APPLICATIONS BULLETIN

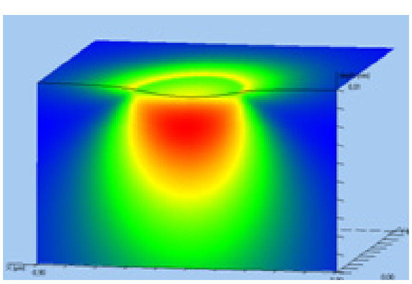
Physical characterization of coated surfaces Part I : Instrumented Indentation

Instrumented indentation and scratch testing are well-established procedures for mechanical characterization of surfaces. The Ultra Nanoindentation Tester (UNHT) and the Nano Scratch Tester (NST) are very sophisticated instruments using advanced measurement techniques to provide accurate data [1].

A very common way to analyze indentation data is the "Oliver & Pharr Method" ([2], [3]) which utilizes the unloading-part of the load-displacement curve to extract mechanical material properties like the Elastic Modulus E_{IT} and Hardness H_{IT}. As this model assumes the sample to be a monolithic half-space, it cannot consider the influence of the substrate in the combined response of the coated surface to the load-induced stresses and deformations and, thus, the results are effective values of the whole sample (E_{eff}, H_{eff}). Therefore, it is very difficult if not impossible, to obtain the actual mechanical properties of the coating (E_C, H_C) utilizing this classic "Oliver & Pharr Method". This model has been extended in many ways and substantiated with a general solution for contact situations on arbitrarily layered materials [4, 5, 6]. This "Oliver & Pharr extended for coatings" model allows a physical analysis of indentation measurements, because it does not only calculate the actual values of generic material properties of each layer of a coating like the elastic modulus (E_{C1}, E_{C2}, etc.), but also the yield strength (Y_{C1}, Y_{C2}, etc.) as it calculates the complete elastic contact field at the point of initial unloading.

As a result of the generality of this model it can also be applied to scratch test data, because it takes the additional load component (lateral load) and measurement effects (e.g. tilting of the stylus) into account [7]. Hence, one is no longer compelled to non-generic and non-physical parameters as scratch hardness or critical loads (LC1, LC2, and LC3) using such a physical analysis of scratch tests, but can now get generic and, thus, more universal material properties like the critical stresses of each fracture mode (I, II, and III). Fig. 1 illustrates the effect on the Von Mises stress distribution of those differences between the classic and extended model, even though the classic "Oliver & Pharr Method" does not even enable one to calculate the complete elastic field and its stress-strain components.

a



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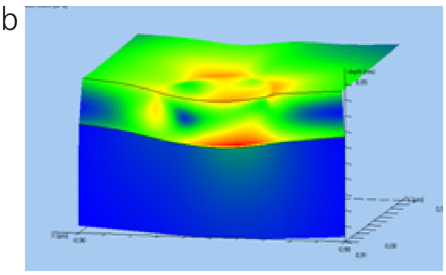

Fig. 1: Von Mises stress distribution of an assumed monolithic halfspace without considering additional loads and measurement effects (a) in comparison to the Von Mises stress distribution (b) taking all those conditions into account like the "Extended Oliver & Pharr Method".

It will be shown how this model can be used to physically analyze both indentation measurements and scratch tests on very different surface structures, namely a 10 µm thick double-layer tribological coating on a Tungsten Carbide (WC) substrate and a 250 nm thin optical anti-reflex (AR) coating on a polymer substrate (Table 1). The material compositions, layer thicknesses, and elastic moduli of the substrates have been determined in advance – the latter by means of indentation into a bulk sample. These systems are not only different with respect to their coating thicknesses, but also with respect to their mechanical structure: the WC substrate is much stiffer than the polymer substrate. Hence, it is assumed and will be shown that the substrates differently influence the effective mechanical parameters obtained by the classic "Oliver & Pharr Method".

