

## The Oliver and Pharr Method for Coatings and M. Fuchs<sup>a</sup>, N. Schwarzer<sup>a</sup> Physical Scratch Test Analysis for Layered Materials

## Introduction to the Oliver&Pharr for Coatings (O&PfC<sup>®</sup>)

For the correct determination of Young's modulus and yield strength of a coating an extension of the classical Oliver and Pharr method [1] is necessary. This extension is based upon a completely analytical mathematical approach [2]. The principle scheme of this new method is presented at **www.siomec.de/O&PfC-demo**. Here it is shown on the example of some Fischerscope® measurements from Helmut Fischer GmbH & Co. KG, that even in those cases, where the so called Bückle rule can't be fulfilled at all and the experimentalist also faces bad foundation conditions, meaningful coating parameters can be extracted by the means of the "Oliver&Pharr method for coated materials" (O&PfC®). The corresponding software used for the evaluation is called Oliver&Pharr for Coatings (O&PfC<sup>®</sup>).

The software of Fischer<sup>®</sup> equipment is able to export all data as an O&PfC<sup>®</sup> project file (except material information), hence the complete evaluation (except input of material information) is processed automatically.

#### Step 1: Obtain classic measurement results

In order to get more information from your performed measurements, firstly, you can start where your measurement and classic evaluation ended: With the load-displacement curve and the results of the classic Oliver&Pharr method [1]. If your measurement equipment is able to export load-depth curves as ASCII files, you can directly import them to O&PfC<sup>®</sup>. In this case, the only things missing yet in this step are the results of the Oliver&Pharr method and the contact radius respectively contact area, both of which you can type in manually. In our example the sample was loaded with up to 20mN using a diamond indenter.

Now, the effective indenter is automatically fitted to the unloading part of your load-displacement curve by O&PfC<sup>®</sup>. Concerning calculation time: ask here for live demonstration.

#### Step 2: Material definition

Secondly, the geometrical and mechanical material parameters of the sample are required. Easy if your measurement equipment allows direct export of O&PfC® project files, like the Fischerscope<sup>®</sup>. Otherwise, the material structures can also be typed in by hand. Here, we investigated a 500µm thick Si substrate covered with a sub-micron hard layer of 790nm thickness.

> Caution: Measurements can be flawed! (e.g.: unclear foundation conditions, unknown residual stresses,

inclusions, defects, unsuitable sample holder etc.)

This can be taken into account by our model!

#### Step 3: Fit of the yet unknown coating Young's modulus

Then the Young's modulus of the layer in question is fitted to the indentation unloading curve, taking into account the effective indenter and the complete solution of the contact problem for layered materials (geometry of the system, parameters of additional layers etc.). As known from the classical O&P method the Poisson's ratio has to be estimated unless it is known.

The result of 289GPa for the coating Young's modulus is in very good agreement with previous measurements performed on an UMIS 2000 system [3].

