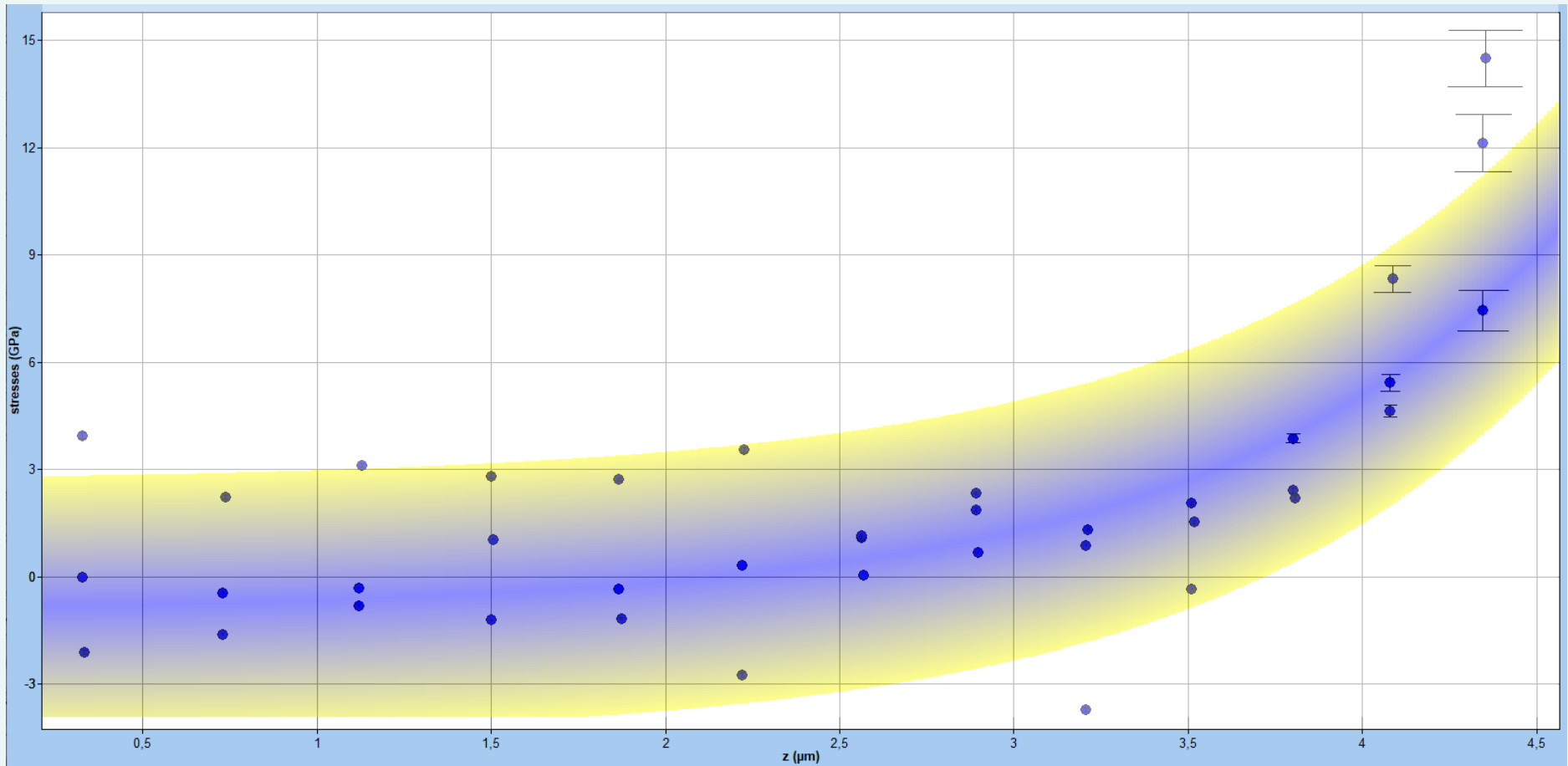
Stress Profiles From Nanoindentation



DLC coating by PECVD with a doped interface on A2 Tool Steel

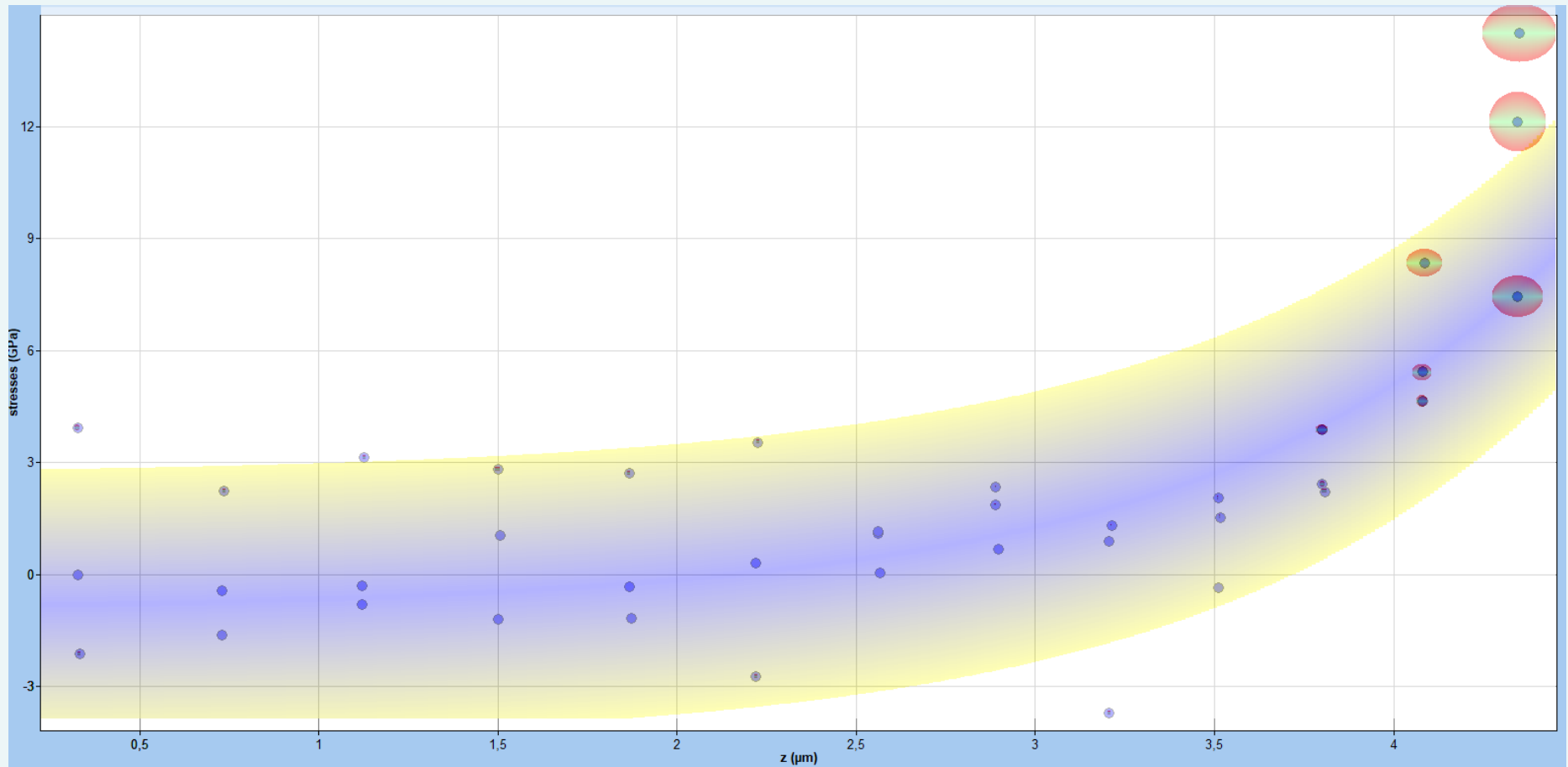
Stress profiles

Errors are higher for the substrate near measurements, because a much lower load was used due to the very thin remaining coating



