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Multiscale and multiparametric tribological analysis of alumina thin films via a machine learning - based approach

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Abstract
In the context of precision mechanical systems' design, control, and exploitation, frictional phenomena in mechanical contacts represent a disturbance that, due to its highly stochastic nature, induces challenges in precision positioning applications. These physical phenomena are highly dependent on the relating conditions of frictional contacts, the scales of the applied forces, the materials in tribological contact, etc. To provide a detailed and systematic insight into these complex interactions, a machine learning - based approach is proposed in this paper. Quantitative data on the frictional forces in a single asperity contact between a silicon nitride probe and the surface of the studied alumina (Al_2O_3) thin film are obtained experimentally by using a scanning probe microscope in the lateral force microscopy measurement mode. With the aim of obtaining the best predictive model of the frictional forces within the studied variable parameters' ranges of normal forces, sliding velocities and temperatures, this data is used to train multiple ML algorithms. To verify the best performing models, allowing to attain the most accurate prediction of frictional forces on a separately measured experimental dataset, the trained ML models are scrutinised according to distinctive statistical metrics. By employing state-of-the-art experimental and numerical methods, the hence developed models provide a valuable tool in obtaining worthy predictive potentiality for multiscale tribological phenomena, depending concurrently on several relevant process parameters.

Micro- and nanotribology, thin films, experimental measurements, machine learning, high predictive accuracy

1. Introduction

The design of precision positioning systems is significantly influenced by the detrimental effects arising on the mechanical contacts of sliding bodies in relative motion, resulting in frictional forces. Frictional phenomena, recognized as one of the main disturbances in these systems, are therefore a perennial subject of extensive investigations. Such research endeavours face, however, modelling and predictive challenges due to the highly stochastic nature of friction, which is influenced by a variety of process parameters, including the type of materials in contact, contact area, normal loads, sliding velocities, temperature, and, in general, the complex interactions of the numerous interrelating physio-chemical effects at multiple scales [1].

This work aims to provide insights into these phenomena at the nano- to microscale of normal loads, while concurrently considering the variability of sliding velocity and temperature. Scanning probe microscopy (SPM) tribological measurements, focusing on the frictional interactions present in single asperity contacts, are thus performed. A thorough analysis of the resulting measurements is then performed by using various machine learning (ML) methods, allowing to attain valuable insights into the effects of scales and variable process parameters.

2. Experimental methodology

The experimental measurements of friction are carried out in this work by using the Bruker® Dimension Icon scanning probe microscope (SPM) in the lateral force microscopy (LFM) configuration, which represents a cutting-edge technique for quantifying nanometric frictional phenomena, approximating well the conditions of single asperity contacts [1, 2]. A silicon nitride (Si_3N_4) microcantilever probe moves herein laterally, while continuous contact is maintained between its tip and the surface of the studied samples. The Si_3N_4 probes apply in this case a constant normal load on a 500 x 500 nm^2 scanning area of the sample, with a set scanning resolution of 512 lines per scan.

Alumina (Al_2O_3) thin film samples are studied, since they have favourable properties as coatings due to their high hardness, wear resistance, inertness, etc. The studied Al_2O_3 thin films are deposited on Si wafer substrates by using the atomic layer deposition (ALD) technique via trimethylaluminium ($Al(CH_3)_3$) precursors, in combination with water vapour at 200 °C, utilizing the thermal mode on a Beneq® TFS 200 ALD device [3]. High-purity (6.0) nitrogen is, in turn, used as the purging gas.

To obtain the values of the forces in the normal (exerted load) and in the lateral (frictional) directions, prior to the measurements each probe is calibrated in terms of its normal and lateral sensitivity [4, 5]. To cover the normal force ranges from nano- to microscales, six different probes, each with a different geometry, are calibrated:

- Bruker® MSNL-10 E & F [6],
- BudgetSensors® AiO-Al A, B & C [7], and
- Nanosensors® PPP-LFMR [8].

In fact, the calibration of the probes' normal and lateral sensitivity yields their stiffness and resonant frequencies, allowing to determine the magnitude of the normal loads with respect to the set-point voltage V_{SP} of the *z*-axis of the SPM's piezoelectric actuator, as shown in Table 1.

