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DEVELOPMENT OF CALIBRATION METHODS FOR THE NANOINDENTATION TEST

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Abstract: The nanoindentation method increasingly gains in importance for the determination of the mechanical properties of thin layers. Comparisons carried out in the field of the nanoindentation method reveal, however, significant deficits in calibration. The paper presents investigation results for the calibration of the essential quantities influencing the uncertainty of measurement. They chiefly comprise the geometrical deviations of the indenter, the depth and the force measuring system, the measuring deviation of zero point of the depth measurement and the compliance of the measuring device.
Keywords: nanoindentation, calibration, hardness

1 INTRODUCTION

The indentation method includes a synchronous measurement of test force F and indentation depth h at first at increasing and then at decrasing forces. From the two curves F = f(h) obtained for the increase in and decrease of force a great deal of information about the mechanical properties of the material to be investigated, e. g. universal hardness, plastic hardness, indentation modulus, creep, relaxation, elastic and plastic deformation work etc. can be extracted. This great information content of the indentation method in the so-called nanorange with indentation depths h < 0.2 µm is of particular interest, because this method often is the only possibility of investigating mechanical properties of thin layers (layer thickness < 2 µm). Such thin layers are increasingly required for functional and protective layers in microelectronics, micromechanics, optics and other applications. The draft standards ISO/CD 14577-1 to -3 [1] for the indentation method considerably contribute to the comparability of measurement results obtained by this method. The standard distinguishes between three ranges: macro-, micro- and nanorange. In a project of the EC program Standards, measurement and Testing "Determination of Hardness and Modulus of Thin Films and Coatings by Nanoindentation - INDICOAT" [2] at present specific measurement conditions for the determination of hardness and elasticity of thin layers are investigated. The corresponding standardisation will benefit from the prenormative investigations. But international comparisons completed in the last few years show that the reproducibility of the measurement results e. g. for the hardness and the indentation modulus between different laboratories is not yet satisfactory [3][4]. Hence it follows that the calibration methods should be improved and uniformly applied. The following investigation serves this purpose.

2 DEVELOPMENT OF CALIBRATION METHODS

An analysis of the quantities influencing the uncertainty of measurement of the nanoindentation method for the measurand hardness shows that the following influence quantities are of particular interest:[5]

- deviations of the actual from the desired geometry (for example, length of the line of junction and tip radius of Vickers indenters)
- measuring deviation of the depth measuring system
- compliance of the measuring device
- measuring deviation of the force measuring system
- measuring deviation of the zero point of depth measurement

2.1 Determination of the indenter geometry

For the determination of the indenter geometry we used a Scanning Force Microscope (SFM) Vertitekt-3 which has a measuring range x = 70 µm, y = 15 µm, z = 15 µm. The SFM contains three piezo translators for the three coordinate axes x, y, z. The piezo translators contain each a capacitive

measurement system which controls the movement of the scanners. Thus the SFM is used like a miniaturized three-coordinate measuring device. Because a calibration of indenters is satisfying only if the uncertainty of measurement between two points in the measuring volume amounts to few nanometers, this SFM was additionally equipped with three laserinterferometers (Michelson type) along the axes x, y, z, thus enabling for the correction of the nonlinearities of the capacitive measurement systems. Previously was already reported about the calibration of indenters with an SFM calibrated with laserinterferometers.[6] But this original arrangement of laserinterferometers had still an Abbe offset which must be carefully considered in view of the long indenter shaft. Although the occuring Abbe error as result of inclinations with the angle α around the x- and the y-axis was determined with a two-axis autocollimator which keeps an uncertainty of measurement U_α = 0,01" and corrected, due to the long shaft of the indenter even at using the high precision two-axis autocollimator one has to take into account an uncorrectable remaining Abbe error f_{Abbe} = (2 ... 3) nm. Therefore and in order to fulfil the uncertainty requirements at the calibration of indenters in cooperation with the Technical University Ilmenau an improved arrangement of the laserinterferometers free of Abbe errors was developed [7] (see Fig. 1).