Table 1: Known sample parameters

Sample TR

Structure (top down)	Compostion	Thickness [µm]	E [GPa]	Y [GPa]
Layer 1	$Al_{1.4}Cr_{0.6}O_3$	3.3	-	-
Layer 2	$Al_{0.7}Ti_{0.3}N$	6.7	310.2	30
Substrate	WC	-	564	22

Sample AR

Structure (top down)	Compostion	Thickness [µm]	E [GPa]	Y [GPa]
Layer 1	SiO_2	0.25	-	-
Substrate	PMMA	-	4	12

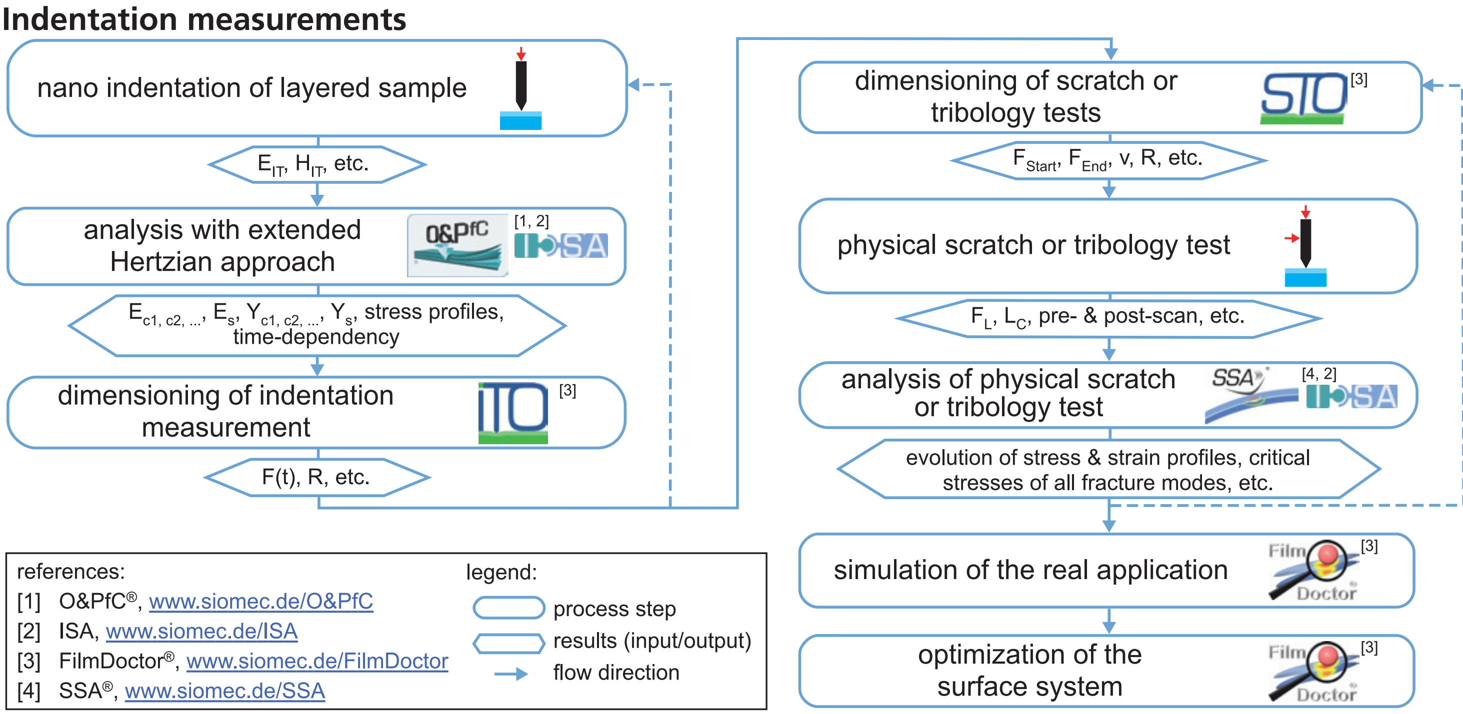

Fig 2 A flow chart of the procedure of mechanical characterization and optimization of arbitrary structured surfaces

