CAPABILITIES OF THE LOAD AND PARTIAL UNLOAD TECHNIQUE

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Abstract − The load and partial unload technique adds partial unload processes to the load curve. The mechanical properties like elastic modulus can be determined for each of these unload curves. This allows for a faster depth dependent measurement of the characteristic quantities. If the elastic modulus of a material is known and not dependent of the depth, an indenter shape correction can be performed using this technique.

The results of such a correction are compared with those gained using a correction based on the Martens hardness of the material. The elastic modulus E_{IT} as well as the Martens hardness H_M are corrected with both methods.

The results of both approaches are comparable down to an indentation depth of 100 nm. A Fischerscope HM2000 was used to perform the measurements.

Keywords: Load and partial unload technique, elastic modulus, Martens hardness

1. INTRODUCTION

The depth dependent evaluation of quantities like elastic modulus E_{IT} or indentation hardness H_{IT} is an important issue for the measurement of compositions and thin coatings. One possibility to determine these values is the load and partial unload technique.

Due to the fact that the measurement consists of a series of indentation tests at the same position, the results correspond to the results from a standard indentation test. Compared to single indentation tests at several positions the approach saves a lot of time.

This method also offers the possibility to perform an indenter shape correction based on the elastic modulus as explained for example in [1], [2], [3]. Additionally, the Martens hardness is also calculated precise after this correction. The result is compared with a correction using the Martens hardness. According to [1], both methods are of equal value. This will be verified in this paper.

The measurements were performed using a Fischerscope HM2000 [4]. Two different methods for the time management for the load and partial unload measurement are explained in chapter (2).

Chapter (3) explains the realisation of the indenter shape corrections using the Martens hardness and the elastic modulus and compares both methods. Advantages and disadvantages of the methods are discussed.

Chapter (4) shows the results of these corrections for several materials.

2. TIMING OF THE LOAD AND PARTIAL UNLOAD CYCLE

2.1. A load and partial unload cycle

A standard indentation test (without creep) consists of the load step and the unload step. For the load and partial unload measurement, three steps for each single cycle can be distinguished: The load is increased to a certain value (pure load step), then reduced to a fraction of the maximum value (unload step). Then the load is increased to the previous maximum (load step) and further to the next, higher maximum load value (pure load step). The load and the pure load step can be merged to an entire load step (see Fig. 1a for a schematic plot of load vs. time and Fig. 1b for a depth vs. load plot).
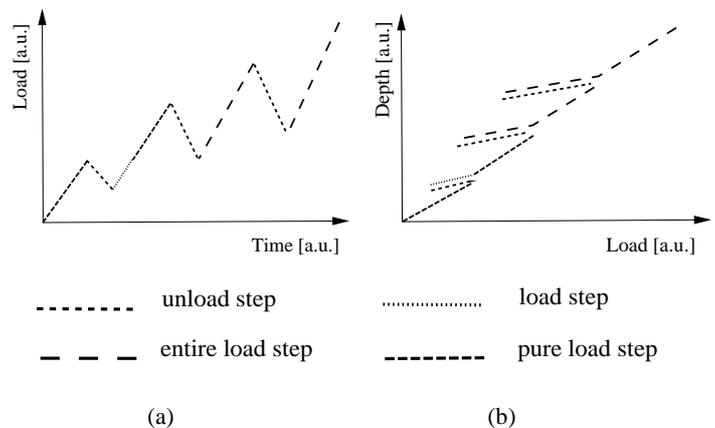

Fig. 1: Steps in a load and partial unload cycle: (a) load and unload steps in a time plot, (b) indentation depth in a load plot. One can differ between three steps for one cycle. The load step and the pure load step can be combined to an entire load step.

2.2. Different time managements

The results of an indention test can be affected by the time during the load application. The load and partial unload technique is a series of these measurements. Therefore, the same time dependence can be seen for some materials during these measurements.

Two different time management methods are presented [5]. They are called standard ESP method and modified ESP method (ESP: Enhanced Stiffness Procedure, software specific name for the load and partial unload measurement).