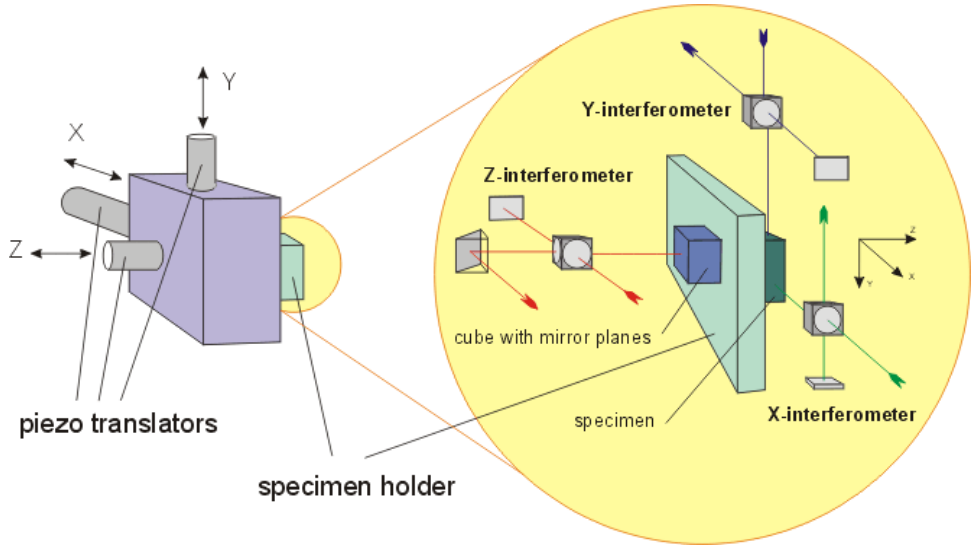

Figure 1. Abbe error free arrangement of the three laserinterferometers in the SFM

The characteristic of this new arrangement is that the measuring beams of the three laserinterferometers along the x-, y- and z-direction intersect in the measuring point on the sample. The uncertainty of measurement between two points with the distance L in the measuring range is estimated as $U = 1.5\ nm + 10^{-4} L$.

The geometry of the indenter is measured by two different object scans each containing 256 x 256 or 512 x 512 scan points. At first an object scan in the range x = 10 µm and y = 10 mm is made in order to determine the overall indenter geometry. Then follows an object scan in the range x = 1.5 µm and y = 1.5 µm in order to investigate the geometry in the immediate surrounding of the indenter tip. Each scan is repeated at least 3 times.

From the two-dimensional data sets using all scan points the real area of the indenter is calculated by the summation of triangle areas from neighbouring scan points. The data table Ai;hi then is used to calculate suited regression curves A = f(h), the so-called area function. Contrary to the procedure in the microrange, in the nanorange it is not senseful to calculate deviations from certain ideal geometrical quantities, like the plane angle or the tip radius, but all deviations are summarized in the area function. Fig. 2 shows the area functions of a Berkovich indenter in two ranges of the contact depth $h_c < 100$ nm and $100\ nm \le h_c \le 1000$ nm together with the theoretical curve for the contact area $A_c = 24.5\ h_c^2$.

As expected, for $h_c < 650$ nm the area function is larger than the theoretical curve mainly due to the effect of tip rounding. The relative uncertainty of the indenter area is estimated $\Delta A/A$ = 5 % until 15 % depending on the contact depth.

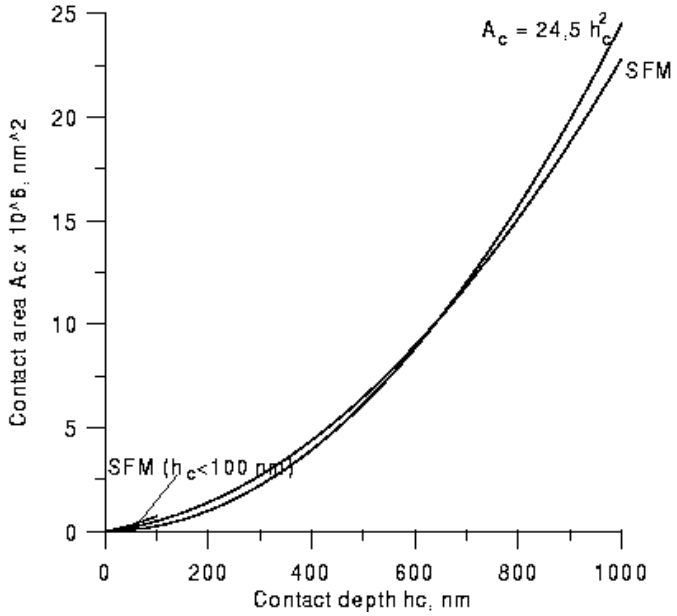

Figure 2. Area functions of a Berkovich indenter together with the theoretical curve of the contact area

Fig. 3 represents several radius functions r = f(h) of a ball-shaped diamond indenter in dependence on the indentation depth from repeated geometry measurements. The nominal radius is r = 10 nm.