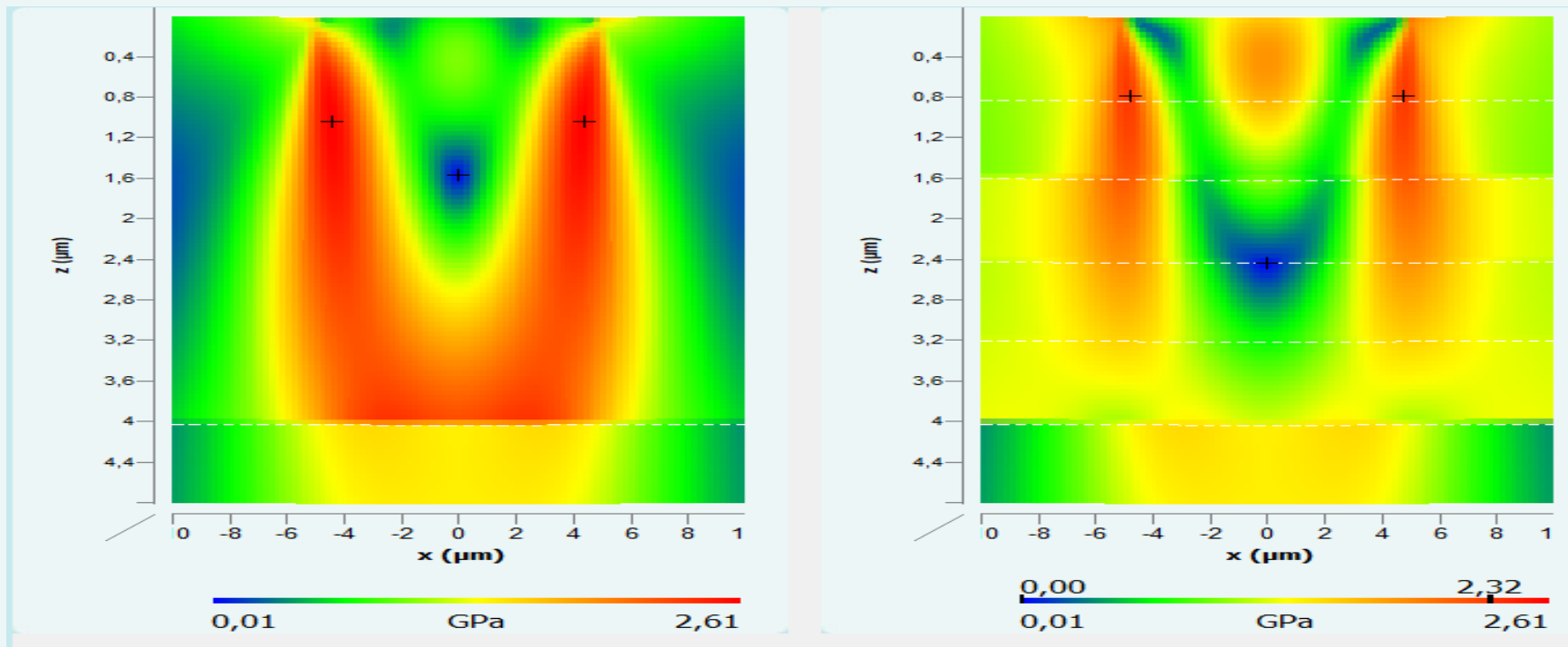
Stress profiles

The increased stress values close to the interface were in good correlation with the observed failure mechanism