The decrease time (unload step) is specified and constant for each step of the measurement cycle for both methods.

For the standard method, the total measurement time is given. Each single entire load step takes the same time. Due to the increasing duration of the load steps (because of the increasing load), the pure load steps decrease in time and the last ones at high load are very short (entire load step = load step + pure load step = const., load step$_{i+1}$>load step$_i$). If the pure load steps are summed up and the load is plotted versus the time of these steps, the timing does not correspond to the timing of a standard indentation test. Different results compared to the standard tests are possible.

On the other hand, pure load steps of the modified ESP mode are chosen in such a way that the sum of these steps correspond exactly to the timing of a standard indentation test. Therefore the duration of the load steps increases as well as the length of the pure load steps (pure load step$_{i+1}$>pure load step$_i$, load step$_{i+1}$>load step$_i$) and more time is needed for the entire load step. The maximum number of cycles in a given time is reduced.

Fig. 2 shows the load vs. time of the standard ESP and modified ESP mode in a schematic plot and for measured data. Fig. 3 shows the measured depth vs. load for both methods on acrylic glass.

If the timing does not affect the measurements, the standard method can be chosen. To avoid possible effects, all results presented in this paper were determined using the modified mode.
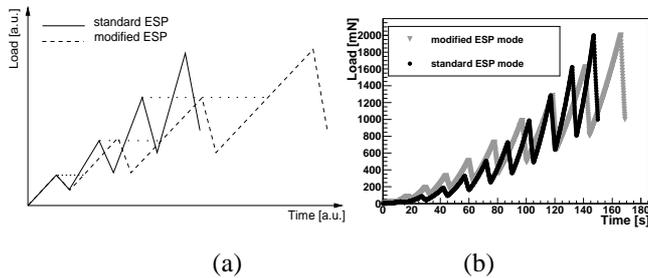

Fig.2: Different time management for standard and modified ESP: (a) schematic plot (not to scale), (b) data from measurement.

3. INDENTER AREA FUNCTION

The tip of a Vickers or Berkovich indenter is not perfect. However, the equations to calculate the hardness or the elastic modulus assume a geometrical perfect body. Thus, the formulas lead to incorrect results and have to be corrected at least for small indentation depths.

Different approaches are possible: direct measurement using an atomic force microscope (AFM) as well as two indirect measurements [1]. The latter are based on the idea that for some reference materials a measured quantity like the elastic modulus or the Martens hardness is independent of the indentation depth. For the presented measurements, a BK7 glass was used for correction as it fulfils the requirements. The elastic modulus and the Martens hardness for other materials were corrected using the resulting parameters.
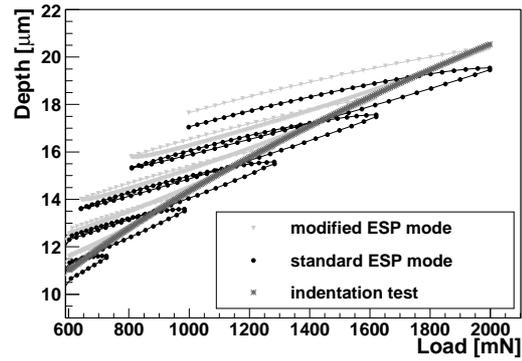

Fig.3: Different time management for standard and modified ESP measurement.

3.1 Hardness correction

The Martens hardness H$_M$ is calculated as [1]:

$$H_M = \frac{F}{A_s(h_{max})} = \frac{F}{26.43\,h_{max}{}^2}. \qquad (1)$$

A$_s$ is the surface area at an indentation depth h$_{max}$. The geometrical factor to calculate the area for Vickers and Berkovich indenters is 26.43. For each load F and corresponding indentation depth h$_{max}$, a Martens hardness H$_M$ can be calculated. With one measurement a potential hardness gradient of the material can be determined.

Because of the tip rounding, measurements for small depths results in an increased hardness value. The depth is corrected such that the resulting Martens hardness is constant. The absolute hardness value is obtained from the data for large depths, no table value is needed. The elastic modulus is also calculated using the corrected depth.