#### Step 4: Evaluation results

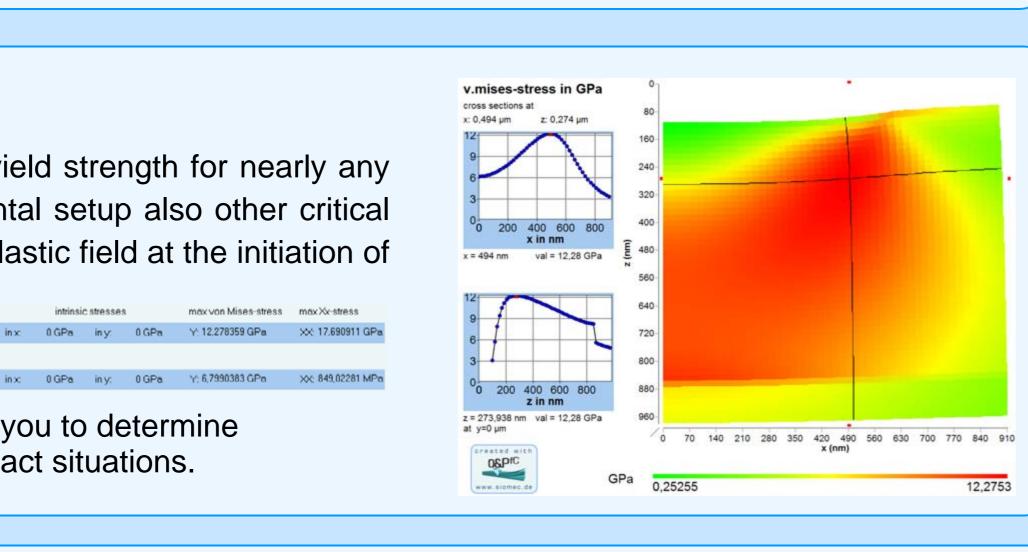
Finally, this Oliver&Pharr for Coatings method (O&PfC<sup>®</sup>) determines the yield strength for nearly any practical relevant material configuration. In dependence on the experimental setup also other critical parameters (like critical tensile stress) can be obtained from the effective elastic field at the initiation of unloading.

#### Conclusion

Your affordable Fischerscope<sup>®</sup> together with our software O&PfC<sup>®</sup> enables you to determine mechincal parameters of nano-thin film coatings and evaluate realistic contact situations.



Gain more physical information



edit database

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## Introduction to the Scratch-Stress Analyser (SSA<sup>®</sup>)

The SIO developed a special software solution (SSA<sup>®</sup>), on the one hand, for the automatic physical analysis of your scratch tests and, on the other hand, to extract more mechanical parameters of the coating. Therefore, it is based on the extended Hertzian theory [2] and involves the Oliver&Pharr for Coatings method [4] allowing a completely analytical mathematical approach. This software will be available in 4 different standard versions and customer optimized ultimate versions. For more information about the different features please visit www.siomec.de/SSA. The following example, which shows you the physical analysis of a scratch test, is based on a nano-scratch measurement of Micro Materials Ltd using their sophisticated NanoTest<sup>™</sup> [5]. The sample consists of a hard nanocomposite TiN/Si<sub>3</sub>N<sub>4</sub> coating (h=780nm, E=490GPa, v=0.2), which has been deposited on a Si-substrate by dual ion beam assisted deposition (IBAD).

## Step 1: Material definiton

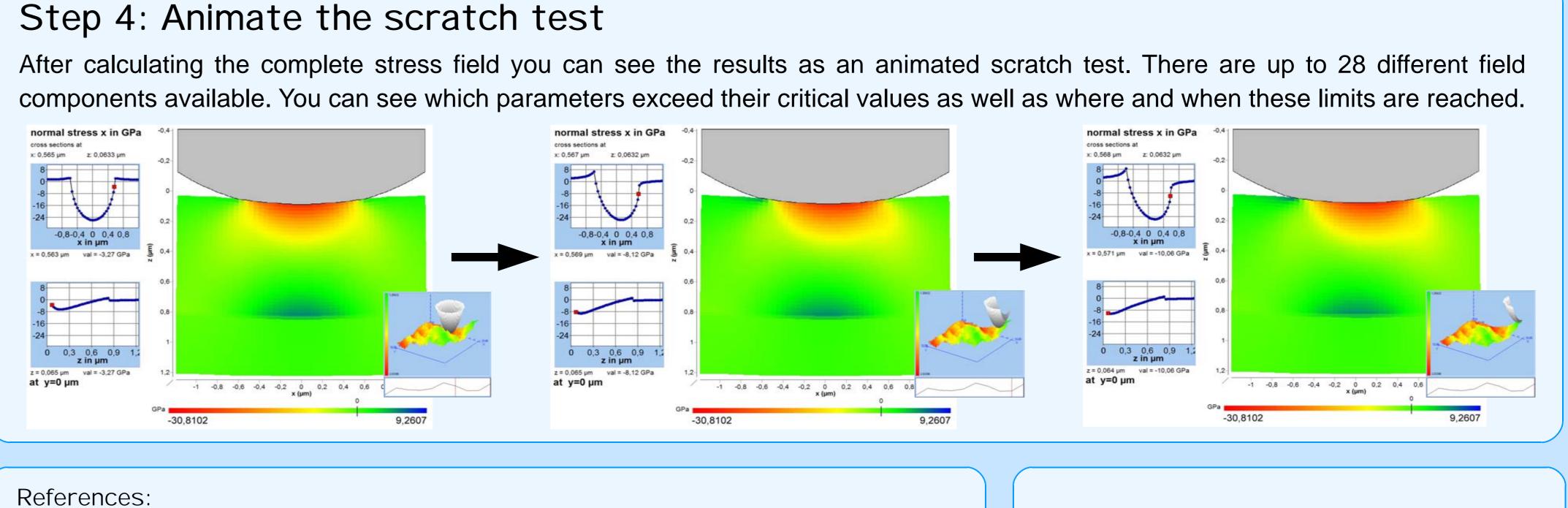
Firstly, define the material properties of your sample. You can either input the poisson's ratio and young's modulus or choose the values from our database for layers and substrate. In the SSA<sup>®</sup> ultimate version you can define up to 100 layers and gradient layers, optionally with (graded) intrinsic stresses.