Table 1 Calibrated normal load (F_N) value ranges for six different probes at variable set-point voltages (V_{SP}) of the SPM's *z*-axis piezoactuator

	Calibrated normal load F_N /µN					
V_{SP} /V	MSNL-10E	MSNL-10F	Aio-Al A	Aio-Al B	Aio-Al C	PPP-LFM R-10
1	0.007	0.03	0.044	0.2	0.282	0.24
2	0.015	0.06	0.088	0.4	0.564	0.481
3	0.022	0.091	0.132	0.6	0.846	0.722
4	0.03	0.121	0.176	0.8	1.129	0.963
5	0.038	0.152	0.22	1	1.411	1.204
6	0.045	0.182	0.264	1.2	1.693	1.445
7	0.053	0.212	0.308	1.4	1.976	1.686
8	0.061	0.243	0.352	1.6	2.258	1.927
9	0.068	0.273	0.397	1.8	2.54	2.168
10	0.076	0.304	0.441	2.38	2.823	2.409
11	0.084	0.334	0.485	2.631	3.105	2.65
12	0.091	0.365	0.529	2.851	3.387	2.891

Based on the performed calibration, probes AiO-Al A and B are, hence, selected for the measurements in the nano- and microranges of normal forces (F_N), respectively. In fact, for the AiO-Al A probe the F_N range for nanotribology measurements is from 10 to 450 nN, while the AiO-Al B probe is used in the F_N range from 300 nN to 2.8 µN.

The lateral calibration of the selected probes is carried out at different F_N values by employing Varenberg's method [5] on the calibration grating TGF11 [9]. The obtained lateral calibration constants for the AiO-Al A and B probes are determined in this way to be 0.20 µN/V and 1.18 µN/V, respectively, with a standard deviation of ± 15 % [4].

To experimentally investigate the concurrent influence of the multiple studied process parameters on the studied single asperity contacts, the testing setup is designed to cover the ranges of normal loads F_N = 10 … 2800 nN, of sliding velocities v = 5 … 3000 nm/s and of temperatures t = 20 … 80 °C.

The measurements are carried out at different temperatures in air, as measured by using the Bruker® Temperature Applications Controller (TAC), while the relative humidity in the enclosure of the used SPM device is constantly monitored via a Texas Instruments® humidity sensor coupled to an Arduino microcontroller logged to a personal computer (PC). The values of relative humidity could, therefore, be maintained throughout the measurements at 45 % ± 1 %. The humidity in the surrounding during the measurements is important due to its impact on the adhesive forces between the probes' tip and the studied samples. The measurement of the variable temperature permits, therefore, determining the resulting adhesion forces, which, in turn, allows superimposing these forces to the applied normal loads, according to the methodology thoroughly described in [4].

The set of measurement points in the multidimensional experimental design space is determined herein by employing the centroidal Voronoi tessellation (CVT) sampling method [10, 11]. This design of experiments (DoE) methodology is applied to the experimental part of the executed study by defining 50 measurement points in the multidimensional experimental variable space for different triplets of the F_N, v and t values, and obtaining the resulting frictional forces (F_f). The measurements are repeated 5 times in each measurement point, resulting in a total of 250 measurements.

The average values of the frictional force for each of the 50 CVT-based measurement points in the considered experimental space are colour coded in Figure 1. It could thus be determined that the range of the measured frictional forces is from 52.5 to 979.37 nN.

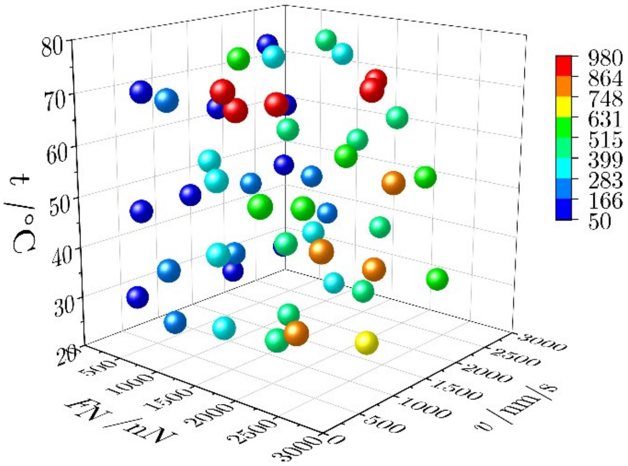

Figure 1. Values of experimentally determined frictional forces F_f for the 50 measurement points in the CVT-based DoE experimental space