The method has the advantage that hundreds of measuring points allow a stable fit from small depths up to the maximum depth with only one measurement cycle. This method is very fast. The disadvantage is that the hardness value can depend on the measuring conditions due to the fact that it is not a matter constant. The conditions have to be chosen declared. It will be shown that the results for this correction are comparable to the results from a correction of the elastic modulus. The measurements are limited down to a depth of 100 nm because of the experimental setup.

3.2 Elastic modulus correction

Another way to correct the indenter shape is a correction of the elastic modulus. To calculate the elastic modulus E$_{IT}$, the contact depth h$_c$ is needed instead of the maximum depth h$_{max}$:

$$h_c = h_{max} - \varepsilon C_s F_{max}. \qquad (2)$$

Here ε is a geometrical factor depending on the indenter, (typically 0.75 for Vickers or Berkovich indenters) and C$_s$ is

the contact compliance. One can calculate the projected area A_p:

$$A_p = 24.50 \, h_c^2, \qquad (3)$$

where 24.50 is a factor depending on the geometry of the Vickers indenter. Now the reduced modulus E_r can be calculated:

$$E_r = \sqrt{\frac{\pi}{A_p}} \cdot \frac{1}{2 C_s} . \qquad (4)$$

Knowing the Poisson ratios of the material ν and the indenter ν_i as well as the elastic modulus of the indenter E_i, the elastic modulus of the material is:

$$E_{IT} = \frac{1 - \nu^2}{\dfrac{1}{E_r} + \dfrac{1 - \nu_i^2}{E_i}} . \qquad (5)$$

The compliance and therefore the elastic modulus are calculated from a fit of a function to the unloading curve using a power law, i.e. only one value for the elastic modulus can be extracted from the unloading curve. The load and partial unload technique with its multiple unloading curves is one possibility to get depth dependent information about the elastic modulus.

It is assumed that the elastic modulus of the reference material for the indenter shape correction is independent of the depth. The measurements without an indenter shape correction show an increase of the elastic modulus for small depths. To correct the measurements an iterative procedure recalculates the projected area and the measured compliance until the elastic modulus is constant [3]. While the contact compliance C_s is needed to calculate the elastic modulus, the measured compliance C_t also consists of the frame compliance C_f.

$$C_s = C_t - C_f \qquad (6)$$

Thus, the compliance needs to be corrected. With the resulting parameters from the projected area and the compliance, the elastic modulus as well as the Martens hardness can be corrected. Furthermore the Martens hardness of a standard indentation test can be corrected. Again, no table value for the elastic modulus is included in the correction. The absolute value is obtained from the data for large depths.

The elastic modulus can be determined with independent methods [6] and compared to the table value, which is an advantage of this method. On the other hand, as a result of the several load and unload cycles, the measurement is time consuming compared with a hardness correction. For long measurement times the temperature can affect the results. Therefore the number of data points is limited.

3.3 Comparison

An indenter shape correction of a load and partial unload cycle was performed using the elastic modulus and the Martens hardness. The corrected values for E_{IT} and H_M were calculated. Also the corrected Martens hardness of a standard indentation test was calculated with both methods. All measurements and corrections were done using a BK7 glass.

The resulting curves for the elastic modulus are shown in Fig. 4. Both measurements are almost constant and agree within the errors. Fig. 5 shows the results for the Martens hardness for the same measurements. Fig. 6 shows the corrected Martens hardness for a standard indentation test. The figures show a good agreement between the correction using the elastic modulus and the Martens hardness.
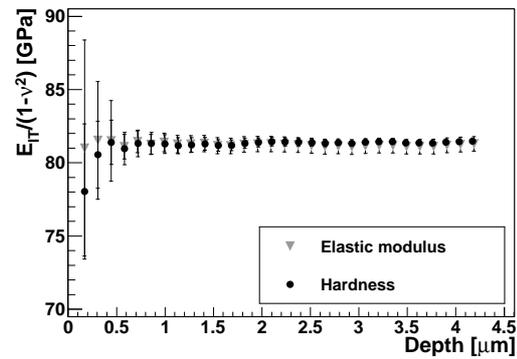

