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# Nanotribological characterization of an X39CrMo17-1 steel thin-film via measurement-based machine learning methods

Marko Perčić,[1, 2] Saša Zelenika [1, 2] and Igor Mezić [1, 3]

[1] *University of Rijeka, Faculty of Engineering & Centre for Micro- and Nanosciences and Technologies, Vukovarska 58, 51000 Rijeka, CROATIA*
[2] *University of Rijeka, Centre for Artificial Intelligence and Cybersecurity, Laboratory for AI in Mechatronics, Radmile Matejčić 2, 51000 Rijeka, CROATIA*
[3] *UC Santa Barbara, Department of Mechanical Engineering, Santa Barbara, CA 93105, USA*

szelenika@uniri.hr

**Abstract**
Frictional characteristics are an important factor in the design and exploitation of precision positioning systems and components. In fact, friction is a complex stochastic phenomenon depending on an intricate interplay between materials' physical and chemical properties, as well as on the contact conditions influenced by the exerted normal forces, sliding velocities, contact area, temperature etc. Experimental measurements of the nanoscale friction force of an X39CrMo17-1 high-alloy steel thin film, deposited on a silicon wafer, are obtained in this work via elaborated lateral force microscopy (LFM) measurements performed on a scanning probe microscope using silicon-nitride microcantilever probes. The thin-film samples are synthesised by using pulsed laser deposition, which enables obtaining precise stochiometric elemental properties. The LFM measurements allow obtaining single-asperity contact conditions in the studied tribo-pair, which is studied experimentally considering the mentioned external influences as the variable process parameters. In order to obtain an in-depth insight into the dependencies of nanoscale friction on these parameters, the obtained experimental data is used to train various machine learning (ML) algorithms. The resulting predicted values of the nanoscale friction force are therefore obtained by using random forest, multilayer perceptron and support vector regression ML methods. In order to assess their predictive performances, all the used algorithms are validated on a separately measured test dataset. The best predictive performances are obtained by using the support vector regression algorithm, resulting in the highest achieved coefficient of determination ($R^2$), allowing the prediction of 90 % of the variance of the experimental measurements.

Nanotribology, high-alloy steel, thin-films, machine learning

## 1. Introduction

Due to a wide array of concurrent detrimental effects that occur in the mechanical contacts of sliding bodies, the accuracy of precision positioning systems presents a major design challenge [1]. Frictional phenomena present therein a major disturbance, constituting an important ongoing research topic. The study of frictional effects, with their inherent stochastic nature, creates, thus, a modelling and prediction challenge, dependent on material types, contact area, normal loads, sliding velocities, temperature, and, in general, the interplay of these and other physio-chemical effects and interactions [1]. The majority of the resulting complex frictional effects have their foundation in the so-called single asperity contact of two surfaces in relative motion [1]. To provide better predictive and compensating frameworks, enabling the attainment of increasingly precise mechanical positioning systems, it is therefore important to acquire the fundamental physical insights into the frictional phenomena that occur in the nanometric domain. With this goal in mind, nanotribological experimental measurements are performed in this work, and the obtained data are used in multiple machine learning (ML) algorithms so as to obtain a qualitative insight of the frictional interactions in the single asperity contact conditions depending on the concurrent influence of the variable process parameters, i.e., normal loads, sliding velocities and temperature.

## 2. Experimental methodology

The scientific approach to the study of nanoscale (single asperity) frictional phenomena performed in this work is based on experimental measurements conducted via a Bruker Dimension Icon scanning probe microscope (SPM) [2] available at the Centre for Micro- and Nanosciences and Technologies (NANORI) of the University of Rijeka, Croatia [3]. The instrument is used in the atomic force microscopy (AFM) configuration – the only state-of-the-art method of measuring nanometric frictional phenomena while experimentally approximating the conditions of a single asperity contact [4-6].

