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How to measure intrinsic stresses via nanoindentation – an example

Abstract

In the following study the measurement an analysis of intrinsic stresses and yield strength via nanoindentation and their separation from each other will be elaborated using a specific example. The results for the intrinsic stresses are compared with those determined by other means. The agreement is excellent.

Introduction

Yield strength values obtained via nanoindentation are often flawed by the intrinsic stresses residing in the surfaces area of the samples in question. Within this study the procedure necessary to separate the intrinsic stresses and determine correct yield strength values is demonstrated on thin films with known biaxial intrinsic stresses.

In [1] the author has proposed the following measuring procedure:

1. The yield strength is determined using the method of the effectively shaped indenter as presented in [1]. However, during unloading, the indenter is only drawn back to a distinct fraction of the maximum load p_0. This load shall be called p_1. The reader should note that p_1 must be chosen such that on the one hand there is "enough unloading curve" for the determination of the shape of the effective indenter, and on the other hand the load is still big enough in order to avoid strong and dominant inelastic unloading effects like e.g. unloading fractures. In addition, a p_1 close to p_0 also assures that the shape of the effective indenter only changes in an insignificant manner during unloading. We call the determined yield strength σ_M^{crit}. Here we now have to add the two elastic fields resulting from the intrinsic stresses σ_{ij}^I and the nanoindenter loading σ_{ij}^L. The von Mises stress can be written in the following form:

$$\sigma_M = \sqrt{\frac{1}{2}\left(\left(\sigma_{xx}-\sigma_{yy}\right)^2+\left(\sigma_{zz}-\sigma_{yy}\right)^2+\left(\sigma_{xx}-\sigma_{zz}\right)^2+6*\left(\tau_{xy}^2+\tau_{xz}^2+\tau_{zy}^2\right)\right)}$$

with $\sigma_{ij}=\sigma_{ij}^I+\sigma_{ij}^L$.

2. Now a slowly oscillating tangential load component t_x with increasing amplitude is added and the resulting lateral shift is measured. So we now have mixed load conditions and assume our - in step one determined - effectively shaped indenter acting with the combined load components p_1 and t_x onto the coating substrate compound.

3. The slowly oscillating tangential loading with increasing amplitude is monitored with very high resolution (as well as the static normal load and displacement, of course) until nonlinear behavior can be detected. Thus, the value of maximum tangential load $t_x=t_{crit}$ or maximum lateral displacement u_σ is determined. Now we introduce the following assumption: The

combined stresses add up to a mechanical stress field producing a maximum von Mises stress $\sigma_M = \sigma_M^{crit}$ somewhere within the investigated coating material.
4. With these two measurements and the resulting measured values p_0, p_1, the corresponding penetration depth, t_{crit} and u_σ we can construct two linear independent equations