Besides the measured values in the CVT-based points, the measurements are conducted for a separate set of 20 points within the same ranges of variable parameters, with, again, 5 repetitions for each point. This separate measurement set is used for the subsequent validation of the developed ML models in an "unseen" dataset that must be different from the initial CVT-based one on which the models will be trained. The dataset for these 20 points is defined by employing the Monte Carlo (MC) method [12], which provides a random sampling of the population within the defined limits of each of the variable parameters. It provides, thus, measurements for random F_N, v and t triplet values aimed at assessing the ML models' predictive performances in a realistic way. The hence obtained average values of the measured F_f values in the MC-based testing dataset are shown colour-coded in Figure 2.

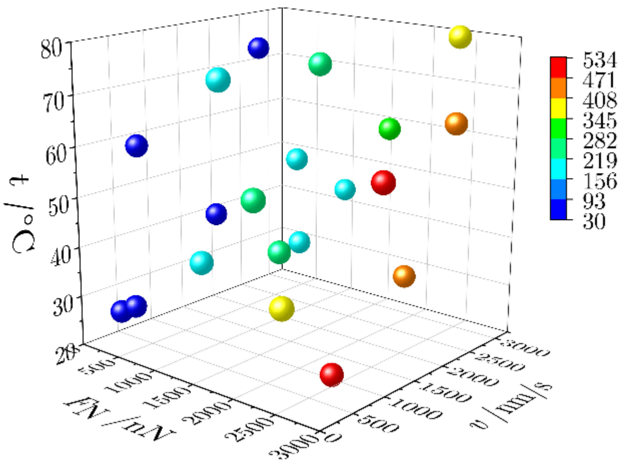

Figure 2. Values of experimentally determined frictional forces F_f for the 20 measurement points in the MC-based testing dataset

Analytical methods are additionally required to achieve a more profound understanding on how the frictional force values F_f, derived from LFM measurements, are influenced by the variation of the process parameters. Statistical analyses serve, in fact, as a foundational tool, providing benchmarks and guidelines for subsequent numerical investigations.

The correlation matrices, generated via the known Pearson's product moment correlations (PPMC) [10] on the collected F_f

data, are therefore presented in Tables 2 and 3 for the measurements in the CVT- and in the MC-based measurement points, respectively. A correlation coefficient of 1 or -1 indicates here a perfect linear correlation, with positive values reflecting a direct proportional relationship, and negative values indicating an inverse one. Higher absolute values mean, in turn, a stronger correlation, while values close to zero suggest a lack of correlation.

The data in Table 2 reveal that sliding velocity v has a small negative effect on the nanoscale frictional force, evidenced by a low correlation coefficient of - 0.150. Normal load F_N and temperature t exhibit, conversely, more significant effects, with the normal loads demonstrating a strong positive correlation, which aligns well with established friction models [1].

Table 2. Matrix of correlation coefficients for the influencing parameters on F_f for the CVT measurement points

	t	F_N	v	F_f
t	1			
F_N	- 0.060	1		
v	0.042	- 0.110	1	
F_f	0.229	0.857	- 0.150	1

The correlation matrix obtained for the MC-based measurements (Table 3), also confirms a high positive impact of the normal loads on the frictional force, while, in contrast to the values obtained for the CVT dataset, the effect of v suggests higher positive values. This corroborates once more the stochastic nature of friction. In contrast to the CVT correlations, the MC-based correlation of F_f to t shows, moreover, a lower positive impact, suggesting that the sample used in the MC case was probably drier, and thus the surface-adsorbed layer of water resulted in a smaller meniscus effect on the single asperity contact.

Table 3. Matrix of correlation coefficients for the influencing parameters on F_f for the MC measurement points

	t	F_N	v	F_f
t	1			
F_N	0.187	1		
v	0.343	0.200	1	
F_f	0.035	0.947	0.281	1

To further explore the influence of each of the variable process parameters on the nanoscale frictional forces F_f, and with the aim of obtaining suitable predictive models, the experimental data will, therefore, be used next to train various ML algorithms.

