

Issues in validation of friction in the nanometric domain

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Abstract

Friction is one of the most challenging problems in micro- and nanosystems' technologies. While frictional phenomena on the macro- and meso-scales are well described and can generally be efficiently compensated via proper control typologies, in the nanometric domain friction is still a matter of persistent studies. Our ongoing work aims at providing a contribution to the study of friction by characterising the parameters influencing its value at the nanometric scale. The dependence of friction on material types, surface topography, loading conditions, velocity of motion, temperature and lubrication will thus be investigated via SPM measurements. Due to the large number of monitored influences, the number and type of measurements will be determined by using novel metamodeling techniques of arranging the measurement campaigns. Preliminary results aimed at a systematic engineering approach to the conduction of a finite set of methodical experiments characterising nanometric friction are thus presented in this paper.

Nanometric friction, experimental determination, scanning probe microscope, design of experiments, friction model

1. Introduction

Devices characterised by micro- and nanopositioning precision are often required in precision engineering as well as micro- and nanosystems' technologies. When devices based on sliding and rolling mechanisms are used, positioning precision is limited mostly by friction with its stochastic nonlinear characteristics. While the elasto-plastic nature of friction on the macro- and meso-scales is described relatively well [1] where frictional effects can be simulated via suitable models and efficiently compensated via proper control typologies [2], the available friction models do not take into account true nanometric motions with predominantly elastic behaviour due to adhesion, plastic effects due to single-asperity contact deformation, nor scaling phenomena related to friction [3]. In fact, the understanding of friction at the level of atomic interactions was enabled only in the last two decades by the affordable availability of scanning probe microscopy (SPM) methods [1].

Our ongoing work in this frame aims thus at providing a contribution to the study of friction by characterising the parameters influencing its value when considering nN range forces and nm/s velocities. The dependence of friction on these parameters is investigated via SPM measurements. Special attention is devoted to the proper calibration of the used probes. Due to the large number of monitored influences, the number and type of measurements is to be determined by using metamodeling techniques. Preliminary results aimed at a systematic engineering approach to the conduction of a finite set of methodical experiments characterising nanometric friction are thus presented.

2. Preliminary experimental measurements

The preliminary experimental assessment of nanometric friction is performed by using the Bruker Dimension Icon SPM. To determine the value of frictional forces, the Lateral Force Microscopy (LFM) contact measurement configuration is used, where the tip of the probe (Fig. 1) moves transversally with respect to the longitudinal axis of probe's triangular cantilever. A separate signal related to the normal and the lateral deflection of the cantilever is thus obtained. The used probes are Bruker's SNL-10 high-resolution type D probes, with a Si tip mounted on

a triangular Si₃N₄ cantilever. In order to quantify frictional forces, the stiffness of the cantilever has to be accurately calibrated.

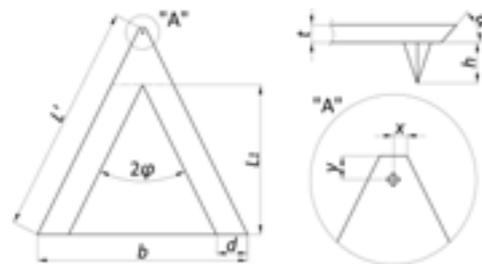


Figure 1. Geometrical parameters of the used Bruker SNL- 10 D probe.

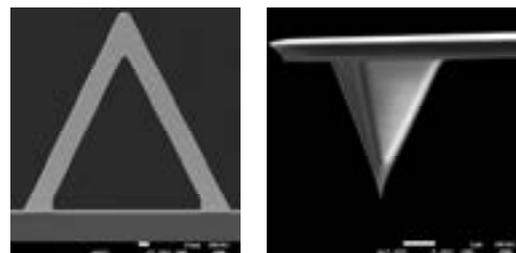


Figure 2. SEM micrographs of Bruker SNL- 10 D SPM probes.

In this work, a validation of the bending stiffness k_b of the cantilever with respect to its nominal value (0.06 N/m) is performed first via the thermal tune method (TTM). TTM involves measuring the power spectral density of cantilever's motion in the time domain in response to dynamic excitations [4]. The thus obtained measurement results (cf. table 2) have been used to assess the approximations induced by calculating the stiffness of the cantilever via the analytical method of parallel beam approximations (PBA) [5], as well as via finite element (FE) calculations performed in Ansys®. The calculations are based on an accurate measurement of the dimensions of the SPM probes performed on the Jeol JSM-7800F Scanning Electron Microscope (SEM), that allows magnifications up to 1,000,000 times and imaging resolutions down to 0.8 nm (Fig. 2). Ten probe samples are scanned allowing to obtain certain statistics on the dispersion of the values of their dimensions (table 1).

Table 1 Measured dimensions of SPM probes from SEM micrographs.

	Average	Std. dev.
$L'/\mu\text{m}$	214.17	0.77
$L_s/\mu\text{m}$	150.58	0.50
$d/\mu\text{m}$	22.90	0.51
$b/\mu\text{m}$	201.59	1.10
$\varphi/^\circ$	26.25	0.81
$\alpha/^\circ$	60.29	5.46
$t/\mu\text{m}$	0.55	0.03
$h/\mu\text{m}$	4.71	0.14
$x/\mu\text{m}$	5.08	0.17
$y/\mu\text{m}$	3.58	0.11

Table 2 Bending and torsional stiffness of the Bruker SNL-10 D probes.