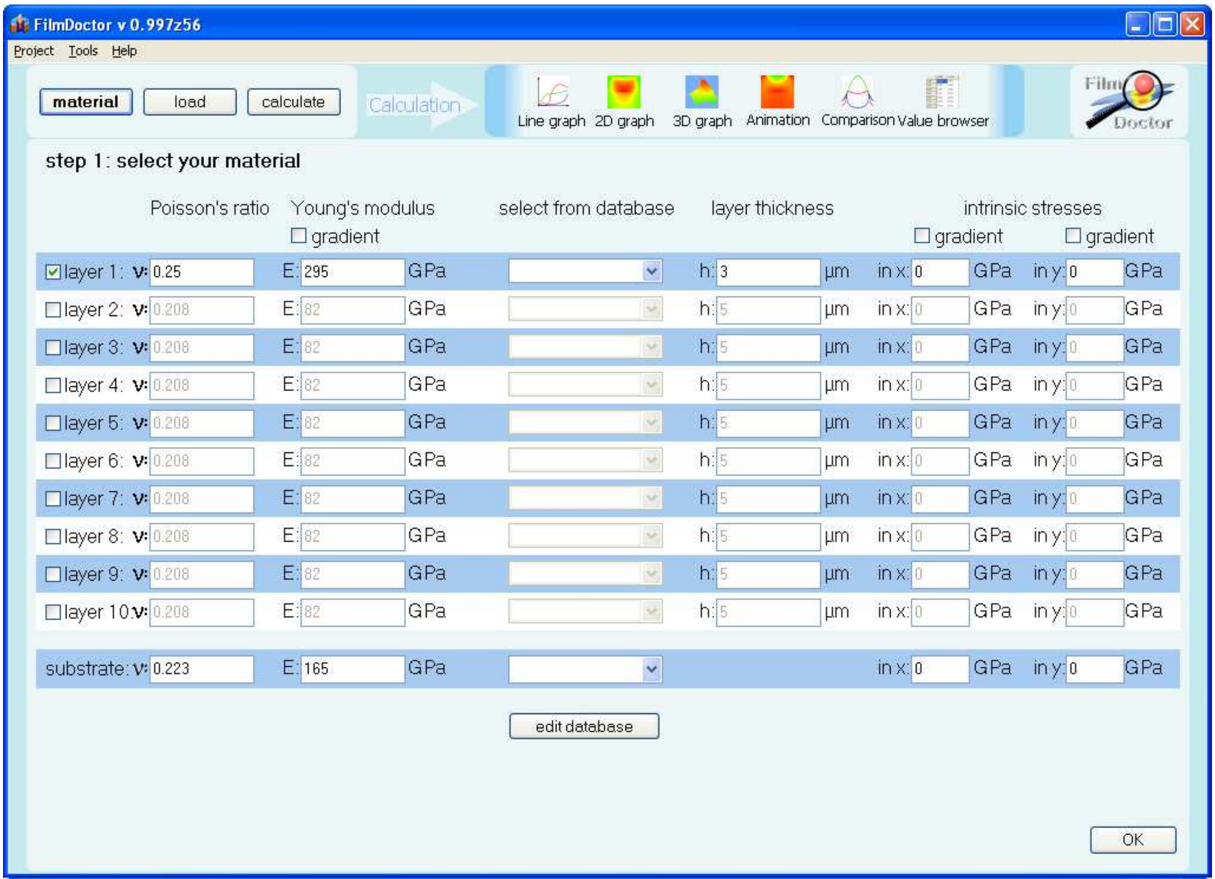
$$\sigma_M^{crit} = \sqrt{\frac{1}{2}\begin{pmatrix} \left(\sigma_{xx}^L - \sigma_{yy}^L + \sigma_{rr}^f\left(f_{xx}^I - f_{yy}^I\right)\right)^2 + \left(\sigma_{zz}^L - \sigma_{yy}^L + \sigma_{rr}^f\left(f_{zz}^I - f_{yy}^I\right)\right)^2 \\ +\left(\sigma_{xx}^L - \sigma_{zz}^L + \sigma_{rr}^f\left(f_{xx}^I - f_{zz}^I\right)\right)^2 \\ +6*\left((\tau_{xy}^L + \sigma_{rr}^f f_{xy}^I)^2 + (\tau_{xz}^L + \sigma_{rr}^f f_{xz}^I)^2 + (\tau_{zy}^L + \sigma_{rr}^f f_{zy}^I)^2\right) \end{pmatrix}},$$

for the critical von Mises stress respectively yield strength with two different nanoindenter stress distributions σ_{ij}^L resulting from the pure normal loading $\sigma_{ij}^L = \sigma_{ij}^{normal\ load}$ and the mixed loading experimental setup $\sigma_{ij}^L = \sigma_{ij}^{mixed\ load}$. We have used the fact, that according to our approach (eq. (8) in [1]) for the intrinsic stresses we can write the intrinsic stress field as $\sigma_{ij}^I = \sigma_{rr}^f * f_{ij}^I(x,y,z) \equiv \sigma_{rr}^f * f_{ij}^I$ with a suitable function f(x,y,z). Due to the linear independence of the two loading conditions we can now extract the intrinsic stress value σ_{rr}^f residing in the coating and the critical von Mises stress of a corresponding unstressed material ($\sigma_{rr}^f = 0$), meaning the von Mises stress this unstressed material would require in order to reach its yield strength limit $\sigma_M = \sigma_M^{crit}$.

The measurement and analyzing procedure in praxis

The above described analyzing procedures shall now be demonstrated on some very first experimental examples. The measurements have been performed by T. Chudoba and V. Linss from the company ASMEC using a so called UNAT measurement system [2] equipped with a lateral force unit (LFU) which can generate und measure lateral forces und displacements with the same resolution like common nanoindenters in normal direction.
Four samples with 3µm CrN-coatings on silicon have been investigated with different but known biaxial stresses. A detailed description of the intrinsic stress determination and the nanoindentation procedures will be published elsewhere [3]. Here we want to concentrate on the analysis of the experimental data. At first, Young's modulus, hardness H and yield strength Y have been determined using classical normal nanoindentation. The results are presented in [1, table 1]. Now we want to follow the concrete procedure for one of those samples. For this we chose sample number 3 of [1].