Dimensioning of indentation measurements

Before an indentation measurement on a coated substrate is performed, it should be – as any physical experiment – properly dimensioned in order to obtain as much information from the sample constituent of interest (in this case the coating) as possible. As the same consequently holds for the subsequent scratch test, this measurement procedure can be summarized as a scheme as shown in Fig. 2. Note that the mechanical properties of the coating are assumed to be completely unknown in this flowchart, because the procedure begins with a non-dimensioned indentation into the coated substrate in order to calculate first – maybe uncertain – values of its mechanical parameters EC1 and YC1. These preliminary values are utilized for the first dimensioning of the final indentation measurement. If this dimensioning showed that the sensitivity of this first measurement is focused on the coating and, thus, the uncertainty of the results is sufficiently low, the first phase of this mechanical characterization procedure is finished.

Alternatively, values of Elastic Modulus and Poisson's ratio can be taken from literature in order to start with a rough dimensioning before undertaking the first indentation measurement [8]. If the actual coating properties are fairly close to the used literature values it might save one a non-dimensioned measurement. However, it would be beyond the scope of this work to explain the dimensioning process in detail, so it will only be shown later in next chapter "Physical analysis of indentation measurements" how to check whether a performed measurement was properly dimensioned.

Physical analysis of indentation measurements

As a result of an indentation measurement performed by the UNHT, measurement data including the results of the classic "Oliver & Pharr Method" will be output in the "Oliver&Pharr for Coatings" (O&PfC) project file format with the file extension "fdop". Measurement data in such a format can be directly opened by the software FilmDoctor® Studio [9], O&PfC® [10], as well as ISA [11] as shown in Fig. 3 in order to start the physical analysis.

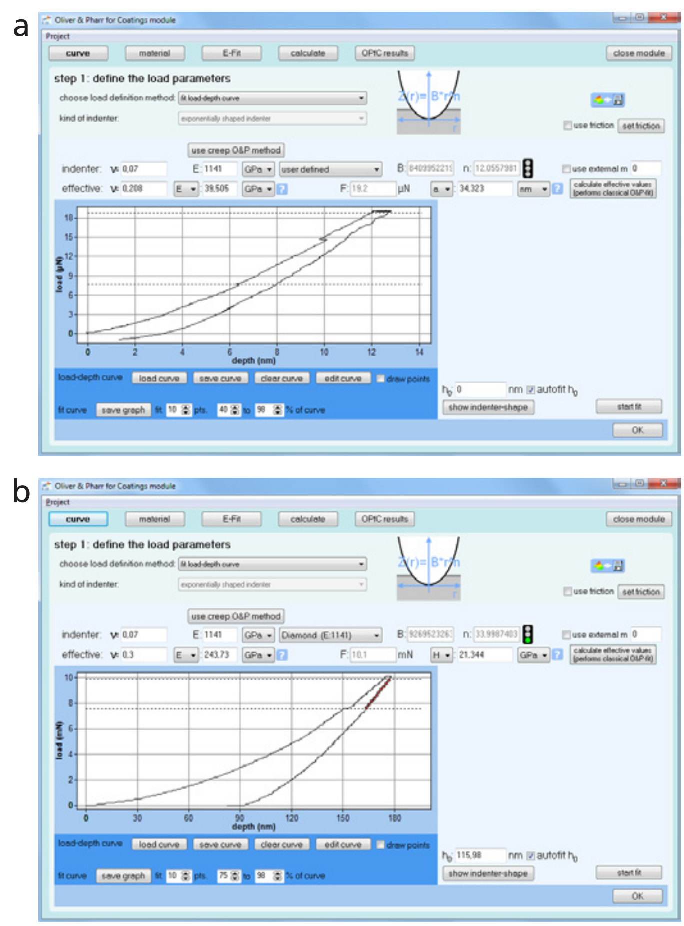

Fig. 3: Measurement data of the AR sample (a) and TR sample (b) as exported by UNHT® loaded into the FilmDoctor® Studio to begin the physical analysis.