## Step 3: Scratch measurement dimensioning

With the special SIO-scan procedures (see flyer) you have control over the contact area at every position of your probe. This also allows the application of the "concept of the effectively shaped indenter" (see figure) or even the continuous stiffness method with the full power of the extended Oliver&Pharr method for coatings (see on the left side). Hence, you can simultaneously determine so far unknown mechanical parameters of your sample (e.g. Young's modulus of a intermediate layer) with the measurement data of your scratch test.

# Alternatively: Scratch parameter definition

If you would like to simulate a virtual scratch measurement, you can specify the load settings (or fit the effective indenter to one of your scratch tips as shown in the figure) and scratch parameters directly. Thus, the number of time-consuming but necessary real scratch tests can be reduced dramatically.



| Ref        | ere      |
|------------|----------|
| [1]<br>[2] | W.<br>N. |
| [3]        | 11<br>V. |
|            | Va       |
| [4]        | N.<br>an |
| [5]        | B.<br>na |
| a)         | Sa       |

### Step 2: Obtain surface topography

Secondly, you can import the topography of your sample. It can be obtained from a linear tactile pre-scan using a no-load (profilometers) or, better, as an 3D topography from AFM or other methods.

I.C. Oliver and G.M. Pharr, J. Mater. Res. 7, 1564 (1992)

 Schwarzer: "The extended Hertzian theory and its uses in analysing indentation experiments", Phil. Mag. 86(33-35) 21 Nov -1 Dec 2006 5153 – 5767, Special Issue: "Instrumented Indentation Testing in Materials Research and Development" 2. Linss, N. Schwarzer, T. Chudoba, M. Karniychuk, F. Richter: "Mechanical Properties of a Graded BCN Sputtered Coating with AryingYoung's Modulus: Deposition, Theoretical Modelling and Nanoindentation", Surf. Coat. Technol., 195 (2005) pp 287 – 297 I. Schwarzer: "An Extension of the Oliver and Pharr Method to Ultra-Thin Structures, Coatings, Functionally Graded Coatings nd Multilayer Systems", online archives of the Saxonian Institute of Surface Mechanics www.siomec.de/pub/2007/010 Beake, V. Vishnyakov, R. Valizadeh, J. Colligon: "Influence of mechanical properties on the nanoscratch behaviour of hard anocomposite TiN/Si3N4 coatings on Si", J. Phys. D: Appl. Phys. 39 (2006) 1392–1397 axonian Institute of Surface Mechanics, Ummanz, Germany