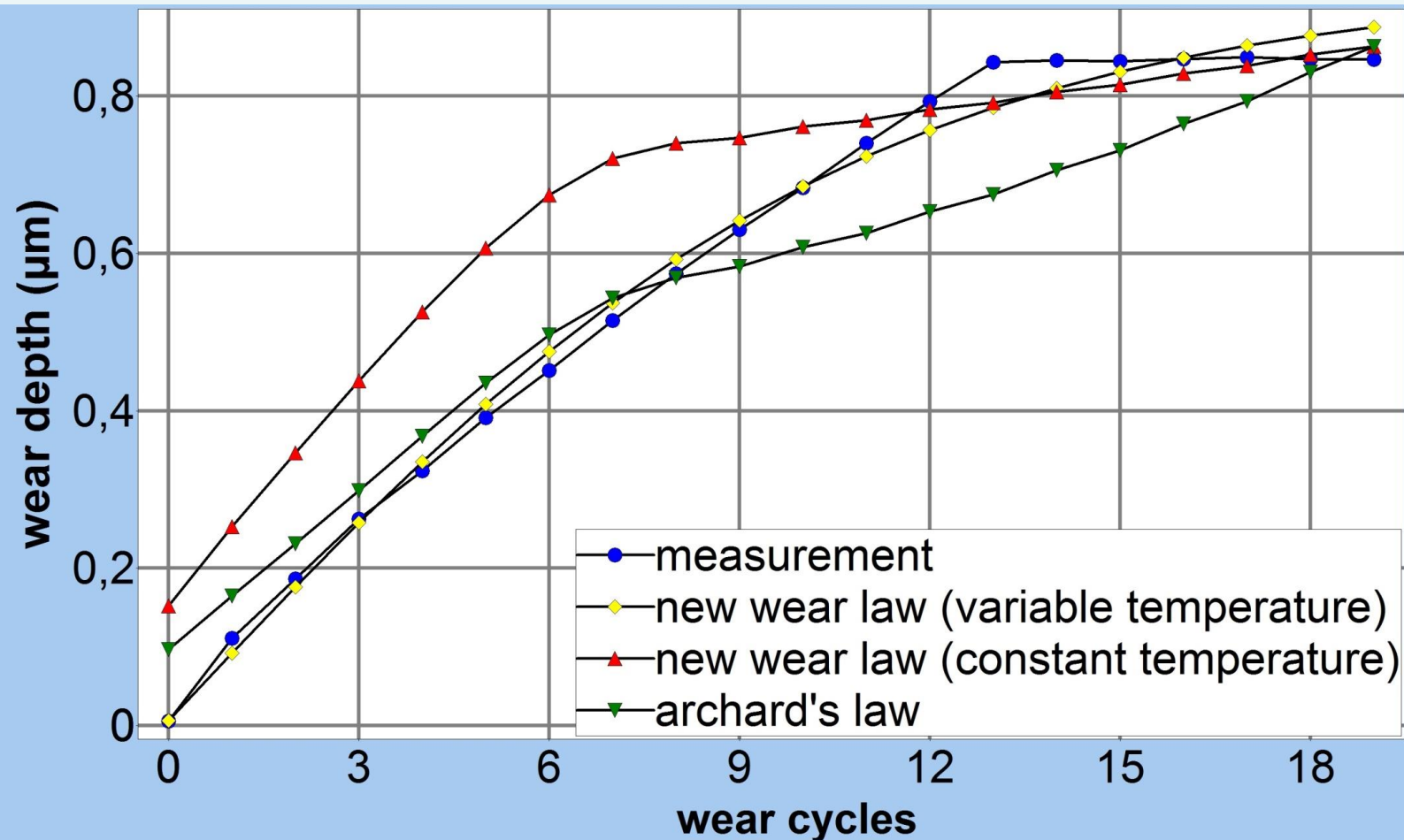


Stress optimization

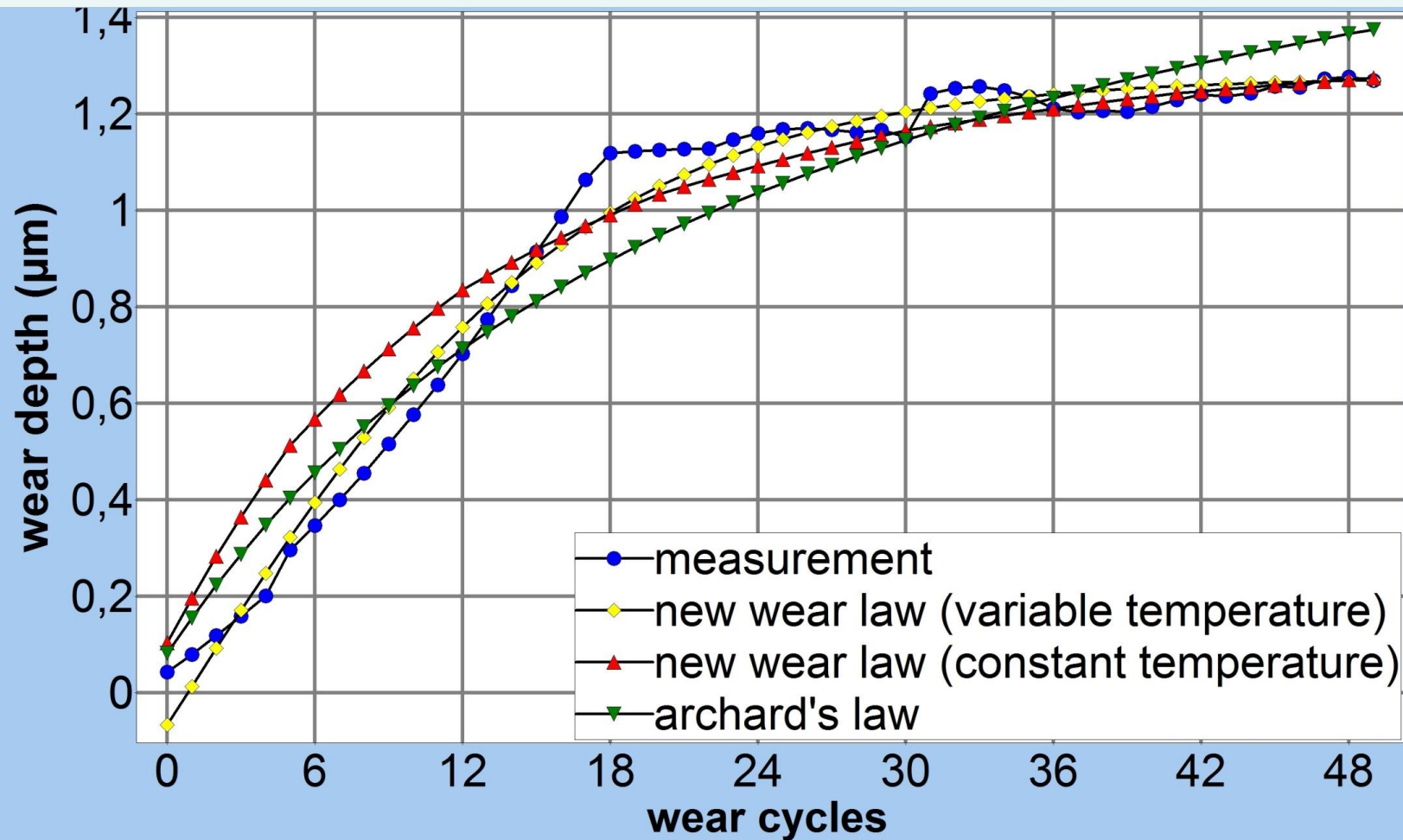
Improve performance by steering (e.g. bias changes) intrinsic stresses during the deposition process
 No material changes or new materials needed