Note that both measurements seem to be well dimensioned for the coating of interest as the contact radius, a, of 34 nm and 382 nm for the AR and TR samples, respectively, are well below the respective layer thicknesses of 250 nm and 3.2 µm – almost only 10% of the layer thickness which is a first indication for a well-dimensioned measurement.
Now the software follows the corresponding steps:
1. fits a power-law function to the unloading part of the load-displacement curve,
2. computes the best effective indenter as described in [3], and
3. calculates the distribution of normal stress in indentation direction for a given number of fit points within the fit range.

Secondly, the surface structures have to be defined (Fig. 4) since FilmDoctor® must take into account the actual structure of the sample in order to determine the influence of the other structure constituents on the measurement information. This other material information is determined in advance. For instance, the elastic modulus of the substrate has been measured on a bulk sample of the substrate material and the layer thicknesses have been determined with the CSM Instruments Calotest.

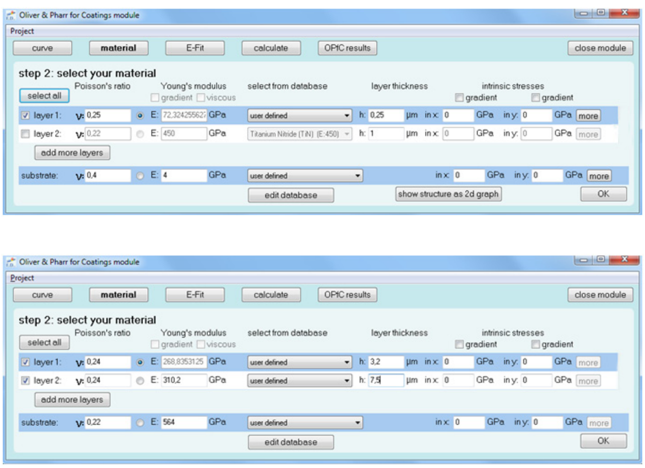

Fig. 4: Sample structure definition by means of number of layers, Poisson's ratio, Young's modulus, and layer thickness for the AR sample (a) and TR sample (b). As can be seen from these figures, more layers, gradient layer structures, and intrinsic stresses can be defined if applicable.

Thirdly, the software calculates the actual elastic modulus E_{IT} of the layer in question utilizing not only the measurement information but also the previously defined material structure information. The results are summarized in Table 2. The difference between the effective elastic modulus being 39.5 GPa for the monolithic halfspace assumed by the classic "Oliver & Pharr Method" and the actual elastic modulus of the layer being 70.3 GPa is as significant as expected due to the thin layer thickness. But note that despite the difference between effective and actual elastic modulus for the relatively thick $Al_{1.4}Cr_{0.6}O_3$ top layer on the TR sample is fairly small, E^{eff} is slightly influenced and, thus, increased by the stiffer underlying interlayer and substrate. In summary, while E is underestimated by the classic "Oliver & Pharr Method" for the AR coating due to the extremely compliant substrate, E is overestimated by it for the top layer of the TR coating due to the stiffer underlying structure.

