Paper

Lateral Force Calibration Method Used for Calibration of Atomic Force Microscope

Magdalena Ekwińska and Zygmunt Rymuza

Abstract—Modern heterogeneous micro- and nanostructures usually integrate modules fabricated using various materials and technologies. Moreover, it has to be emphasized that the macro and micro nanoscale material parameters are not the same. For this reason it has become crucial to identify the nanomechanical properties of the materials commonly used in micro- and nanostructure technology. One of such tests is a nanowear test performed using the atomic force microscope (AFM). However, to obtain quantitative measurement results a precision calibration step is necessary. In this paper a novel approach to calibration of lateral force acting on the tip of an AFM cantilever is discussed. Presented method is based on application of known lateral force directly on the tip using a special test structure. Such an approach allows for measurements of nanowear parameters (force, displacement) with the uncertainty better than ±3%. The calibration structure designed specifically for this calibration method is also presented.

Keywords—AFM, calibration structure, cantilever, MEMS.

1. Introduction

The current trend for miniaturization of mechanical components brought not only the shift of manufacturing technology from conventional to silicon, but most of all made it necessary to describe the behavior of microelectromechanical system (MEMS) in the scale in which they operate. In micro and nanoscale the forces applied as well as the areas of contact are much smaller than in macroscale. In addition the microscale influence of such forces as adhesion or capillary forces is much more significant than the macroscale one. Under these circumstances the well known macroscale material parameters are not applicable in microscale and the easiest way to describe the microscale material properties is to perform an experiment in the same scale. Such investigations may be done with the use of atomic force microscope (AFM), which is a powerful device for estimation of, e.g., micro- and nanoscale wear resistance of materials. AFM is composed of a probe scanner, probe displacement detector, electronics connected with a computer and a system of isolation from vibrations. The scanner, which is the heart of the system, enables movement between the sample and the probe to be achieved. The scanner is usually a ceramic piezoelectric device that may move a sample or a probe. The probe is a cantilever, that is a lever with a sharp (cone or pyramidal) tip at one end. During wear test the cantilever tip is in contact with the sample surface. Depending on the applied scanning direction the lever is bending or twisting. The detecting system of cantilever displacement is usually a laser beam and a four-segment photodiode. The laser beam is focused on one end of the cantilever, where it is deflected and falls on the detector. The change of the laser beam position on the detector is later converted to the force (lateral or normal) signal in arbitrary units [a.u.] (Fig. 1). Therefore, a calibration is needed.

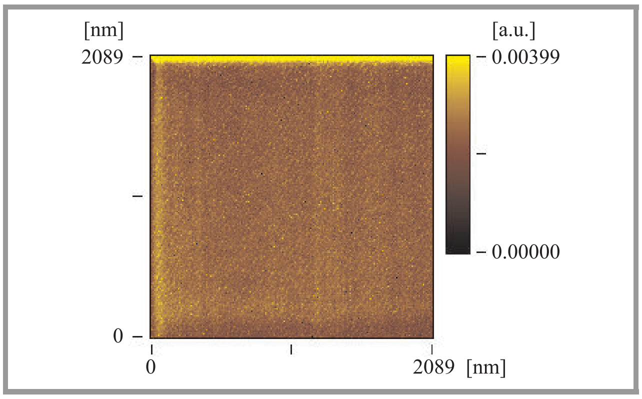


Fig. 1. Lateral force signal obtained during nanowear test.

2. Calibration Method

In the literature there are several calibration techniques for lateral forces. The most known are analytical and geometrical [1] methods, two-step calibrating method for a scan parallel to the long axis of the cantilever [2], improved wedge calibration [3], etc.

The first method is a simple analytical one in which the cantilever stiffness is established from the macroscale equation. This method involves precise measurements of cantilever dimensions. These dimensions may be either taken from a catalog or measured experimentally. The maximum uncertainty may reach 100% of the measured value.