Analyzing procedure step I: pure normal indentation with max. load p_0



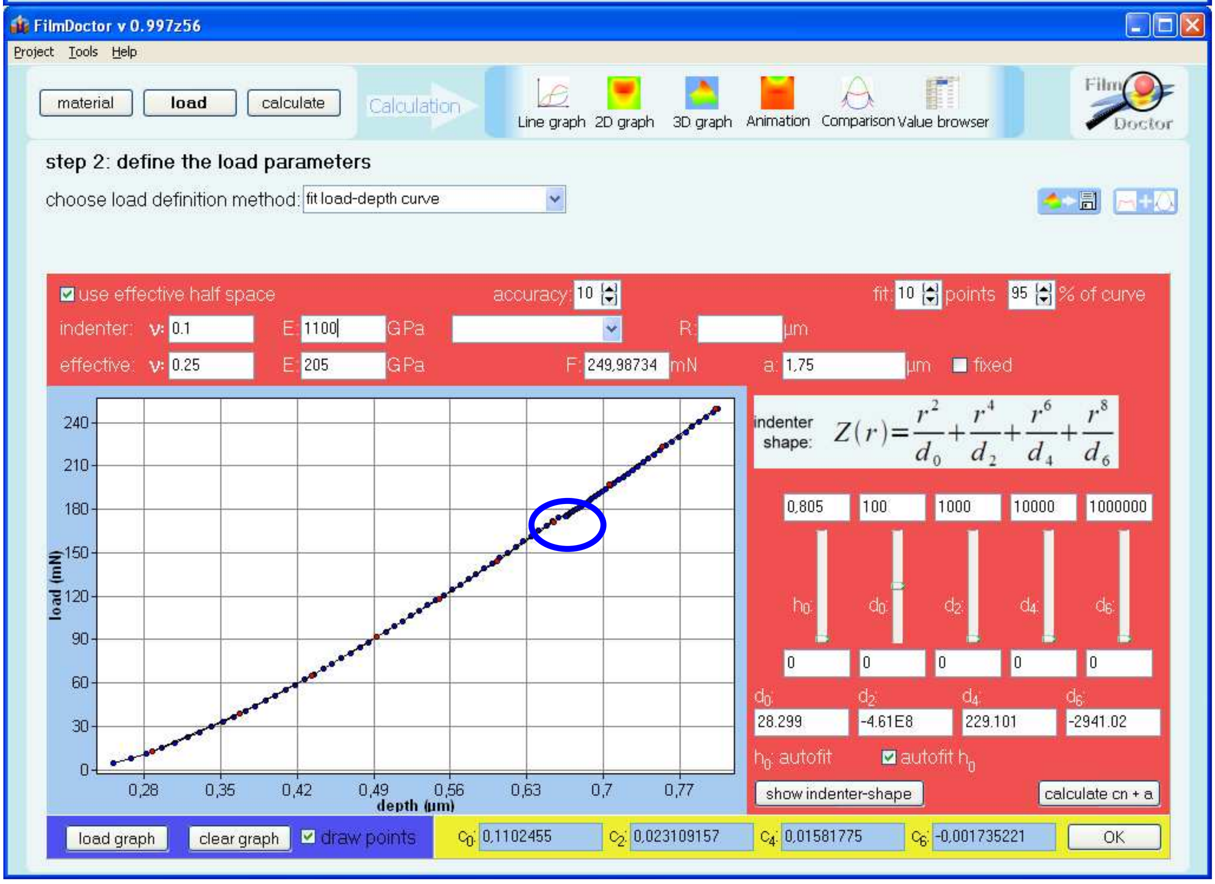


Fig. 1 and 2: Material data-input and fit of effective indenter to unloading curve

Using the software FilmDoctor, we first type in the material data Young's modulus, Poissons's ratio and thickness. As substrate we have silicon with a known Young's modulus of 165GPa and Poisson's ratio of 0.223. For the film we are estimating the Poisson's ratio and use the values determined by the means of the procedure described in [4]. Now we chose "fit load-depth-curve" from the load definition page, load the indentation curve and fit a paraboloid indenter to the same. The fit can be done by hand or automatically. The next step is the evaluation of the elastic field of the effective indenter in the moment of beginning unloading (maximum load p_0). So, after setting up the parameters for the calculation (fig. 3).