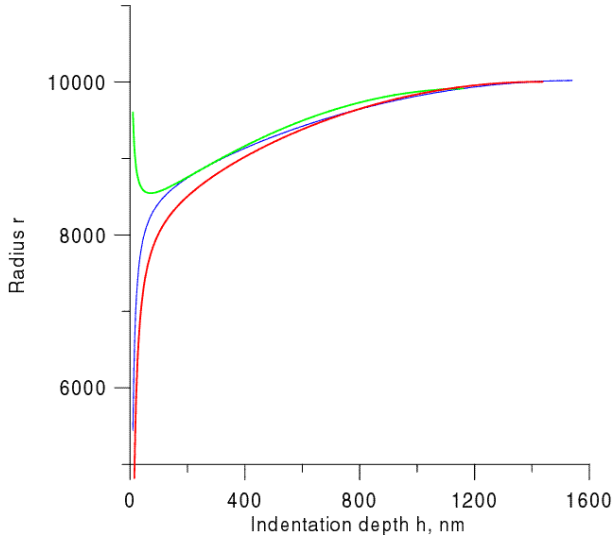

Figure 3. Radius function of a ball-shaped diamond indenter in dependence on the indentation depth

From the diagram it is visible that the radius r = 9 µm is reached already after an indentation depth of ≈ 400 nm and that the standard deviation of radius repeatability at repeated geometry measurements of the indenter with the SFM amounts only to $s_r \leq 40$ nm.

2.2 Measuring deviation of the depth measuring system

The measuring deviations of the depth measuring system can be determined by comparison with a laserinterferometer whose laser wavelength λ is in the visible range. The reachable calibration uncertainty amounts to U = (0.5 ... 1.0) nm. Fig. 4 shows the calibration setup with a multi-pass Jamin laserinterferometer developed in the NPL.[8]

This laserinterferometer has a resolution of 0.1 nm and an uncertainty U = 0.6 nm. In order to keep still smaller uncertainties which are desirable especially at nanoindentation measurements with indentation depths of only few nanometers, in the future one has to develop calibration methods which are founded on radiations with shorter wavelengths.

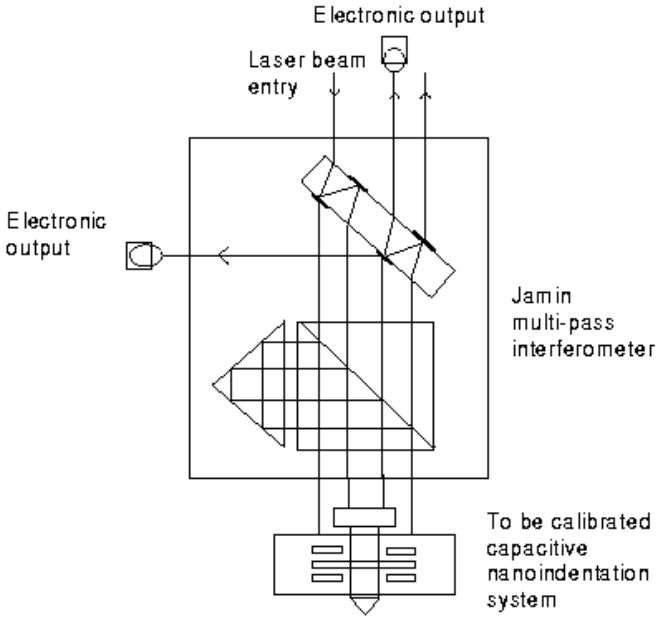

Figure 4. Calibration setup for the depth measuring system with a Jamin laserinterferometer

2.3 Compliance of the measuring device

For the determination of the compliance of the measuring device the method of Oliver and Pharr [9] has proven its worth. It is based on the fact to separate the compliance of the sample (which actually has to be measured) from the compliance of the measuring device. Fig. 5 shows the results of determination of the compliance of the universal hardness measuring device Fischerscope H100 with the three reference materials fused silica, optical glass BK 7 and tungsten single crystal.

