UNCERTAINTIES OF HARDNESS MEASUREMENTS
Anna CAMPBELLOVÁ, Miroslav VALTR and Petr KLAPETEK
Czech Metrology Institute, Okružní 31, 638 00 Brno, Czech Republic

Abstract
Nanoinindentation instruments are commonly used to study hardness and related mechanical properties at the nanoscale. However, traceability and uncertainty aspects of measurement process remain often left aside. For most of the cases, no uncertainty calculation is performed by instruments nor users, or only the ideal accuracy and resolution values given by manufacturer are used for estimation of the measurement uncertainty. As the nanoindentation instrument is a very complex device, usually at the state of art of present instrumentation, it can be expected that there are many effects affecting the measurement uncertainty. Proper instrument calibration and proper uncertainty propagation within the calculations is therefore important for both metrologists and normal users.

1. INTRODUCTION
The key task of metrology is to provide measurement traceability, to obtain right results, and to determine proper uncertainties. At the nanoscale, this can be quite complicated, as both the instrumentation and measurands are very complex. Here we present an approach for traceability and uncertainties used for nanoscale hardness measurements using a commercial hardness tester (Ultra Nano Hardness Tester from CSM Instruments).
In this contribution an AFM cantilever is used to transfer force traceability for measurement of small forces in nanoindentation experiments. A cantilever is not primarily concerned as a force sensor or transfer standard, however we can benefit from its small size and wide range of force constants available on market. The cantilever is first calibrated on a mass comparator, which is traceable to the national mass standard, and is then used to calibrate the force sensor of a CSM UNHT instrument. The uncertainty of the loading curve measurements is analysed and the resulting uncertainties for quantities, that are computed from loading curves such as hardness or elastic modulus, are studied. The standard procedure for obtaining Young's modulus and the indentation hardness is the Oliver-Pharr method [1], which involves a significant amount of data processing, such as fitting procedures etc. Due to its complexity the standard Guide to Uncertainty Measurements (GUM) [4] is difficult to use and not reliable. The Monte Carlo method for the evaluation of uncertainties offers a suitable alternative.
It is shown that the uncertainty of nanoindentation results cannot be based only on errors of least-squares fitting or other data processing procedures but that it is inherently connected to the nanoindentor itself and that the nanoindentor calibration uncertainty plays a key role in the whole measurement uncertainty budget.

2. METHODS
The depth sensor of the hardness tester was calibrated by comparing the data it returns on a set of small indents with the depths measured by an atomic force microscope (Veeco). A matrix of 16 indents was made for different values of the indentation force ranging from 0.1 mN to 10 mN. The residual depth obtained by the Oliver-Pharr method directly from the UNHT was used.
The force sensor was calibrated using an AFM cantilever as a transfer standard. The AFM cantilever stiffness was determined using a mass comparator, which is traceable to the national standard, and a piezoelement, which was calibrated by an interferometer. The calibration of the force sensor itself is based on the analytic description of an ideal, rectangular cantilever under an applied force. This can be determined analytically and is described in standard textbooks, e.g. [2]. The deflection of a cantilever of length L and stiffness k under a force F applied at a distance from the fixed point y is

\[ z = \frac{F}{k} \left( \frac{y}{L} \right)^3. \]

This relation can be used for a least squares fit to find the stiffness or to calculate the theoretical force from a known deflection. The position of the fixed point is hard to determine and can contribute significantly to the total uncertainty. As shown in [3] it is better to introduce the (unknown) position of the fixed point \( y_0 \) and fit \( y \) as a linear function of the inverse cubic root of the loading curve slope \( c = \frac{dF}{dz} \). This determines both the stiffness and the shift. Since the length, the position and the shift are known for each depth value a theoretical force value can be calculated. These values together with the measured values provide a calibration curve.

The evaluation of uncertainties arising in the calibration of both the depth sensor and the force sensor cannot be treated by the standard methods described in the Guide to Uncertainty Measurements (GUM) [4]. The models are non-linear and involve quantities with non-Gaussian distributions. This often leads to incorrect best value estimates, uncertainty estimates or coverage intervals. On the other hand, there are almost no restrictions on use of the Monte Carlo method [5]. The basic concept of Monte Carlo method is to model the input data by a suitable probability distribution function (PDF), process the generated data, statistically evaluate the outcome. Care is needed to choose the correct PDF, in our work we used a normal distribution for experimentally measured data with statistical uncertainties of type A and uniform distributions for quantities whose uncertainties had to be estimated (type B).

3. RESULTS

The data both from the UNHT and the AFM are shown in fig. 1. The ratio of the depth values was fitted as a linear function of the applied force. Relaxation effects, method dependent effects and surface roughness are not taken into account.
Three types of cantilevers were used for the calibrations LRF, PPP-SEIHR from Nanosensors and All-in-One from Budget Sensors. A set of force distance curves was measured at different positions along the cantilever, at each position several force distance curves were taken. The load was kept low so that the loading would be elastic, usually between 50 and 70 µN. Each force distance curve was fitted using the ordinary least squares method with a straight line. The slopes were used for a total least squares fit of the distance $y$ and the inverse cubic root of the slope $c^{-1/3}$ in order to find the value of the shift $y_0$. The value of the depth sensor was corrected according to the depth sensor calibration and then inserted in the equation above. Calibration curves were measured for several cantilevers. They could be fit by e.g. a linear relation. However, we preferred to use all calibration curves in our Monte Carlo evaluations.

As for the uncertainty of the force value, the largest contributions come from the uncertainty in the stiffness of the cantilever, the position of the indent along the cantilever and the uncertainty of the depth. The dependence of the relative uncertainty is shown illustratively in fig. 2 for two positions A,B along a chosen cantilever.
The Oliver-Pharr method involves the contact surface. This has been calibrated within the nanoindenter device itself for each indenter. The assumed shape is

\[ A_c = a_2 h^2 + a_1 h + a_{1/2} h^{1/2} + a_{1/4} h^{1/4} + a_{1/8} h^{1/8} + a_{1/16} h^{1/16}. \]

No uncertainty analysis of the coefficients was performed so far and will be subject to further work.

As an example, the hardness and Young’s modulus were measured on copper (Cu) by the UNHT. The Monte Carlo method was used for the whole data processing: including the correction with respect to depth and force calibration and the Oliver-Pharr analysis. The resulting indentation hardness was found to be \((1956 \pm 275)\) MPa. The resulting elastic modulus was found \((146 \pm 25)\) GPa. The relative uncertainties are 14 %, resp. 17 %. The dominant source of this is the uncertainty in the cantilever stiffness (approx. 11 %), which affects the force calibration. The larger value for the elastic modulus uncertainty reflects the fact that its calculation is more involved than the calculation of the nanoindentation hardness. It also includes a fitting procedure and the slope of this linear fit contributes also significantly to the overall uncertainty budget.

5. CONCLUSIONS

In this contribution the calibration procedure of a nanoindenter was discussed. This procedure can be also used to calibrate AFM cantilevers in a fast, simple and traceable way.

it was shown how indentation hardness and elastic modulus can be measured traceably and an estimate of the resulting uncertainty is given. A Monte Carlo method of uncertainty evaluation is a fast and simple method to determine uncertainties which cannot be determined by standard means.
REFERENCES