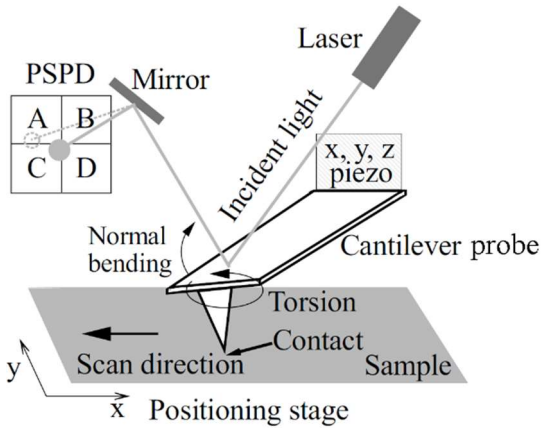


**Figure 1.** Scheme of the used LFM measurement configuration [7].

The frictional force on the analysed samples is hence measured by using the lateral force microscopy (LFM – known also as the Friction Force Microscopy (FFM)) measurement modality, shown schematically in Fig. 1, while the experiments are controlled by using the respective NanoScope software. A silicon nitride Bruker SNL-10 microcantilever probe [8] moves hence laterally, while a continuous contact is maintained between its tip and the surface of the studied samples. The probe is applying a constant normal load on a 500 x 500 $nm^2$ area of the sample, and the set scanning resolution is 512 lines per scan. To obtain a precise value of the forces in the normal (exerted load) as well as in the lateral (frictional) directions, prior to the measurements each probe is calibrated in terms of its normal and lateral sensitivity [7]. These calibrations are critical in converting the voltages generated by the laser beam, reflected via the optical lever induced by the motion of the probe caused by its interactions with the surface of the sample, i.e., by the normal, the adhesive and the friction forces, on the SPM position-sensitive photo-diode (PSPD), into quantitative force spectroscopy measurements [7].

The nanotribological experimental measurements are carried on with three variable process parameters: normal load $F_N$ in the range from 10 to 150 nN, sliding velocity $v$ varying from 5 to 500 nm/s, and temperature $\vartheta$ changing from 20 to 80 °C. Due to the complex and time-consuming nature of the used experimental technique, the number of points selected for the measurements on the used samples is 50, with five repetitions for each measurement point, resulting in a total number of 250 measurements. The performed experiments are configured by applying a novel design-of-experiment (DoE) methodology, i.e., by sampling the experimental design space within the mentioned boundaries using the centroidal Voronoi tessellation (CVT) technique [9, 10] that enables a uniform distribution of the measurement points in the multivariate experimental design space. The CVT sampling method is efficiently implemented in this work in the commercially available GoSumD software [11].

Although most mechanical precision components and assemblies are designed and manufactured from high-quality stainless steel, which combines excellent mechanical properties with the inertness to atmospheric and exploitation conditions, this material type is seldomly analysed in the micro-, and, especially, in the nanodomain. In this work this challenge is approached by studying the nanometric tribological properties of a high-alloy martensitic steel X39CrMo17-1, characterised by a complex chemical composition, whose nanotribological characterisation is not reported in available literature. The respective samples are synthesised as thin-films on a silicon wafer substrate by using the pulsed laser deposition (PLD) method [12] at the facilities of the Institute of Physics, in Zagreb, Croatia. PLD, shown principally in Fig. 2a, is a physical vapour deposition (PVD) technique where high power laser pulses in a vacuum chamber are used to melt, vaporize and ionize the surface of the target material – in this case the high-alloy steel. This ablation produces a plasma plume, visible in Fig. 2b, that rapidly expands away from the target, while the ablated material is deposited on the surface of the substrate (the silicon wafer). An optical emission spectroscopy (OES) method is used for the in-situ characterization of the obtained plasma, which enables optimizing the pulsed laser characteristics, thus obtaining the control of the deposition process so as to achieve the desired atomic structure of the deposited thin-films. The technique is widely used in the production of a broad range of superconductive and insulating circuit components, as well as for biocompatible and medical applications. Its main advantage is that it enables the stochiometric transfer of the material from the target to the surface of the substrate, allowing a precise chemical composition of the used target material to be deposited in the form of thin-films [12].

The thus synthesised X39CrMo17-1 thin-film samples are characterised by means of X-ray photoelectron spectroscopy (XPS) and of secondary ion mass spectrometry (SIMS) measurements on the instruments available in the mentioned NANORI Centre [3]. Such measurements allow confirming the elemental composition, as well as establishing the 100 nm thickness of the steel thin-film deposited on the Si substrate.