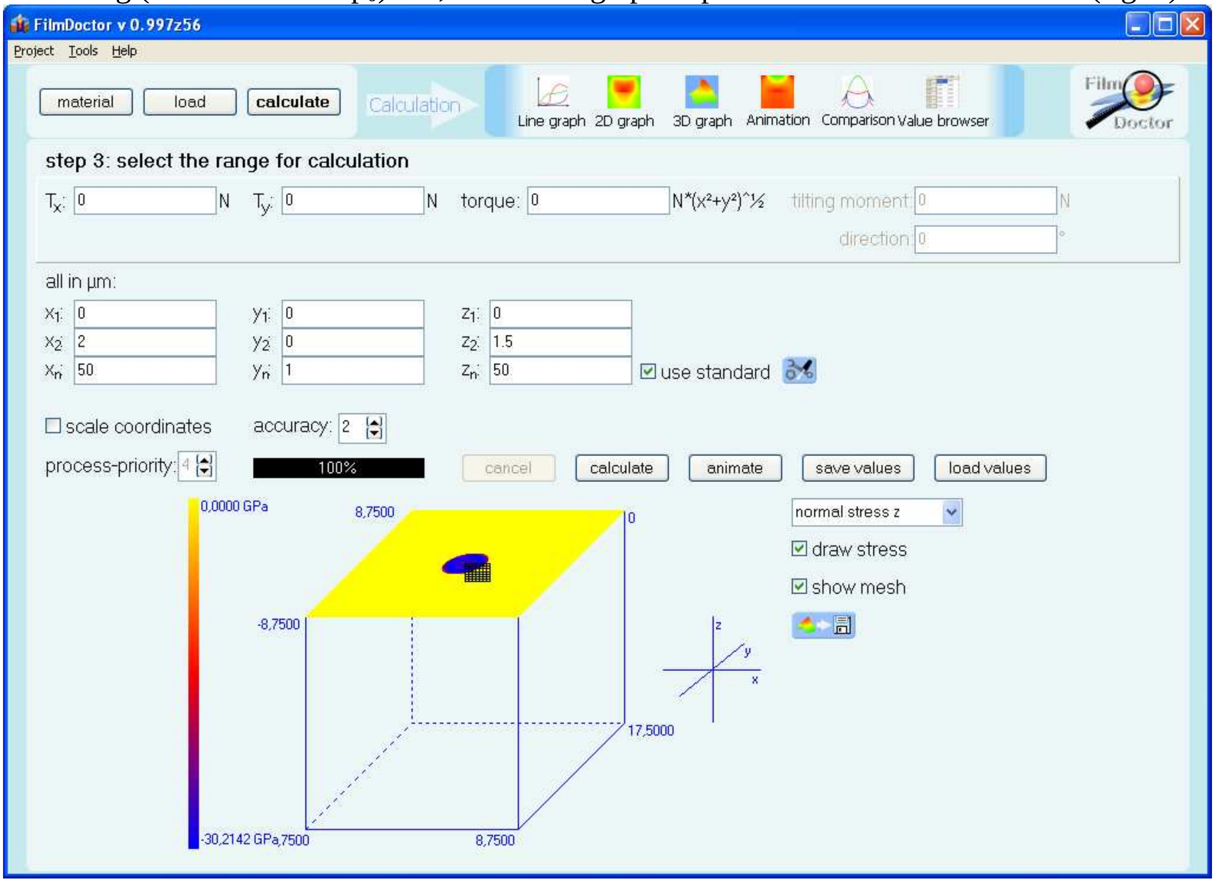
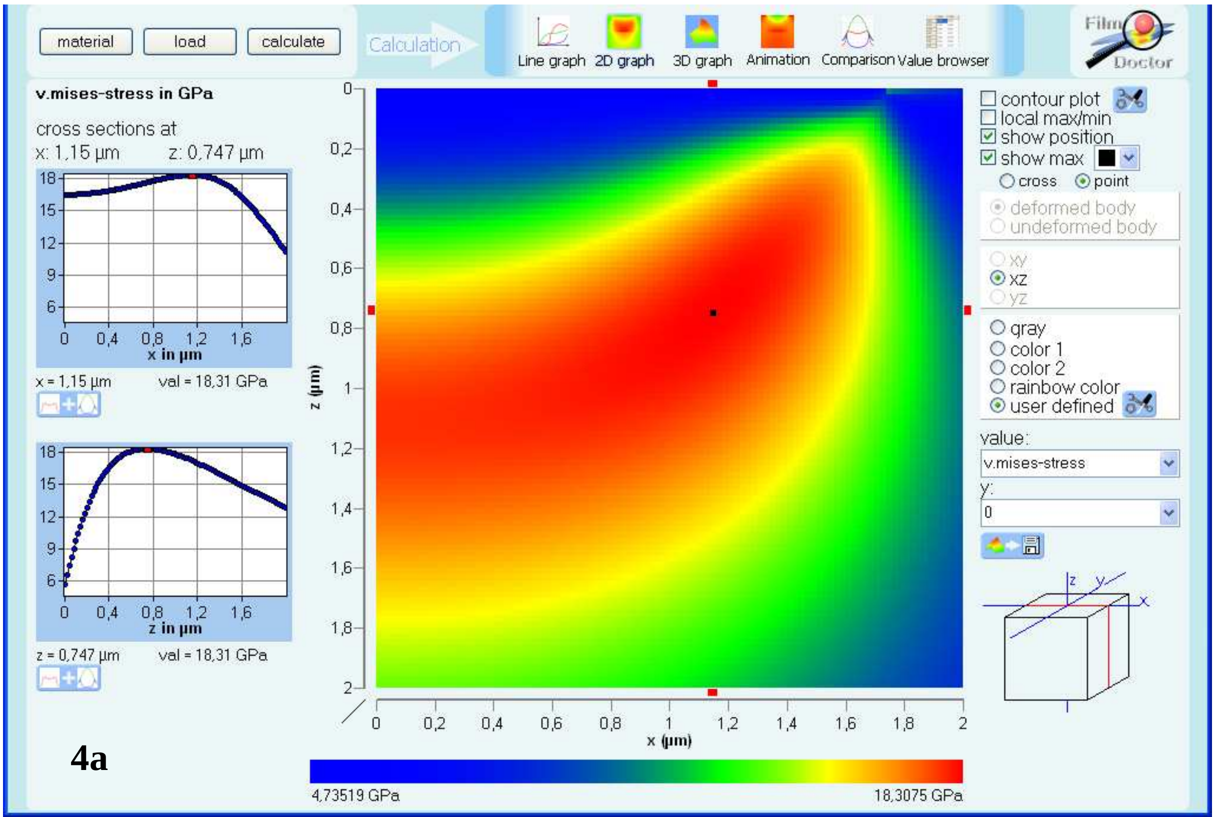