Fig.4: Corrected elastic modulus using a correction based on the elastic modulus (grey) and the Martens hardness (black).
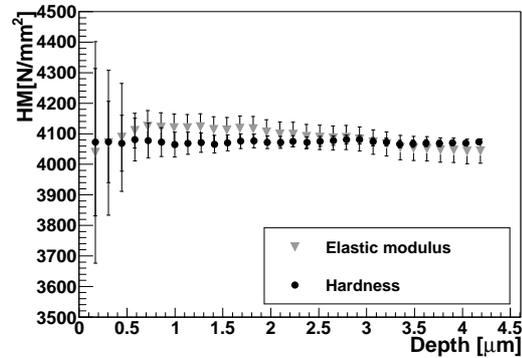

Fig.5: Corrected Martens hardness using a correction based on the elastic modulus (grey) and the Martens hardness (black).

4. Results

The parameters obtained from the corrections on BK7 glass are used to evaluate the elastic modulus and the Martens hardness for several materials. The values are calculated with both methods from chapter (3) and compared to each other. The elastic modulus was also compared to the table values. A selection of the results for glasses and metals is shown in Table 1.
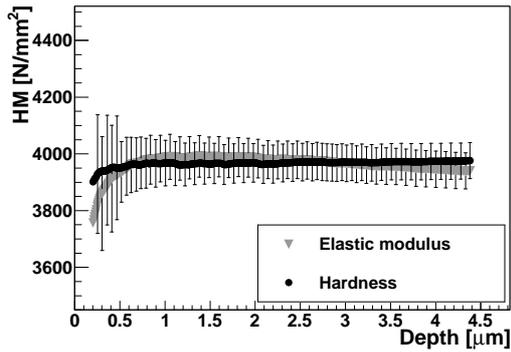
Fig.6: Corrected Martens hardness of a standard indentation test, using a correction based on the elastic modulus (grey) and the Martens hardness (black).

Table 1: Results for the elastic modulus and the Martens hardness for both corrections. The elastic modulus is also compared to the table values. The first eight materials are different glasses, the last three are titanium, copper and a hardness reference plate (HVP, steel). The uncertainties from the corrections are smaller than 2%.

Material	Table value [6],[7] E_{IT} [GPa]	Elastic modulus E_{IT} [GPa]	Elastic modulus H_M [N/mm²]	Martens Hardness E_{IT} [GPa]	Martens Hardness H_M [N/mm²]
BK7	82	76.9	4050	77.0	4030
Fused silica	72	64.7	3870	64.3	3800
LAK10	116	114.0	5370	114.4	5400
F5	58	60.0	3040	60.0	3030
BASF64	105	105.6	4850	106.0	4900
FK5	62	58.5	3450	58.4	3380
SF10	64	63.8	3120	64.0	3150
SF1	56	58.2	2870	57.9	2900
Ti	116	120.5		122.0	
Cu	130	130.8		132.4	
HVP	204	201.3		203.5	

The results in Table 1 show a good agreement for the corrections using the Martens Hardness and the elastic modulus. The uncertainties from the corrections are typically smaller than 2% and increase slightly for small indentation depths. The Martens hardness is only given if a constant value was measured. The data was compared down to a depth of 100 nm. There are differences between the table value of the elastic modulus and the measured ones which indicate that some effects like a variable epsilon and radial displacement corrections still need to be taken into account.

5. CONCLUSIONS

Indenter shape corrections using the projected area extracted from the load and partial unload technique and the depth information from the indentation process were performed. Neither of the methods need a table value as input, all information was extracted from the measured data. The corrected elastic modulus and the corrected Martens hardness were calculated for both methods. A comparison results in the conclusion that the values are equal valid at least down to an indentation depth of 100 nm. Both approaches can be used for the indenter shape correction. BK7 glass can be used as a reference material for both corrections, as there is no dependence on the depth for the elastic modulus and the Martens hardness.

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