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From nanometric to meso-scale characterisation of friction using nanoindentation

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Abstract

Thin films are widely used as coatings in MEMS and NEMS as well as in precision devices, where they are often subjected to contact stresses and friction. Their frictional characteristics are hence of outmost importance. On the other hand, recently an original structured experimental methodology was developed and successfully implemented in determining the correlation between multiple process parameters and the resulting nanoscale friction, where an elaborated design of experiments approach is used, whereas the obtained measurements are analysed via advanced predictive machine learning algorithms. The same methodology is used in this work to characterise the meso-scale friction of an Al_2O_3 film, extending thus the value ranges of the most important influencing parameters beyond those achievable via scanning probe microscopy studies. A state-of-the-art nanoindentation device is hence used. The friction force is determined from nanoscratch experiments resulting from using the Berkovich tip in the range of scratch speeds from 2 μm/s to 50 μm/s and variable normal forces from 500 μN to 2 mN. The post-processing of the attained data allows obtaining a polynomial correlation function that can be used as an empirical model of thin films' meso-scale friction.

Thin film friction, experimental determination, nanoindentation, DoE, meta-modelling

1. **Introduction**

Metallic oxide films are widely used as coatings in micro- and nano-electromechanical systems (MEMS and NEMS) as well as in precision devices, where they are often subjected to contact stresses and friction. These effects result from complex interactions of various physical phenomena, thus making the attainment of a respective model difficult. An original structured experimental methodology was, however, recently proposed and successfully implemented in determining the correlation between multiple process parameters and the resulting nanoscale friction as determined via scanning probe microscopy (SPM). A design of experiments (DoE) approach, based on centroidal Voronoi tessellation (CVT) sampling, is used in this frame, whereas the obtained measurements are analysed via advanced predictive machine learning algorithms [1].

In this work the same methodology is used to characterise the friction of an alumina (Al_2O_3) film by employing nanoindentation experiments, extending thus the value ranges of the most important influencing parameters (normal force and sliding velocity) beyond those achievable by SPM studies (i.e., to a range hereafter indicated as meso-scale). In fact, in literature [2, 3] nanoindentation is nowadays proposed as a valid method of experimentally determining friction forces. A state-of-the-art nanoindentation device, allowing to attain normal load resolutions down to 50 nN and a 2 μN resolution on the lateral force, is hence used. The obtained data, in the form of a polynomial correlation function that can be used as an empirical model of thin films' meso-scale friction, provide basis for extending the available friction models to a multiscale model that could considerably improve the design of NEMS, MEMS and precision positioning systems to be used in micro- and nanometric accuracy and precision devices.

2. Experimental methodology

The experimental measurements used to determine the friction force values are carried out on a Keysight Technologies' G200 Nanoindenter with an XP indentation head by using the standard Berkovich tip. The lateral force measurement (LFM) mode is used to obtain the values of the friction force F_f while varying the normal force F_N and the sliding velocity v in the range of, respectively, from 500 μN to 2 mN and from 2 μm/s to 50 μm/s (Fig. 1) [4]. The sample used in all the experiments is an Al_2O_3 film deposited via atomic layer deposition (ALD) on an Si substrate, determined to be very homogeneous and of high purity in the whole production batch of several speciments [1]. It is determined that the average modulus of elasticity of the used sample is 180.7 GPa ± 11.18 GPa, while its average hardness is 13.94 GPa ± 0.95 GPa. During all experiments the temperature in the chamber is ~ 27 °C.

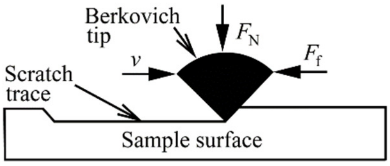


Figure 1. Scheme of the LFM scratch tests.

The range of applicable F_N loads is, in turn, determined so that it is assured that the LFM measurements are indeed characterising only the thin film (i.e, avoiding the influence of the substrate). Film's thickness of ~ 100 nm was assessed earlier by using secondary ion mass spectrometry (SIMS) [1]. Initial experiments are thus conducted by using the so-called "G-Series Ramp Load Scratch with LFM", i.e., by ramping F_N from 0 mN to 5 mN, allowing to determine that for $F_N \leq 2$ mN the "ploughing" depth is ≤ 80 nm; $F_N = 2$ mN is thus used as the upper limit of F_N. The method used for the LFM tests is, on the other hand, the "G-series single direction wear test with LFM", where a constant F_N value is imposed and a measurement along a single track is performed. To avoid the influence of any residual material on the nanoindenter tip, as well as to check its condition, after a batch of five scratches, an indentation batch of four indents in the reference sample is performed.