Fig. 3: Setting up the calculation-parameters and starting the evaluation

We find the von Mises Maximum (fig. 4 with 18.8357GPa), which is still mixed with the yet unknown intrinsic stress.



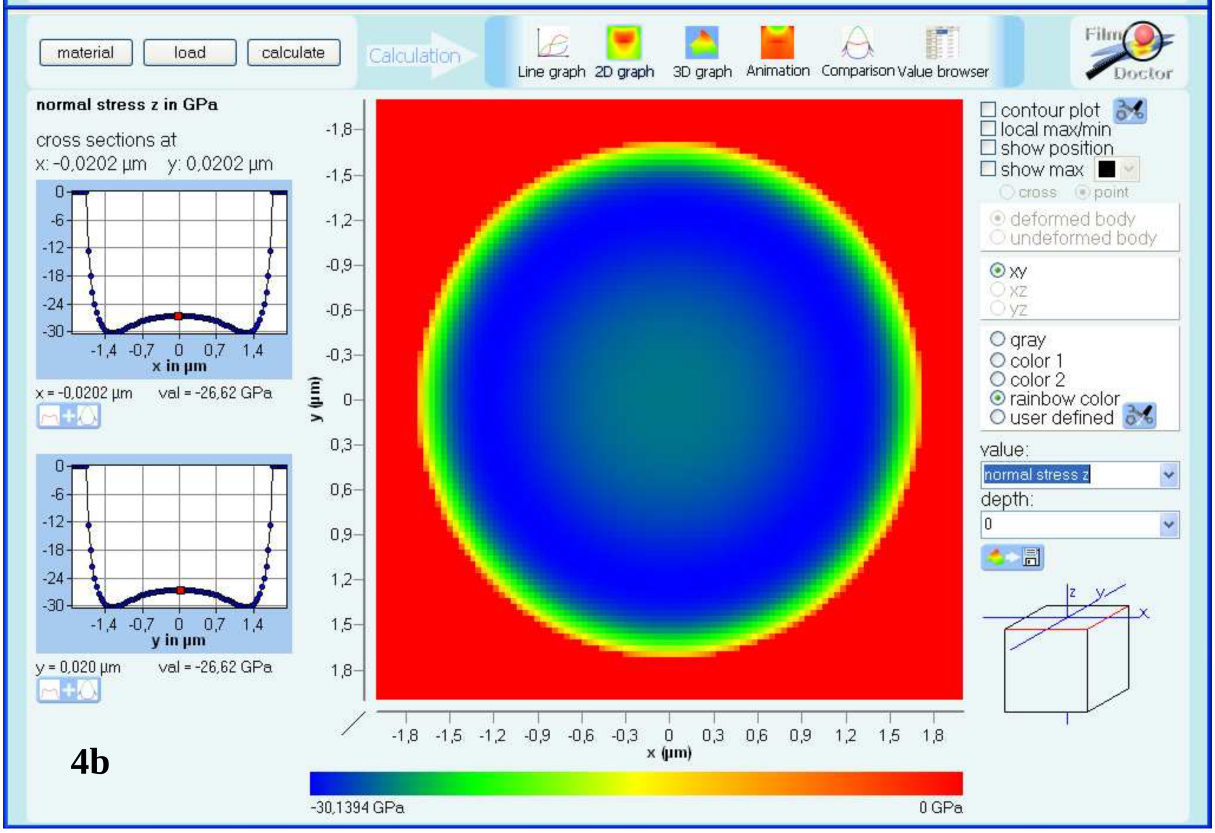


Fig. 4a: Resulting von Mises stress for the contact parameters given in fig. 2 at max. load p_0.
Fig. 4b: As the effective indenter produces a rather non-Hertzian normal surface pressure distribution (normal stress z), the von Mises maximum is to be found outside the axis of indentation.

Analyzing procedure step II: mixed normal and lateral indentation with maximum load 0.7*p_0

Now we reduce the normal load to 0.7*p_0 and start the lateral loading of the sample surface until again plastic flow respectively any other inelastic behavior can be observed. As effective indenter we either determine the new one for the reduced load by again fitting a paraboloid to the unloading curve after the lateral loading procedure or simply use the effective indenter from step I at reduced normal load. However, the latter is only possible when the surface shape of the inelastically deformed surface is not significantly changed during the lateral loading procedure, which would result in an incontinuity of the unloading curve at the position of the introduced load (blue ellipse in fig. 2).