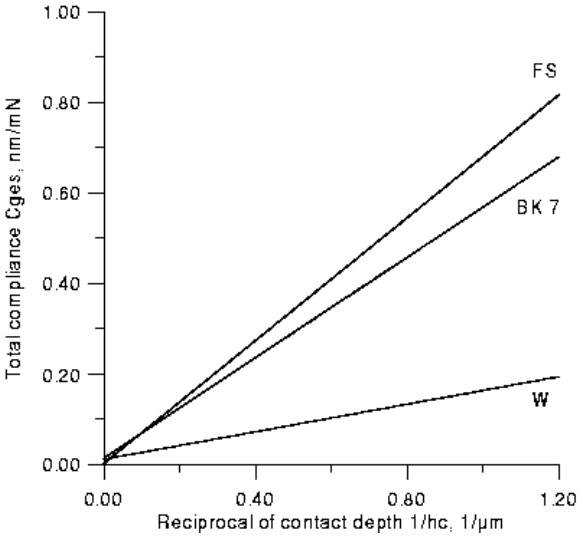

Figure 5. Results of the determination of the compliance of a universal hardness measuring device with the three reference materials Fused Silica (FS), optical glass BK7 and tungsten single crystal (W)

The compliance of the measuring device is the intersect of the compliance curve with the axis of the total compliance C_{total}. The method is the safer the better the total compliance $C_{total} = 1/S$ (S - stiffness) $= \Delta h/\Delta F$ (F - test force) can be determined from the tangent slope of the force decreasing curve during the indentation test. Under this point of view a reference material with an as small as possible slope in the diagram $C_{total} = f(1/h_c)$ is best suited; this means a reference material with an as high as possible Young's modulus - in our case for the three reference materials depicted in Fig.5 it applies to single crystal tungsten.

2.4 Measuring deviation of the force measuring system

For the calibration of the force measuring system, high resolution balances are used.[10] Due to the small path length the use of weighing cells with path compensation is recommended. The use of bending beams is also possible. If an error limit value $\Delta F/F = 1\%$ is assumed a test force $F \geq 1$ µN until 10 µN can be calibrated with these methods. For the test force range $F < 1$ µN calibration methods must be developed in future.

2.5 Measuring deviation of the zero point of the depth measurement

The measuring deviation of the zero point of the depth measurement can be obtained from the standard deviation of the regression curve $F = f(h)$ which is calculated from a certain number of measuring value pairs (F_i; h_i) after the indenter has hit the sample surface. Table 1 represents typical values of the measuring deviation of the zero point for samples of fused silica, optical glass BK 7 and tungsten single crystal, which were ascertained with the universal hardness measuring device Fischerscope H100.

Table 1. Measuring deviations of the zero point of depth measurement ascertained with the universal hardness measuring device Fischerscope H100

Sample	Zero point uncertainty U_{zero}, nm	Standard deviation of zero point uncertainty s_{Uzero}, nm	Surface roughness R_a, nm
Optical glass BK 7	1.36	0.39	2
Fused silica	1.65	0.48	1
Tungsten single crystal	1.79	1.22	4

As analyzed in [11], the measuring deviation of the zero point depends on the surface roughness R_a. From this at the same time follows that sophisticated nanoindentation measurements are meaningful only on samples with the small surface roughness $R_a \leq 2$ nm.

3 SUMMARY

The calibration methods presented offer realistic prospects for the necessary reduction of the uncertainty of nanoindentation measurements. Recently, on various international levels (SMT program of EC, VAMAS, CIRP, etc.) prenormative investigations into the nanoindentation method especially aimed at the determination of mechanical properties of thin layers could increasingly be observed; they all include calibration methods. What is important for the present and the future standardization of the nanoindentation method is that sufficiently verified calibration methods should be uniformly applied. Regardless of the progress achieved in the calibration methods and the propects of standardization, our attention should be focused on the fact that new requirements for this method and the considerable increase in the resolution of nanoindentation measuring devices recently developed call for calibration methods in the subnanometer range for the indentation depth and in the submicronewton range for the test force.

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