Good Practice Guide "Elastic property measurement of nanobeams by cantilever force measurement inside a Scanning Electron Microscope"

EMRP Project NEW05 MechProNO Traceable measurement of mechanical properties of nano-objects

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1. Introduction

The measurement of the elastic modulus of small micro-sized beams using the bending method is described in this good practice guide. The bending is realized by a cantilever-based force sensor in the vacuum chamber of a scanning electron microscope.

The method is based on in-situ bending tests on beam-like objects with a micromanipulator equipped with a force measurement tool [1]. The objects were cut out of commercially available free-standing silicon nitride membranes by the Focused Ion Beam (FIB) technique.

Only few parameters have to be checked for the determination of the Young´s Modulus:

- the correct force
- the displacement and
- the geometry parameters of the beams

Especially for brittle materials as silicon nitride the bending test is a suitable method in comparison to tensile testing, where no or less information about the deformation capability are available out of the measuring set-up. In a bending test this information can be extracted easily by the detection of the displacement.

A detailed uncertainty budget for the new method was set-up. FEM calculations support the required information. Finally the report clearly shows potentials and limitations of the used technique on a practically examined testing system. Moreover it gives recommendations how to prepare such a test on a thin solid film system, that means which parameters are important to get reliable results.

2. Experimental details

The beams investigated are amorphous silicon nitride beams with a thickness of 50 nm, 100 nm, 200 nm and 500 nm, a width of 2 µm and different lengths.

The beams were bent with a Force-Measurement-System (FMS) from Kleindiek Nanotechnik, Germany [2]. This system is an extension tool for a micromanipulator from the mentioned supplier. The tool is equipped with a 400 µm long silicon cantilever, coated with a piezo-resistive material. In contact with another material it gives a voltage signal. The FMS is mounted in the vacuum chamber of the SEM and every step can be observed.

Before starting to measure on the silicon nitride beams, the system has to be calibrated. Therefore a certified calibration spring from PTB with known spring constant (k_1 = 1,94 N/m) is displaced exactly 1 µm [3]. Together with the detected voltage a calibration value is calculated. All measured voltages can now be multiplied by this calibration value and give the corresponding force value. To check whether this calibration value is plausible a second calibration spring with known spring constant (k_2 = 8,98 N/m) is also displaced 1 µm.

A second parameter has to be checked before starting to measure: the stiffness of the used cantilever. During the test bending of the cantilever should not take place. Therefore a maximum loading force for the FMS was determined. Experimentally a calibrated microelectronic mechanical system (MEMS) device from PTB with a moveable main shaft was displaced about 6 µm. The stiffness of the MEMS is stable until a maximum displacement of 8 µm. When comparing both the theoretical and the measured force-displacement curves it can be shown that the slope (stiffness) is equal until a maximum force of about 7 µN. With higher forces the slopes differ more and more, which indicates that the cantilever is bent.

The silicon nitride beams can be tested now. Unfortunately the system offers no automatic solution to apply constant forces or loading rates. Thus the beams were bent manually via driving knobs moving the micromanipulator in the three axes. However, preliminary tests showed that the manual bending and associated different bending speeds did not influence the resulting value significantly. The detected force has to be correlated to the displacement. The displacement was measured with the image correlation software VEDDAC from Chemnitzer Werkstoffmechanik GmbH [4] out of a series of images taken every second during the bending test by the SEM. Fig. 1 shows a measurement curve.

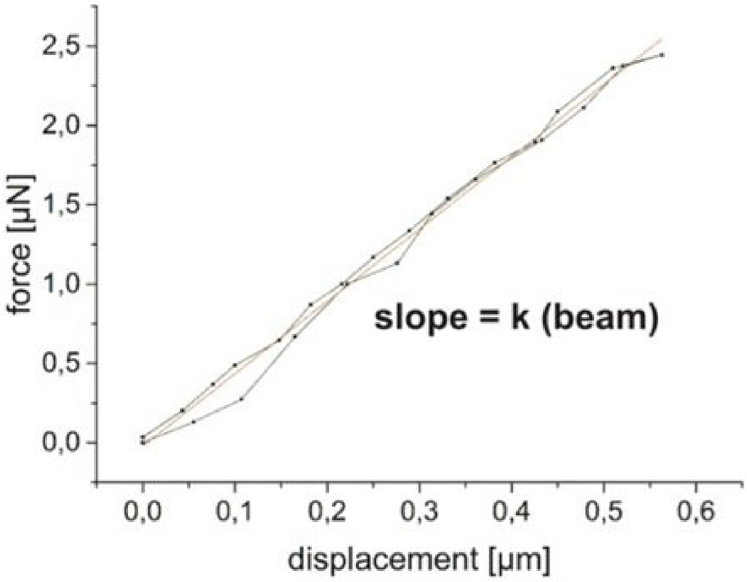

Fig. 1 Force-deflection curve of a silicon nitride beam measured by the cantilever force sensor inside the SEM