<https://worldformulaapps.com/portfolio/intrinsic-stress>



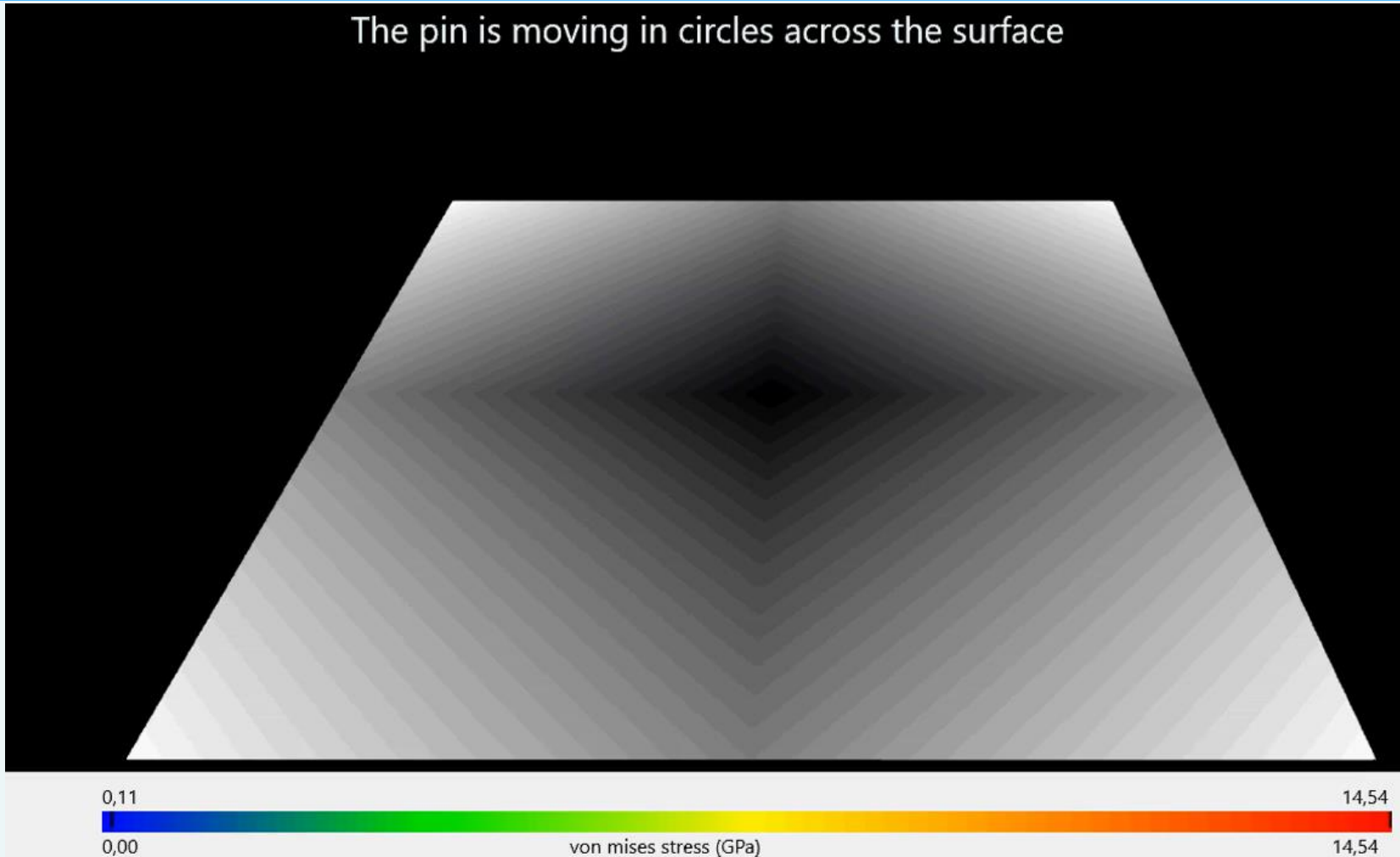
[13] A. Gies, T. Chudoba, N. Schwarzer, J. Becker: „Influence of the coating structure of a-C:H-W coatings on their wear-performance: a theoretical approach and its practical confirmation“, accepted for publication in ICMCTF 2013-proceedings



[14] T. Liskiewicz; B. Beake; N. Schwarzer; M. Davies: „ Short note on improved integration of mechanical testing in predictive wear models“, accepted for publication in ICMCTF 2013-proceedings