The second method is a geometric one [1] in which the cantilever torsion angle is established. Conversion of the torsion angle into lateral force is obtained by the analysis of the optical geometry of the laser-beam path. In this calculation precise estimation of the cantilever tip height is also required. The uncertainty of the discussed method is around 30%.

The two-step method of calibration described in [2], [4] for a scan parallel to the long axis of the cantilever unfortunately cannot be considered as a quantitative one.

The wedge calibration method [3] is a direct calibration of the lateral force by applying a known turning moment or by the definition of the torsion moment of the cantilever on a substrate with well-defined slopes. An improved wedge calibration method utilizes a commercial calibration grating TGF11 (manufactured by NT – MDT Inc. in Moscow, Russia) takes into account the effect of the tip radius of curvature and eliminates the need for multiple measurements with different loads. The inconveniences of this method are quite complicated calculations and the need for stiffness calibration in normal direction. Moreover, during calibration additional parameters such as friction coefficient and adhesion force between cantilever and sample surface should be used. The uncertainty of this method is between 3 and 11% (95% level of confidence) depending on the applied normal force.

There is also a method in which the resonance frequency of cantilever with additional mass is used. The main disadvantage in this case is the application of the additional mass, which may lead to cantilever tip damage or change its parameters. The error in this method is 15%.

The present work deals with a new method of lateral force calibration. In this method the whole AFM is treated as a black box. Extortion is a well known value of lateral force applied at the end of the cantilever tip. This force causes cantilever torsion, then a change of the position of the laser beam spot on the detector, and a change of the signal from the detector, and finally a change of the output parameter: namely, the lateral force signal in arbitrary units. Comparison of the applied force in nN and the force in a.u. received from the device enables estimation of calibration parameter K in nN/a.u.

3. Calibration Sample

In order to calibrate the lateral force, a system with elastic element was elaborated [5], [6] (Fig. 2). There are three main parts of the elastic sample: surface on which the cantilever tip is located (*1*) with specially prepared roughness, flat spring which is bended in S shape (*2*), surface to which AFM table is mounted (*3*). During calibration of the lateral force this calibration device is placed on the AFM table and the cantilever tip is approached to the surface (*1*). After obtaining contact a known value of displacement is applied by the AFM scanner. The surface to which the AFM table is mounted (*3*) moves according to the displacement applied by the piezoscanner. If surface (*1*) of the calibration sample were free then it would move. During calibration the surface cannot move because it is held in one position by the cantilever tip. That causes bending of the element (*2*) of the calibration sample. It is worth underlining that the displacement of the bending element is smaller than the one applied by the AFM table. The small displacement of the element (*2*) multiplied by the stiffness of the calibrating device (which has to be obtained earlier) is the real value of the applied force acting on the cantilever tip trapped between microasperities of the surface (*1*). This force causes twisting of the cantilever and the change of the lateral force signal in arbitrary units. This procedure is repeated from 100 to 500 times. As a result of the calibration of the AFM a cloud of points representing the dependence of the lateral force (in a.u.) between cantilever and elastic element from bending (caused by the scanner movements), is obtained (Fig. 3).

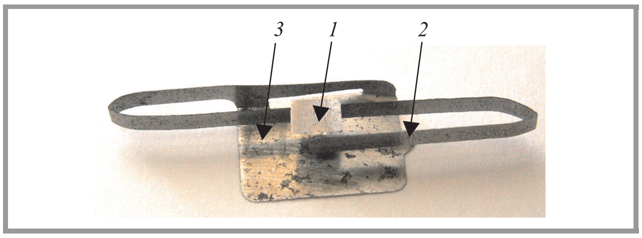


Fig. 2. Lateral force calibration sample. Explanations: *1* – element, which is elastically deformed during calibration, *2* – area where AFM's tip stands during calibration, *3* – device holder.