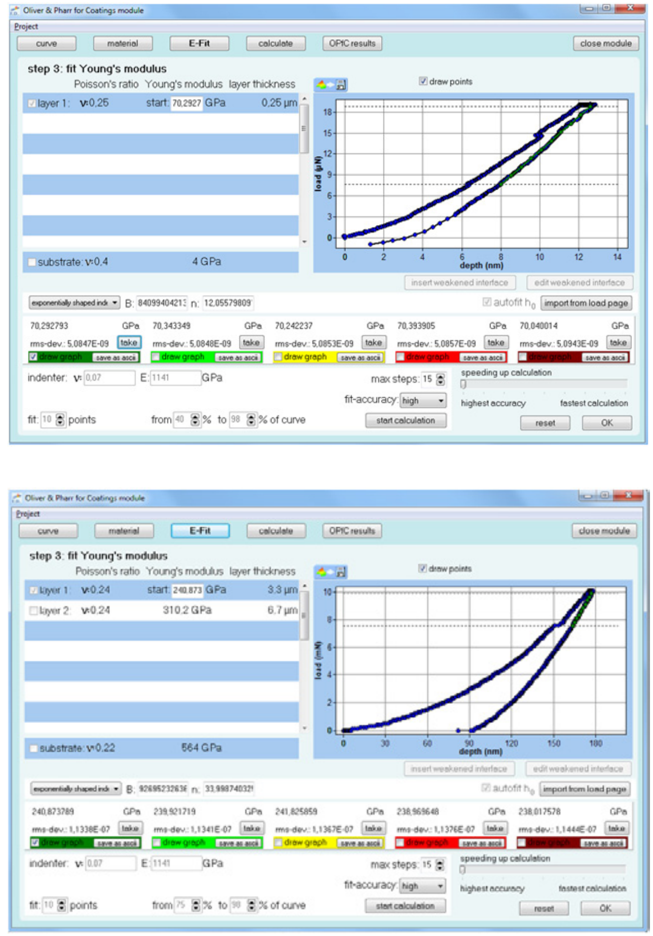

Fig. 5: The true elastic modulus of the layer of interest has been calculated by FilmDoctor® Studio and is shown in the left-hand panel (green underlined)

Although the so called "10% rule" or "Bückle rule", which proposes that the ratio of maximum indentation depth to layer thickness should be less than 10%, is fulfilled as the maximum indentation depth is about 13 nm on the AR sample and 170 nm on the TR sample (both approximately 5.2% only), the calculation of E_{IT} by means of the classic "Oliver & Pharr Method" fails in both cases. Hence, this rule is not valid for the determination of Young's modulus in that case!

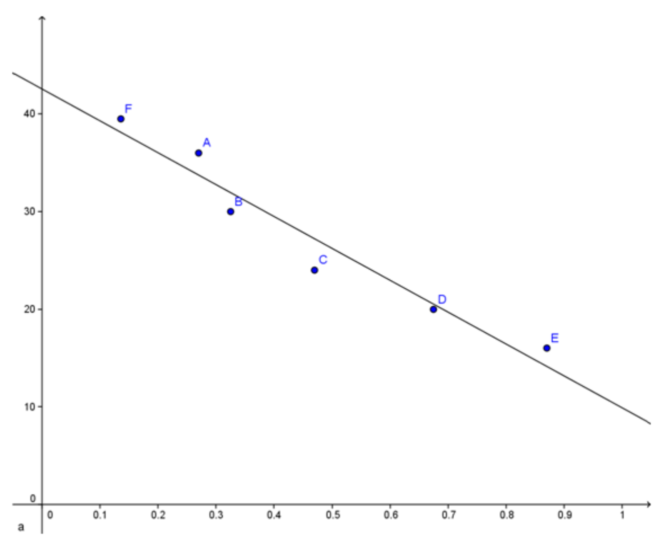

Fig. 6: Calculation of the Young's modulus of the AR coating (as recommended by ISO 14577) results in 42.5 GPa what is undoubtedly far too low.