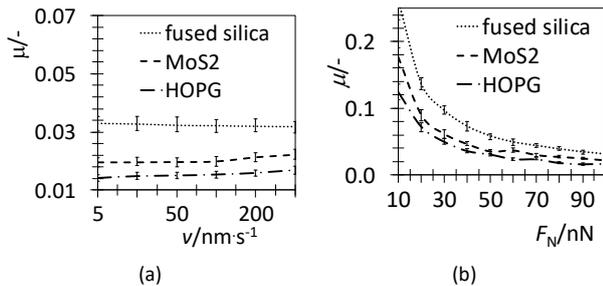
	TTM	PBA	FE
$k_b/\text{N}\cdot\text{m}^{-1}$	0.086	$0.056 \pm 12\%$	$0.098 \pm 8\%$
$k_t/\text{Nm}\cdot\text{rad}^{-1}$		$79.37 \pm 16\%$	$92.59 \pm 11\%$

The thus obtained analytical and numerical values of k_b and of the torsional stiffness k_t reported in table 2 confirm, in fact, that the uncertainty of the values of the dimensions has a marked (up to $\pm 15\%$) influence on the determined stiffness values as well.

Once the probes are calibrated, measurements aimed at determining frictional forces and the coefficients of friction μ can be performed. This implies the need to determine a factor correlating the voltage signal on the SPM photodetector to forces inducing probes' torsion. This is achieved by using TGF11 calibration samples based on trapezoidal steps along the (111) crystallographic plane of a Si substrate, allowing to establish a value of $0.26 \mu\text{N}/\text{V} \pm 0.07 \mu\text{N}/\text{V}$. Preliminary measurements are hence made on fused silica, highly oriented pyrolytic graphite (HOPG) and molybdenum-disulphide (MoS_2) samples. The measured topography of the samples is characterised by small values of surface roughness parameters R_a , R_q and R_z (table 3).

Table 3 Surface roughness parameters of the analysed samples.

	R_a/nm	R_q/nm	R_z/nm
fused silica	5.2	7.58	8.3
HOPG	5.85	8.89	7.1
MoS_2	8.04	10.18	15.0

**Figure 3.** μ vs. v for $F_N = 100$ nN (a) and μ vs. F_N for $v = 500$ nm/s (b).

The measurements of friction forces are carried out next on $500 \text{ nm} \times 500 \text{ nm}$ surfaces. A range of sliding velocities v between 5 nm/s and 500 nm/s is considered, while a constant normal force $F_N = 100 \text{ nN}$ is maintained. Then F_N is varied between $10 \text{ nN} \dots 100 \text{ nN}$ with a constant velocity $v = 500 \text{ nm/s}$. The results shown, respectively, in Fig. 3a and 3b, allow establishing that μ has a slight rising tendency for increasing v , with average μ values of 0.032 for fused silica, 0.015 for HOPG and 0.020 for MoS_2 . When F_N values are increased for a constant v , a pronounced decrease of μ is obtained, especially for lower normal forces. This could be due to the capillary effects induced by residual humidity. In the case of both measurement campaigns, the results present uncertainties of 5% to 10% .

Based on these measurements, it can be concluded that the quantitative validation of frictional phenomena in the

nanometric domain is subject to uncertainties induced mainly by the difficult determination of the stiffness of the used SPM probes, by capillary effects as well as by the large number of influencing parameters and of the resulting measurements.

3. Design of experiments

Given the above considerations, a systematic engineering approach to a finite set of experiments aimed at determining the influence of a set of parameters comprising material types, surface topography, loading, velocity, temperature and lubrication is planned. To take into consideration the interdependence of the foreseen influencing parameters and their value ranges, the widely-used methods for experimental design, based on design of experiment (DoE) methodologies, are validated. These methods are generally used for industrial purposes, thus providing results generally limited to the determination of the values of the control variables for which the dependent variable reaches local extrema. In the herein considered case, however, the functional dependency of the observed variable (friction force) in the whole range of variation of the control variables is of interest, and hence it seems appropriate to resort to the usage of design of experiments based on metamodeling techniques [6], that will hence be used to limit the number of needed measurements. In fact, the structure of metamodels can be based on measurements where one variable is modified, while the others are kept constant. On the other hand, the missing information between the points of measurement could be predicted by evolutionary algorithms [7]. This novel combined approach will have as goal the derivation of a robust design of measurement campaigns, that should provide full functional dependencies between the monitored variables.

4. Conclusion and outlook

The results of the performed preliminary measurements of friction in the nanometric domain performed on an SPM device, prove the importance of the calibration of the used probes, but also the inherent difficulties in performing a defined set of structured measurements aimed at assessing the functional dependency among the large number of influencing parameters and the value of friction. A novel approach to the design of experimental measurements is hence considered so as to allow the determination of the influence of all the characteristic parameters on the value of nanometric friction. This study should also allow new insights on the genesis of stick-slip.

The results of such a systematic engineering approach should contribute to obtain correlation function(s) linking the process variables to the value of nanometric friction, and hence to eventually extend the mathematical formulation of established friction models to the nanometric range. The newly formulated model(s) will then be validated. This will, in turn, constitute the basis for the development of advanced control typologies aimed at achieving truly nanometric positioning precision.

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