3. Modelling methodology

Various ML algorithms are used in this work: multi-layer perceptron (MLP), random forest (RF) ensembles, and support vector regression (SVR), whose salient features have been outlined in previous art [10, 13]. The respective models are developed by using the Scikit-learn [14] and MatLab® implementations. All the used ML methods are therefore used to develop nanoscale friction models for the considered sample material via the following steps [10]:

- data preparation (normalization, standardization),
- training of the algorithms on the experimental (CVT-based) datasets, and
- optimization of their characteristic hyper-parameters.

The metrics used for the evaluation of the thus developed models' predictive performances are mean absolute errors (MAE), root-mean square errors (RMSE), and coefficients of determination R^2 [10, 15]. The values of the hence attained predictive results (metric values) on the testing (MC-based) dataset are reported in Table 4, where R^2 is selected to be the most dominant (but not exclusive) metric, with values of R^2 above 0.7 considered as good predictive performances. It can be seen here that the predictive performances of all the developed ML models show similar high predictive performances, with the RF algorithm being slightly better - thus allowing to predict 81 % of the variance of the nanoscale frictional force values (R^2 = 0.814).

Table 4. Comparison of predictive performances on the test (MC-based) dataset for the used ML models of nanoscale friction

Algorithm	RMSE	MAE	R^2
MLP	397.35	345.66	0.765
RF	290.48	235.97	0.814
SVM	170.01	89.70	0.660

4. Results and discussion

The predictive performances of all trained ML models are shown in Figure 3 in comparison to the average measured frictional force values F_f obtained for the 20 MC-based experimental points, i.e., the "unseen" dataset, providing an assessment benchmark. It can be observed that all developed ML models, despite their relatively high R^2 performance metric, result in over-estimated F_f values for most of the measurement points. In fact, around 75 % of measured data is over-estimated by even up to 200 % by all the models, even though the general trend is following positively the experimental data. This clearly confirms that the predictive performances of the ML models for such complex and stochastic phenomena, as is nanometric friction, is hard to assess with statistical methods, and the conventional performance metrics provide merely an indication of the quality of the trend-following capabilities of the predictive models.

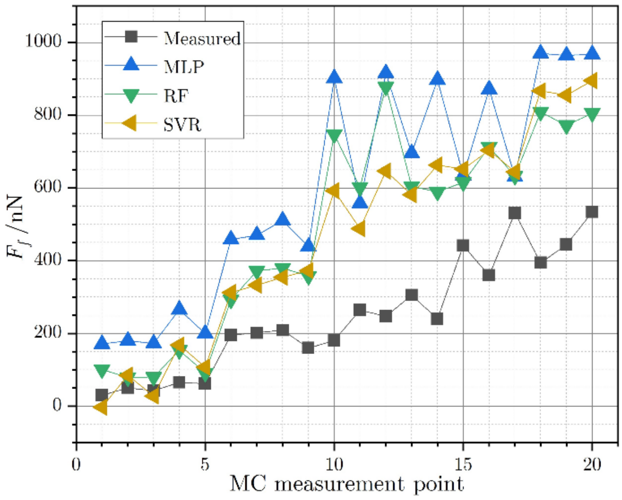

Figure 3. Values of the predicted and the experimentally measured F_f values for the 20 measurement points in the MC-based testing dataset

What is more, although according to the R^2 metric the best predictive model is RF, it still exhibits significant oscillations in the predicted values, albeit to a smaller degree than the MLP model. The SVR model exhibits, in turn, the smoothest prediction, even though its R^2 value is the lowest. Therefore, SVR would seem to have the highest potential to be further refined by using more experimental data for its training.

5. Conclusions and outlook

By applying black-box machine learning models to experimentally gathered nanotribological data, an analysis of multiscale and multiparametric nano- and micrometric frictional behaviour of alumina thin films, produced via atomic laser deposition, is presented in this work.

The obtained results reveal that it is feasible to effectively predict the concurrent effects of multiple process parameters on the frictional forces, achieving satisfactory accuracy levels, with R^2 values ranging from 0.66 to 0.81. The developed machine learning models provide therefore solid basis for further modelling endeavours employing artificial intelligence (AI) models, which could potentially provide further insights into the complex interactions of the diverse parameters influencing nanoscale friction.

The performed research presents, finally, a glimpse to the experimental and modelling intricacies involved in studying nanofrictional phenomena, which need to be investigated further to obtain the insights required for ultra-high precision positioning applications.

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