3. Evaluation

The real thicknesses of the silicon nitride beams were determined by TEM. Seven tests were done per thickness with seven beams of similar dimensions. The detected force signal of the FMS software and the manually measured displacement were correlated for each bending test. The resulting curve was fitted linearly and a stiffness value *k* (the slope of the curve) for the tested object was obtained. With the exact measured geometry – in this case a rectangular one - of the beam a Young´s Modulus can be calculated according to the Euler-Bernoulli equation:

$$E = \frac{4 \cdot L^3}{W \cdot T^3} k \tag{1}$$

with the effective length *L*, width *W*, thickness *T* and beam stiffness *k*. Fig. 2 shows the calculated Young´s Moduli in overview. For the nominally 100 nm, 200 nm and 500nm thick beams the values are more or less stable. The obtained Young´s Moduli are listed in Table 1. In the literature lots of different values can be found for thin film amorphous silicon nitride: 210 GPa – 350 GPa for 100 nm to 500 nm thin beams measured by resonance spectra [29], 100 GPa – 300 GPa for 200 nm thin films deposited by various techniques [5], measured with IIT, and about 250 GPa for 160 nm thick film using bulge testing [6]. The calculated values in this study fit into the range given by the literature.

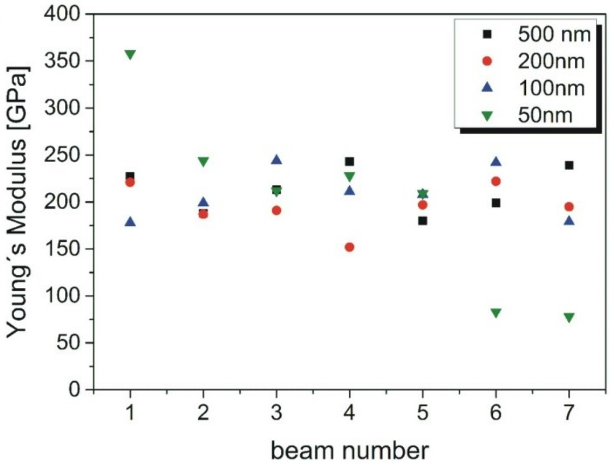

Fig. 2: Young´s Moduli for nominally 50 nm, 100 nm, 200 nm and 500 nm thick silicon nitride beams measured by the cantilever force sensor inside the SEM.

Table 1 TEM measured thicknesses and mean Young´s Moduli obtained by the FMS method

Nominal thickness [nm]	TEM measured thickness [nm]	Young´s Modulus mean value [GPa]
50	50	(202)
100	97	209
200	182	195
500	515	213

4. Uncertainty budget

The uncertainty for the force value can be set to about 10 %. The measured displacement has to be considered with an error of about 1%. In most of the cases the tip and the surface of the tested beam were not exactly at 90°. The positioning of the tip was roughly done by hand before the whole equipment is pumped in the SEM. The tip can´t be rotated. Here the uncertainty of 1 % can be estimated additionally for the force value. All the mentioned errors influence mainly the stiffness *k*, which is directly proportional to the Young´s Modulus. Further uncertainties are the geometry parameters of the beams, which have a much larger influence on the resulting value. First the uncertainty of the width and the length of the tested beam amount to 3 % for the error in SEM length scale measurement. The possibility that the tip slides some nanometers on the smooth beam surface during large displacements - and therefore changes the effective length of the beam - was considered in the calculation of the Young´s Modulus in part 3. The thickness of the silicon nitride film was determined by TEM as exact as possible, because this parameter is multiplied by the negative third power in the Euler-Bernoulli equation. Nevertheless an uncertainty of 3 % has to be taken into account for the TEM thickness measurement.

An error propagation calculation can be done for all the termed error sources and Table 2 shows the obtained values.

$$\Delta E = \sqrt{\left(\frac{4 \cdot L^3}{W \cdot T^3} \cdot \Delta k\right)^2 + \left(\frac{12 \cdot k \cdot L^2}{W \cdot T^3} \cdot \Delta L\right)^2 + \left(\frac{4 \cdot k \cdot L^3}{W^2 \cdot T^3} \cdot \Delta W\right)^2 + \left(\frac{12 \cdot k \cdot L^3}{W \cdot T^4} \cdot \Delta T\right)^2} \tag{4}$$

Table 2 Calculated uncertainty values for the Young´s Modulus

Thickness [nm] nominal	Young´s Modulus E [GPa] mean value	Standard deviation for the Young´s Modulus [GPa]	Uncertainty ±ΔE [GPa]
100	209	24	21
200	195	22	15
500	213	25	20

There are some more errors which can influence the mechanical behavior and the result. The beams were machined with the help of Gallium-Ions. These ions are implanted in the material and thus may change the composition and mechanical properties within a superficial layer of the tested beam. In a former study we found out that this implantation depth can be set to maximum of 10 nm for 30 kV ion beam at the edges of the tested object, which is less than 1 % damaged volume of the whole object [7]. Thus the influence of the implanted ions can be neglected here as well as the presence of intrinsic stresses in the film which should be completely released by cutting free the membrane. One important evidence on intrinsic stress is the buckling of the released structure [8], which was observed only in case of the 50 nm thick film.

Usually the beam is bended at the end, but concerning the instrumentation setup it is difficult to strike the exact end position. To estimate this uncertainty, corresponding force-displacement-curves were simulated by FEM.

Finally to complete the list of uncertainties both the elastic-plastic deformation of the FMS tip and the thermal drift has to be taken into account. These errors should be very small, because the tested objects are quite soft compared to the tip and the FIB/SEM room was under climate control.

Literature

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