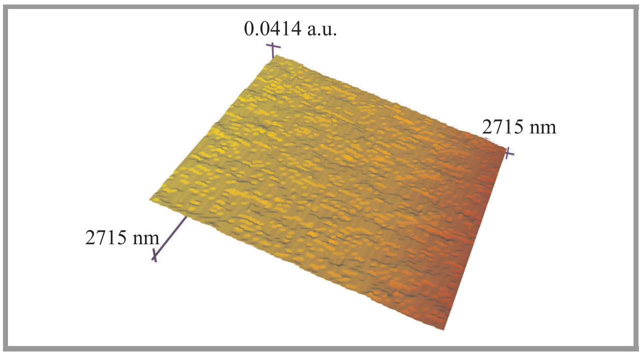
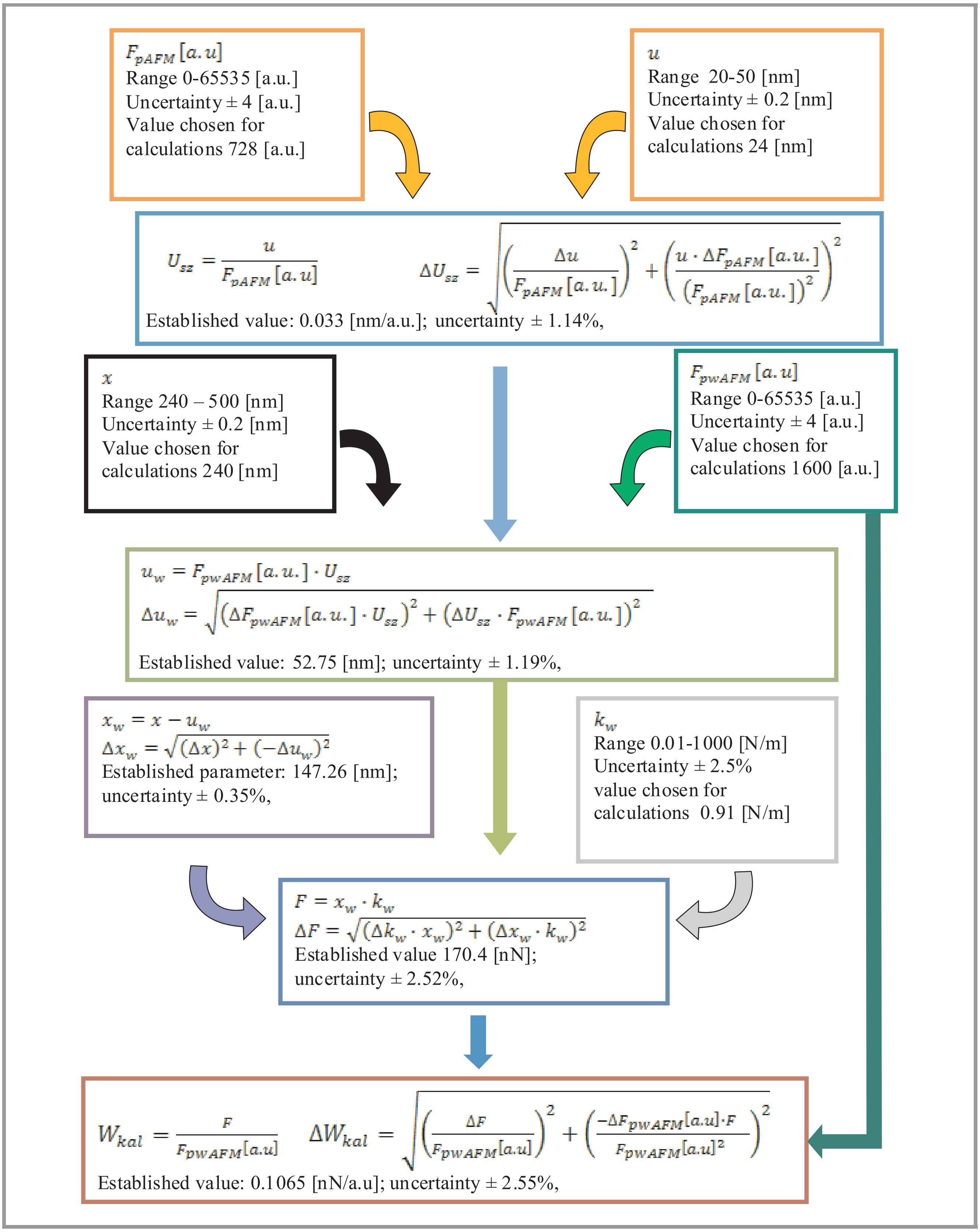


Fig. 3. AFM image presenting the dependence of lateral force between cantilever and elastic element on its bending (caused by scanner movements).