3. Design of experiments

As stated above, the variable parameters' design space is sampled by using a DoE approach based on the CVT method [1]. The ten measurement points (F_N - v pairs) are hence determined. The measurements in these points are repeated five times to determine the respective uncertainties, allowing to confirm the rather large (expected) variance of F_f (2 % to 35 %), associated to all small force measurements of highly stochastic phenomena such as friction [1]. The mean obtained F_f values for all the design points are analysed by using the response surface methodology (RSM), which allows the determination of a correlation function in polynomial form [5]. The used RSM method consists of multiple nonlinear regression modelling and an analysis of the resulting variance. The used regression model is a second-order (quadratic) polynomial, whose coefficients are determined via a Gauss-Newton-based iterative algorithm [5] that reached the predefined convergence criterion ($1{\cdot}10^{-6}$) after an average of 50 iterations. The resulting value of the coefficient of determination is $R^2 = 82.42$ %.

4. Results and discussion

To gain an insight into the interaction of each considered process parameter on the obtained F_f values, the experimental data is statistically analysed via a respective correlation matrix [6] shown in table 1. It can thus be observed that both variable parameters have a relatively strong positive (proportional) impact on the meso-scale friction force F_f.

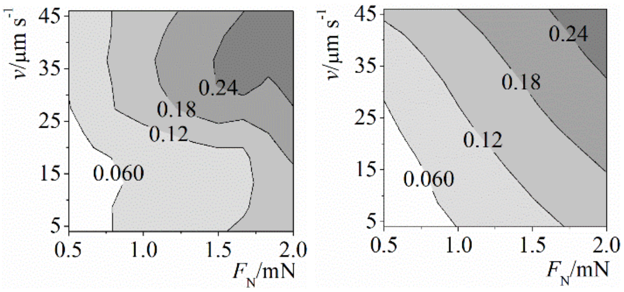
Table 1 Correlation matrix of experimental data.

	F_N	v	F_f
F_N	1		
v	0.1089	1	
F_f	0.7557	0.6576	1

In fact, higher velocities of the tip result in higher friction force values due, probably, to a rate of material build-up in front of the tip, thus implying a larger work to overcome motion resistance. Similarly, the effect of larger normal forces, as expected, induces a growth of F_f via a deeper tip's penetration into the film's surface (higher plastic deformations). The derived polynomial meta-model [5] can hence be expressed as:

$$F_f = -0.076 + 0.142 \cdot F_N + 0.0237 \cdot v - 0.0204 \cdot F_N^2 - 0.000004 \cdot v^2 + 0.00056 \cdot F_N \cdot v \qquad (1)$$

The comparison of the experimental results with the obtained model is given in Fig. 2, while the respective numerical values are given in table 2 for all the ten measurement points. The graphs in Fig. 2 show a clear similitude of the trends of the experimental and the model-derived data. The rise of the F_f values for rising v and F_N values is evident, and displays a similar trend for both the considered variable parameters, i.e., it results in similar correlation factors (cf. table 1).



(a) (b)

Figure 2. Contour surfaces of F_f (in mN) for varying v and F_N values: experimental (a) and data obtained from the model (b).

The numerical values of the obtained mean experimental F_f values and the respective relative standard deviations (RSD) values are, in turn, given in table 2. The already evidenced high overall stochasticity is the main reason for not using higher-order polynomials in the development of the model, since it would only give rise to overfitting. Furthermore, the model-derived F_f values, when compared to the mean experimental data, induce a standard error (± σ) of around 7 % to 12 %.

Table 2 Average experimental and model-based F_f values for the ten measurement points with the respective deviations.

F_N/mN	v/ µm s^{-1}	F_f/mN (exp.)	RSD/% (exp.)	F_f/mN (mod.)	Std. err./% (mod.-exp.)
1.8	4	0.139	11.80	0.13	10.97
1.1	9	0.078	12.72	0.08	9.50
0.7	13	0.051	35.13	0.05	10.40
1.6	20	0.115	34.51	0.16	7.09
0.8	24	0.098	27.64	0.09	10.86
1.5	26	0.210	4.77	0.17	12.22
0.5	33	0.066	21.49	0.07	9.03
1.1	39	0.183	2.13	0.17	11.04
2.0	46	0.285	4.76	0.28	10.23
1.4	46	0.203	5.72	0.22	9.25

5. Conclusions and outlook

The determination of the experimentally-derived meta-model of the dependence of the meso-scale friction force on variable sliding velocities and normal forces on an Al_2O_3 thin film, synthesized via ALD, is described in this work. The experimental measurements, performed via nanoindentation, result in a marked positive correlation of the process parameters on the F_f values, with similar absolute correlation values. The determined polynomial meta-model provides a good fit of the experimental data, with a relative error much smaller that the uncertainty of the experimental values themselves. Higher order models are, in turn, avoided due to a possible overfitting pitfall, related again to the high stochasticity of the measurement values.

The research performed in this work, making available an initial understanding and some general trends of meso-scale frictional phenomena of thin films with the associated experimental difficulties, provides a benchmark for future research. Our group is performing in this frame already similar measurements on titanium dioxide (TiO_2), molybdenum disulphide (MoS_2) and aluminium (Al) thin film samples. In a pursuit of truly multi-scale friction models, to be used in a broad range of technological applications requiring micro- and nanometric accuracy and precision, the attained results should, finally, be integrated with our already performed nano- [1] and macro-scale [7] tribological research.

Acknowledgements

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