Please note, that ISO 14577 recommends (in simple words) to conduct a series of measurements at different loads, plot the resulting effective elastic moduli as a function of ac/h, and linearly extrapolate them to ac/h = 0. This would result in an elastic modulus of the AR coating of 42.5 GPa as shown in Fig. 6, still much too low. Therefore, the ISO 14577 is not perfectly adapted for such sophisticated surface structures, either!

a

b

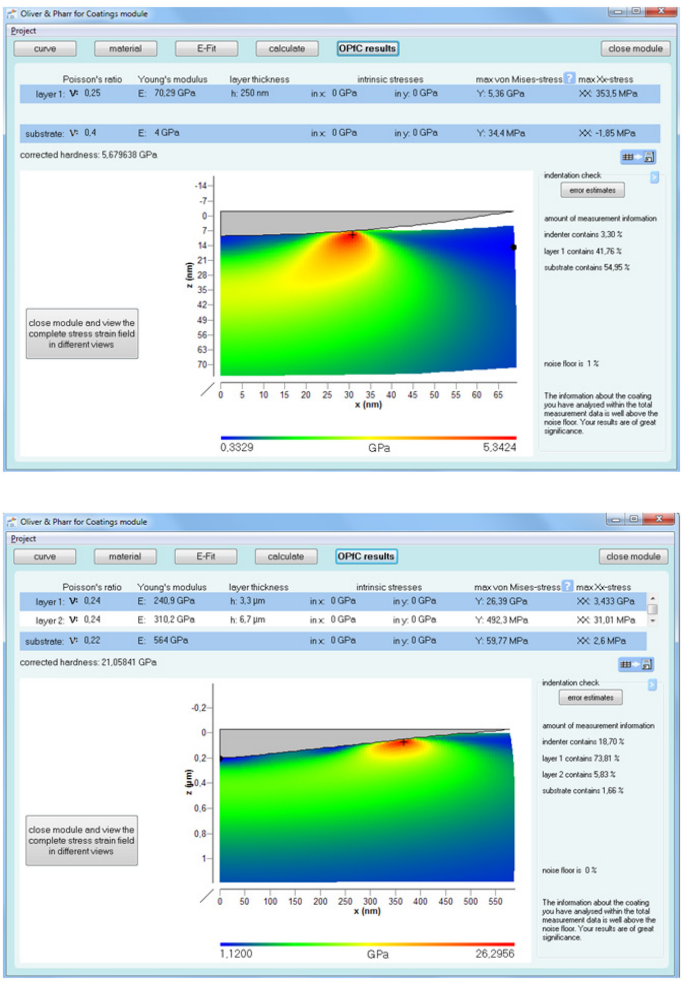

Fig. 7: Results of the physical analysis by FilmDoctor® for the AR sample (a) and TR sample (b) including the elastic modulus E, yield strength (if applicable), distribution of von-Mises stress as cross section through the sample from the center of indentation to the contact edge, and the share of measurement information for each constituent.

Finally, FilmDoctor® Studio calculates 28 field components of the complete elastic field at the beginning of unloading and, therefore, allows one to determine also the yield strength Y (or σY) which is the maximum Von Mises stress in the layer of interest if plastic flow happened only in this sample constituent. Fig. 7 shows, among other results, the distribution of von-Mises stress as cross section through the sample at the sample surface and center of indentation. It is obvious that the stress concentrates almost only in the top layer as both stress plots show only a section of the top layer in depth. Therefore, on the one hand, the maximum of von-Mises stress is equal to the yield strength which is 5.4 GPa and 20.7 GPa for the AR coating and top layer of the TR sample, respectively, because plastic deformation happened according to the residual contact depth shown by the load-displacement curves. On the other hand, this result supports the earlier mentioned indication that both measurements are well-dimensioned.

Conclusion

This is also supported by the calculated share of measurement information as 41.8% and 75.5% for the AR coating and TR top layer, respectively, shown in the right column of Fig. 7, while the noise floor of 1% is sufficiently low in both cases.

	Sample TR Layer 1 ($Al_{1.4}Cr_{0.6}O_3$)	Sample AR Layer 1 (SiO_2)
E_{eff} [GPa]	261.7	39.5
E_{C1} [GPa]	240.9	70.3
Y [GPa]	26.4	5.4
H_{eff} [GPa]	22	5.2
H_{C1} [GPa]	21.1	5.7

Table 2: Summary of calculated properties for each sample

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