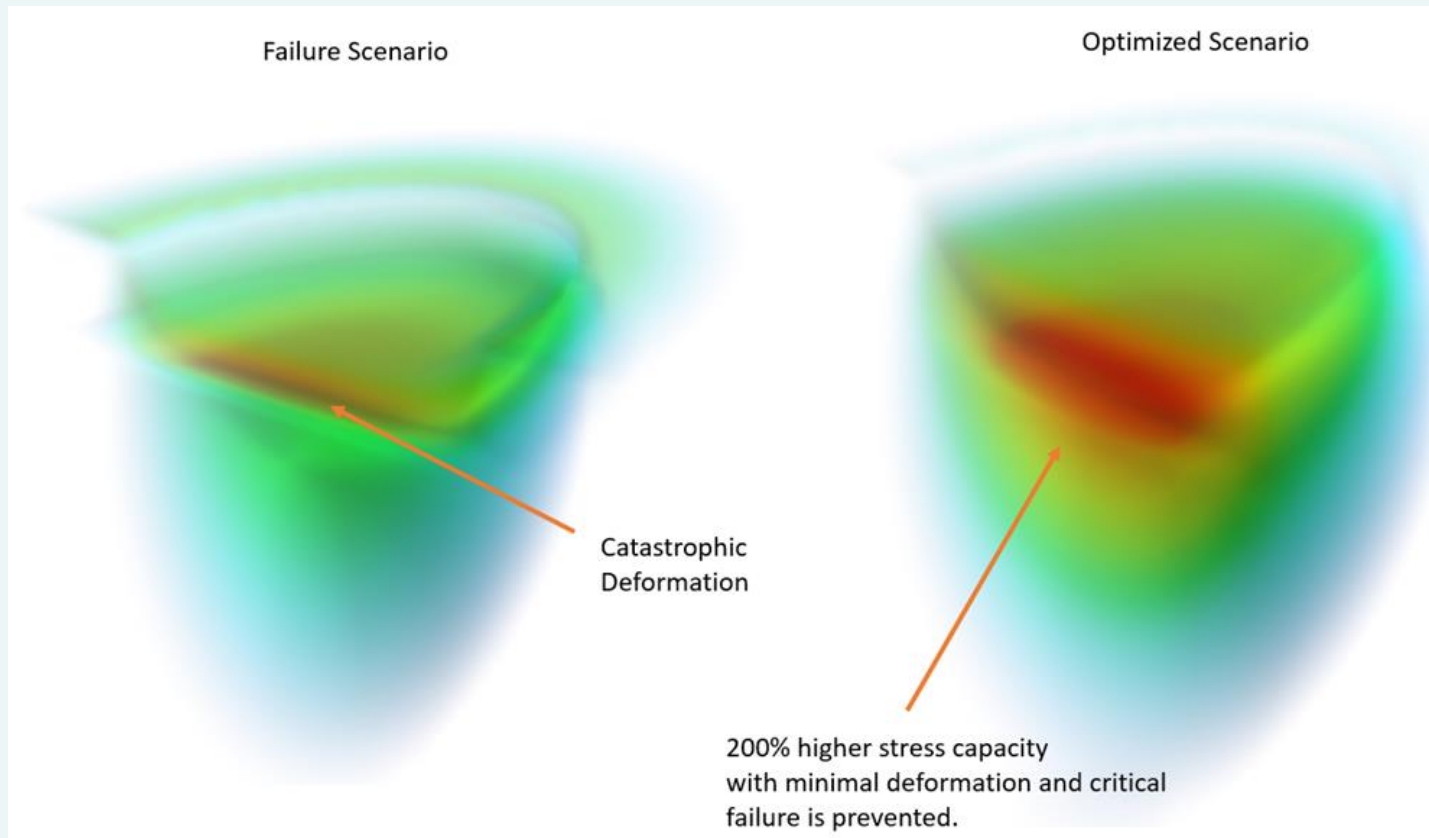
Predictive Wear Modelling

The pin is moving in circles across the surface



<https://worldformulaapps.com/portfolio/pin-on-disc>

Combining different programming languages (actually python) to create more view options.





Conclusion

Combining calotte grinding with indentation measurements offers a unique way to gather profile information from the coating.

Results which couldn't be found with any other methods were obtained and could explain the failure.

Extension to time depending material behaviour offers a lot of new options.

- Locally determined properties incl. stress.
- Quantified Errors at each location and for the ensemble of data.
- Surface roughness, load, tip-size and calo ball size optimized for the layered system.



Software that even a 14 year old intern used effectively to optimize hip implants.

[watch on youtube](#)

jugend  **forscht**
schüler experimentieren

2022 – Wir machen mit!