The author wants to point out here, that instead of a lateral load also inclined normal indention should allow to obtain a second measurement sufficiently linear independent from the pure normal loading. However, this would require a well calibrated equipment clearly assigning lateral t_x and tilting load M_t to the indenter inclination and normal load.

Analyzing procedure step III: evaluation of the von Mises stress for the mixed normal and lateral indentation with normal load 0.7*p_0 and lateral load t_x

We assume that at a normal load of 0.7*p_0 and a lateral load t_x inelastic behavior has been detected. Now we evaluate the von Mises stress for this mixed loading situation. As described in [1] we have to take into account, that usually lateral or inclined indentation also produces indenter tilting. In the example considered here we found the following loading conditions:

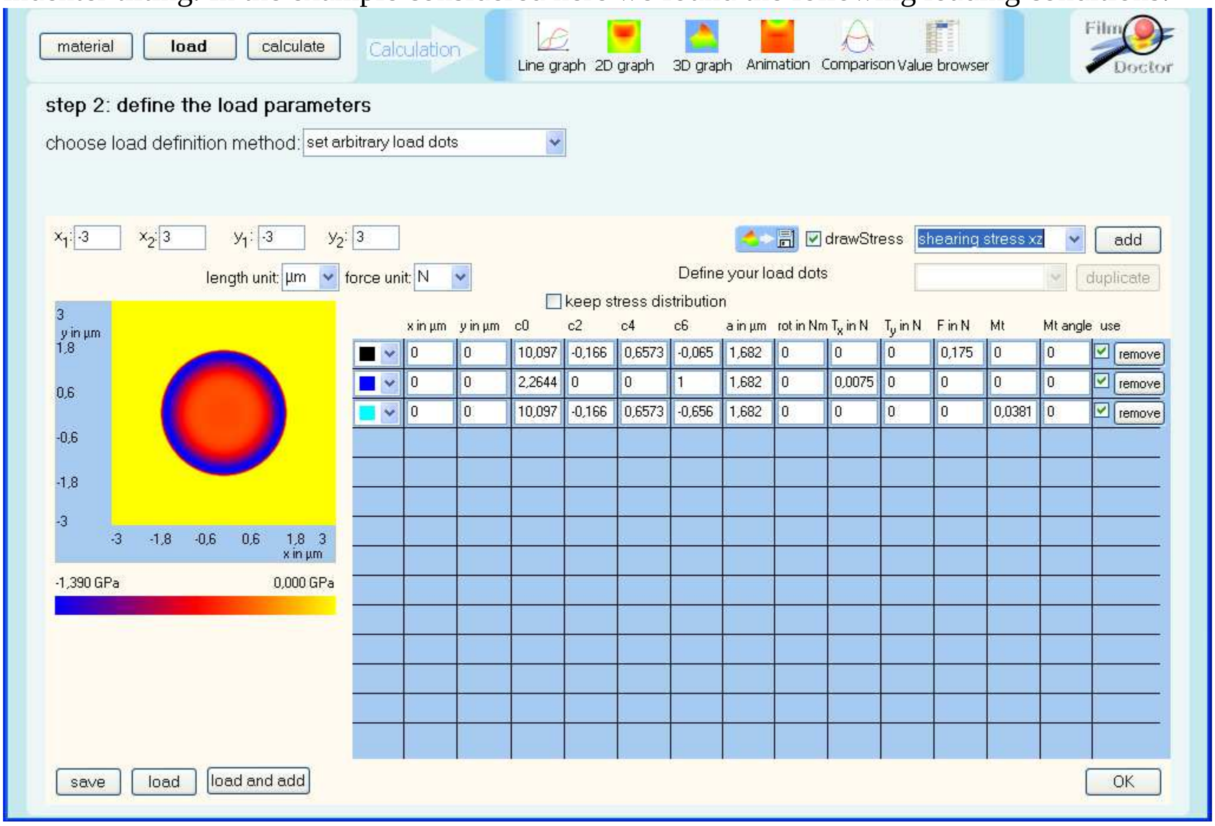


Fig. 5: Parameters for the mixed load evaluation

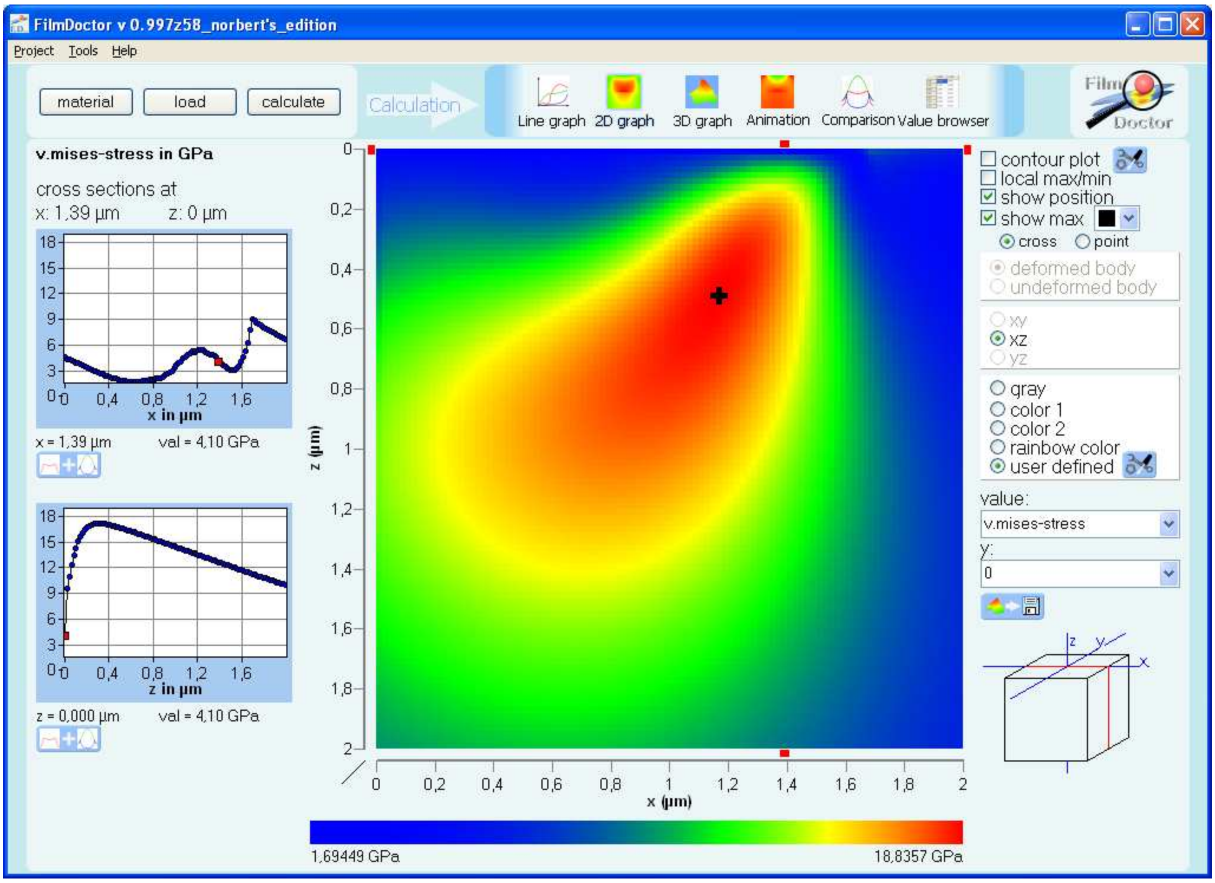


Fig. 6: Resulting von Mises stress for the mixed load situation.

Analyzing procedure step IV: evaluation of the "pure" yield strength and the intrinsic stress

From the equations given above we can easily obtain a formula for the intrinsic film stress σ_{rr}^{f}:

$$\sigma_{rr}^{f} = \frac{\begin{aligned}&-6\left(f_{xy}^{I}\tau_{xy}^{L} + f_{xz}^{I}\tau_{xz}^{L} + f_{yz}^{I}\tau_{yz}^{L}\right) + f_{xx}^{I}\left(-2\sigma_{xx}^{L} + \sigma_{yy}^{L} + \sigma_{zz}^{L}\right)\\ &+ f_{yy}^{I}\left(-2\sigma_{yy}^{L} + \sigma_{xx}^{L} + \sigma_{zz}^{L}\right) + f_{zz}^{I}\left(-2\sigma_{zz}^{L} + \sigma_{yy}^{L} + \sigma_{xx}^{L}\right)\\ &\pm\sqrt{\begin{pmatrix} 4\begin{pmatrix} f_{xx}^{I\,2} + f_{yy}^{I\,2} + 3\left(f_{xy}^{I\,2} + f_{xz}^{I\,2} + f_{yz}^{I\,2}\right) \\ -f_{yy}^{I}f_{zz}^{I} + f_{zz}^{I\,2} - f_{xx}^{I}\left(f_{yy}^{I} + f_{zz}^{I}\right)\end{pmatrix} \\ * \begin{pmatrix}\sigma_{M}^{crit\,2} - \sigma_{xx}^{L\,2} - \sigma_{yy}^{L\,2} - 3\left(\tau_{xy}^{L\,2} + \tau_{xz}^{L\,2} + \tau_{yz}^{L\,2}\right) \\ +\sigma_{yy}^{L}\sigma_{zz}^{L} - \sigma_{zz}^{L\,2} + \sigma_{xx}^{L}\left(\sigma_{yy}^{L} + \sigma_{zz}^{L}\right)\end{pmatrix} \\ + \begin{pmatrix}-6\left(f_{xy}^{I}\tau_{xy}^{L} + f_{xz}^{I}\tau_{xz}^{L} + f_{yz}^{I}\tau_{yz}^{L}\right) + f_{xx}^{I}\left(-2\sigma_{xx}^{L} + \sigma_{yy}^{L} + \sigma_{zz}^{L}\right) \\ + f_{yy}^{I}\left(-2\sigma_{yy}^{L} + \sigma_{xx}^{L} + \sigma_{zz}^{L}\right) + f_{zz}^{I}\left(-2\sigma_{zz}^{L} + \sigma_{yy}^{L} + \sigma_{xx}^{L}\right)\end{pmatrix}^{2}\end{pmatrix}}\end{aligned}}{2\begin{pmatrix} f_{xx}^{I\,2} + f_{yy}^{I\,2} + 3\left(f_{xy}^{I\,2} + f_{xz}^{I\,2} + f_{yz}^{I\,2}\right) \\ -f_{yy}^{I}f_{zz}^{I} + f_{zz}^{I\,2} - f_{xx}^{I}\left(f_{yy}^{I} + f_{zz}^{I}\right)\end{pmatrix}}. \qquad (1)$$