Due to the monolithic structure of the calibration sample and the use of the AFM scanner as an element responsible for the movements of the calibration sample the calibration procedure is simple and may be done directly on a cantilever mounted on AFM just before or just after wear test measurements. The uncertainty of the presented calibration method does not exceed ±3%. Figure 4 presents schematic estimation of the uncertainty of the calibration method. Apart from uncertainty of the AFM measurements and that of the calibration of lateral force, the range of force has big influence on the measurement results.

The results of uncertainty estimation for different force ranges is presented in Fig. 5. It can be seen that the uncertainty do not exceed 40% in the worst case. The worst case is when the calibrating procedure is made for very small force ranges, e.g., several nanometers and several dozen, the stiffness of the cantilever differs from the stiffness of the calibration sample a hundred times and the forces



$F_{pAFM}\,[a.u]$
Range 0-65535 [a.u.]
Uncertainty ± 4 [a.u.]
Value chosen for calculations 728 [a.u.]

u
Range 20-50 [nm]
Uncertainty ± 0.2 [nm]
Value chosen for calculations 24 [nm]

$$U_{sz} = \frac{u}{F_{pAFM}\,[a.u]} \qquad \Delta U_{sz} = \sqrt{\left(\frac{\Delta u}{F_{pAFM}\,[a.u.]}\right)^2 + \left(\frac{u \cdot \Delta F_{pAFM}\,[a.u.]}{\left(F_{pAFM}\,[a.u.]\right)^2}\right)^2}$$

Established value: 0.033 [nm/a.u.]; uncertainty ± 1.14%,

x
Range 240 – 500 [nm]
Uncertainty ± 0.2 [nm]
Value chosen for calculations 240 [nm]

$F_{pwAFM}\,[a.u]$
Range 0-65535 [a.u.]
Uncertainty ± 4 [a.u.]
Value chosen for calculations 1600 [a.u.]

$$u_w = F_{pwAFM}\,[a.u.] \cdot U_{sz}$$

$$\Delta u_w = \sqrt{\left(\Delta F_{pwAFM}\,[a.u.] \cdot U_{sz}\right)^2 + \left(\Delta U_{sz} \cdot F_{pwAFM}\,[a.u.]\right)^2}$$

Established value: 52.75 [nm]; uncertainty ± 1.19%,

$$x_w = x - u_w$$

$$\Delta x_w = \sqrt{(\Delta x)^2 + (-\Delta u_w)^2}$$

Established parameter: 147.26 [nm]; uncertainty ± 0.35%,

k_w
Range 0.01-1000 [N/m]
Uncertainty ± 2.5%
value chosen for calculations 0.91 [N/m]

$$F = x_w \cdot k_w$$

$$\Delta F = \sqrt{(\Delta k_w \cdot x_w)^2 + (\Delta x_w \cdot k_w)^2}$$

Established value 170.4 [nN]; uncertainty ± 2.52%,

$$W_{kal} = \frac{F}{F_{pwAFM}[a.u]} \qquad \Delta W_{kal} = \sqrt{\left(\frac{\Delta F}{F_{pwAFM}[a.u]}\right)^2 + \left(\frac{-\Delta F_{pwAFM}[a.u] \cdot F}{F_{pwAFM}[a.u]^2}\right)^2}$$

Established value: 0.1065 [nN/a.u]; uncertainty ± 2.55%,

Fig. 4. Estimation of the uncertainty of the lateral force calibration method. Explanations: u – displacement of the cantilever tip during calibration procedure, F_{pAFM} – lateral force, U_{sz} – multiplier describing how big displacement of the cantilever tip causes a change of 1 a.u. of the lateral force during rigid/stiff sample test, F_{pwAFM} – change of the lateral force value, u_w – displacement of the cantilever tip during calibration on the calibration sample, x_w – real displacement, x – applied displacement, k_w – stiffness of the calibration sample, W_{kal} – calibration coefficient, F – lateral force in real units [nN].