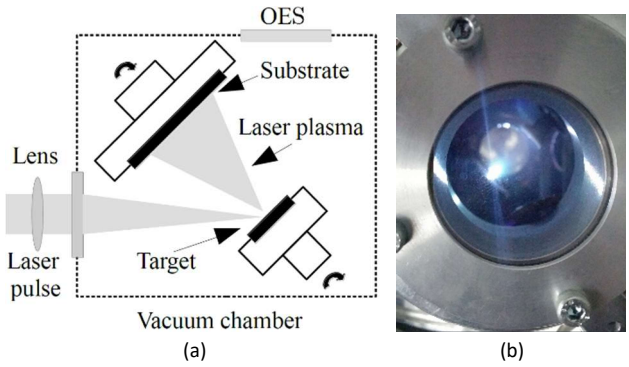


**Figure 2.** Scheme of the used PLD process (a), and plasma plumes in the vacuum chamber as seen while synthetizing the X39CrMo17-1 samples.

The values of the nanometric friction force $F_f$ in all the 50 measurement points, defined via the used CVT DoE methodology within the ranges of the concurrently varied process parameters, and obtained by performing the described LFM measurements, are shown colour-coded in Fig. 3. The stochastic nature of the studied nanoscale friction force is clearly evidenced here, with the higher $F_f$ values obtained for higher normal loads $F_N$ and lower temperatures $\vartheta$, while the influence of sliding velocity $v$ does not show an intuitive correlation with $F_f$.

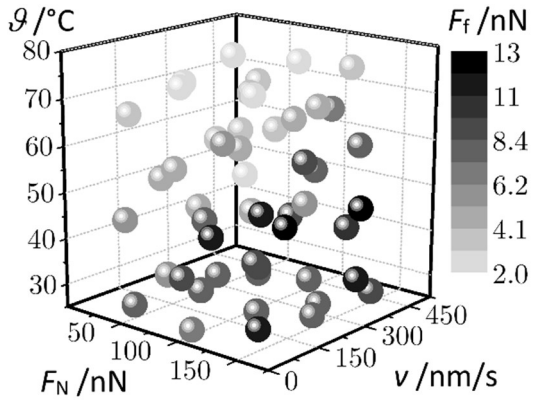


**Figure 3.** Colour-coded distribution of experimentally determined nanometric friction force values $F_f$ in the 50 measurement points for the X39CrMo17-1 thin-film sample obtained via PLD vs. the influencing parameters temperature $\vartheta$, total normal load $F_N$ and sliding velocity $v$.

**Table 1**. Matrix of correlation coefficients for the influencing parameters on the nanometric friction force $F_f$ in the CVT measurement points.

| | $v$ | $F_N$ | $\vartheta$ | $F_f$ |
|---|---|---|---|---|
| **v** | 1 | | | |
| $F_N$ | 0.032 | 1 | | |
| $\vartheta$ | 0.028 | 0.079 | 1 | |
| $F_f$ | 0.018 | 0.36 | -0.57 | 1 |

Further methods of analysis are needed to obtain a deeper insight into the sensitivity of the $F_f$ values, attained via the LFM measurements, on the variation of the influencing process parameters. Statistical analysis is hence used as a benchmark, as

well as to obtain guidelines for the subsequent numerical analyses. The correlation matrix obtained by using the Pearson's product moment correlation (PPMC) [9] on the set of acquired $F_f$ data is summarised in Table 1. In PPMC a correlation coefficient of 1 or -1 represents a perfect (linear) correlation of positive (proportional) or negative (inversely proportional) dependence of the dependent variable on the considered influencing parameter, with the higher absolute values indicating a stronger dependence. A zero (or near-zero) value indicates, in turn, that there is no correlation. From the values deported in Table 1 it can therefore be deduced that the effect of sliding velocity $v$ has a low impact on the nanoscale friction force $F_f$, with a low correlation factor of 0.018. The effects of the normal load $F_N$ and of temperature $\vartheta$ have, in turn, respectively a high positive and a high negative effect on the friction force. The high positive effect of the normal load is, obviously, predictable from the conventional friction models [1]. The cause of the negative and relatively high effect of temperature on the friction force, can, in turn, be speculated to originate from the evaporation of an adsorbed water layer on the surface of the studied samples. In fact, evaporation would diminish the attractive force of the meniscus, and thus lower the normal load.