Assuming now only biaxial stress one simply has to compare the expected value for the unstressed case with the measured one σ_{M}^{crit} and could evaluate σ_{rr}^{f} using equation (1), which can be dramatically simplified due to the fact that within the coating $f_{xx}^{I} = f_{yy}^{I} = 1$ and all other $f_{ij}^{I} = 0$.

$$\sigma_{rr}^{f} = \frac{-\sigma_{xx}^{L} - \sigma_{yy}^{L} + 2\sigma_{zz}^{L} \pm \sqrt{4\sigma_{M}^{crit\,2} - 3\left(\left(\sigma_{xx}^{L} - \sigma_{yy}^{L}\right)^{2} + 3\left(\tau_{xy}^{L\,2} + \tau_{xz}^{L\,2} + \tau_{yz}^{L\,2}\right)\right)}}{2}. \qquad (2)$$

So the two measurements provide us with two equations we can solve with respect to σ_{M}^{crit} and σ_{rr}^{f} [5].

For the sample considered here we obtain σ_{M}^{crit} =16.6GPa and σ_{rr}^{f} =-2.03GPa, with the latter being in very good agreement with the value measured by other means (-1.9GPa, c.f. [3]). By taking the biaxial intrinsic stress fixed to its directly measured value of σ_{rr}^{f} =-1.9GPa one would obtain two possible values for the yield strength, namely 16.79GPa in mixed loading and 16.71GPa in the pure normal loading case.

From the small difference of these two values and the considerations presented in [5] one can easily deduce, that measurement of intrinsic stresses via nanoindenter requires a very high accuracy and well calibrated equipment. However, the procedure described here could also be used as a simple estimator for the maximum value the intrinsic stress can not exceed and thus, giving more precise error bars for the yield strength (respectively hardness) determined from nanoindentation data. This way big hardness or yield strength values only obtained due to huge intrinsic compressive stresses will not pass as absolute material properties. Such an information is of special importance when nanoindenter results from pure normal loading states are going to be used in applications with mixed loading conditions.

All evaluations have been performed using a special prototype of the software FilmDoctor [6].

References:

[1] N. Schwarzer, “Intrinsic stresses – Their influence on the yield strength and their measurement via nanoindentation”, publication of the Saxonian Institute of Surface Mechanics, online at www.siomec.de/pub/2007/001

[2] V. Linss, T. Chudoba, M. Karniychuk, F. Richter, Thin Solid Films, 494 (2006) 179-183.

[3] T. Chudoba, N. Schwarzer, V. Linss, Thin Solid Films, to be submitted

[4] N. Schwarzer: “Analysing system for the investigation of ultra-thin coatings – determination of their Young’s modulus, hardness and yields strength using the method of the ‘Effective Indenter’”, publication of the Saxonian Institute of Surface Mechanics, online at www.siomec.de/doc/2007/001

[5] N. Schwarzer, "Modelling of the mechanics of thin films using analytical linear elastic approaches", Habilitationsschrift der TU-Chemnitz 2004, FB Physik Fester Körper, published in the online-archive of the Technical University of Chemnitz available at: http://archiv.tu-chemnitz.de/pub/2004/0077

[6] FilmDoctor: software package for the evaluation of the elastic field of arbitrary combinations of normal, rotating and lateral loads of the type $\sim r^n \sqrt{a^2 - r^2}$ (with n=0,2,4,6), available from the internet at: http://www.siomec.de/downloads (contact: service@siomec.de).