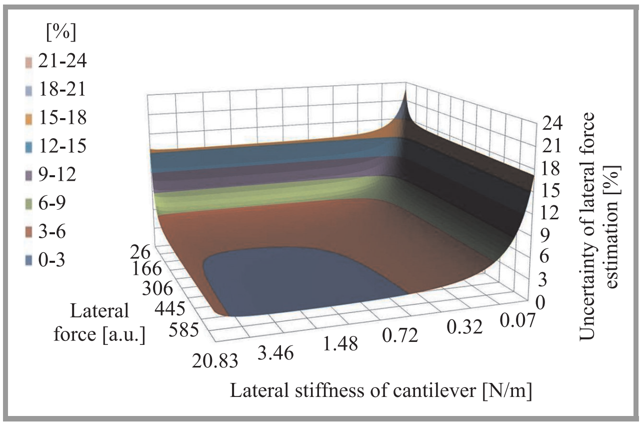


Fig. 5. Uncertainty of the real value of the lateral force estimation as a function of the measured lateral force and cantilever stiffness, with the calibration sample stiffness $k_w = 1$ N/m, uncertainty of the stiffness of the calibration sample ±0.025 N/m (2.5% of stiffness value).

during real measurements (measurement after calibration) are around several dozen [a.u.].

4. Summary

A new method of calibration of lateral force in AFM was presented. This method may be used for most of the cantilevers available on the market. In this new calibration approach a new additional calibration device is used. During the calibration procedure the cantilever is fixed in the microscope in the same way as during measurements. A dependence of the lateral force in arbitrary units on the applied force is obtained as a result of the two-step calibration. This relationship depends on lateral stiffness of the whole measuring system in the microscope, lateral stiffness of the cantilever and the stiffness of the fixation of the cantilever to the microscope. The calibration should carried out for every newly mounted cantilever.

The presented calibration method is novel, easy to use and applicable to a cantilever that is already mounted in the AFM system. The primary source of the calibration uncertainty as well as the uncertainty during later lateral force measurements is the uncertainty of the calibration of the calibration sample itself. The uncertainty of the stiffness measurement of the calibration sample is ±2 to 3%. The uncertainty of the lateral force measurement on the AFM is smaller than 0.01% of the measuring range.

Using the presented method the uncertainty of the calibration of lateral force in AFM does not exceed ±3%. To achieve this the following conditions should be fulfilled:

- uncertainty of the stiffness of lateral force calibration sample should not exceed ±2.5%;
- stiffness of the measuring cantilever should not depart far from the stiffness of the calibration sample (it should not be smaller than 0.7 k_w and simultaneously it should not be higher than 10 k_w, where $k_w =$ stiffness of the calibration sample);
- force range during and after calibration should not be lower than 0.5% of the AFM measuring range;
- displacement range applied during and after calibration should not be lower than 0.5% of measuring range of the AFM.

This method enables better precision of measurements to be obtained than with the use of the methods known from the literature. Besides it may be used for all types of cantilevers and does not depend on clamping of the cantilever or stiffness of the whole measuring unit.

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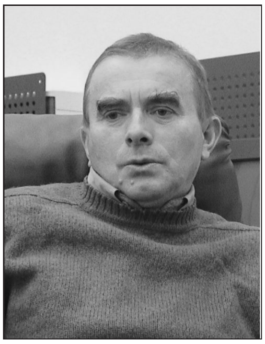

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