To gain even deeper insights into the effects of each of the variable process parameters on the nanoscale friction force, the obtained experimental data are used next to train multiple ML algorithms.

## 3. Machine learning modelling

To obtain the predictive models linking the process variables to the value of the nanometric friction force $F_f$, the results obtained experimentally are analysed, as depicted in Fig. 4., by using state-of-the-art ML numerical methods.

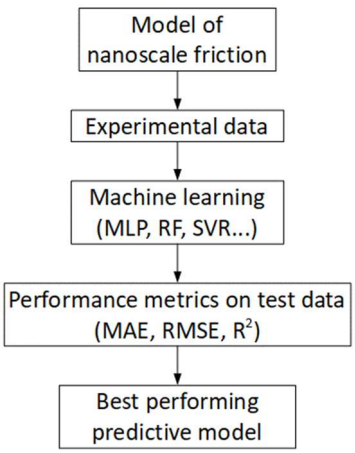


**Figure 4.** Implemented methodology for the development of a predictive model of nanoscale friction using ML methods [13].

Various ML algorithms are thus used in this work: multi-layer perceptron (MLP), support vector regression (SVR), and random forest (RF) ensembles; the respective models are developed by using the Scikit-learn [14] and GoSumD [11] implementations.

MLP is a deep artificial neural network, meaning it consists of more than two layers of perceptron, i.e., algorithms used for the supervised learning of functions as binary classifiers. The latter are composed of an input layer receiving the signal, an output layer that evaluates or predicts the input and, in between those two, an arbitrary number of hidden layers that are the true computational engine of the algorithm [15].

RF is a type of ensemble ML algorithm called bootstrap aggregation or bagging. Bootstrap is a powerful statistical method for estimating a quantity from data samples, such as mean values. RFs are based on combining multiple decision trees into a single stronger predictor. Each tree is trained therein independently with a randomly selected subset of the experimental data [16].

Finally, in the support vector machine (SVM) algorithms a hyperplane is selected to best separate the points in the input variable space, by their class [15, 17]. In 2D this separation is easily visualized, but the same approach works also for multidimensional data. The SVM (or the SVR, for the support vector regression variant, as applied in this research) learning algorithm seeks for the coefficients that result in the best separation of the classes by the hyperplane. The best or optimal hyperplane is the one that has the largest distance between the hyperplane itself and the closest data points, i.e., the biggest margin. Only these cases are relevant in defining the hyperplane and constructing the classifier, i.e., a function which describes a set of instances that have common features [17].

All the described methods are used to develop nanoscale friction models for the considered sample material via the following steps:

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

During the training of all the models, overfitting is avoided and the best performing hyper-parameters are validated by employing $k$-fold cross-validation. Cross-validation is a technique used to evaluate the predictive models by partitioning the original sample (the experimental CVT-based dataset) into a training set used to train the model, and a validation set used to evaluate its optimal parameters [18]. All the ML models used in this study are, thus, subject to 10 x 10 cross-validation, i.e., 10 repetitions of 10-fold cross-validations are performed on the complete CVT-based training dataset.

The metrics used for evaluating the developed ML models' predictive performances are the mean absolute errors (MAE), the root-mean square errors (RMSE) and the coefficients of determination $R^2$ [19]. The evaluation metrics is applied on separate experimental measurements, i.e., on an un-seen testing dataset, with the results evaluated in terms of the best possible predictions [13].

The values of the hence attained predictive results (metric values) on the testing datasets are reported in Table 2, 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 thus be seen that the predictive performances of all the developed ML models show similar high predictive performances, with the SVR algorithm showing slightly better performances – thus allowing to predict 90 % of the variance of the nanoscale friction force values ($R^2$ = 0.90).

**Table 2** Comparison of predictive performances on the test datasets for the used ML models of nanoscale friction.

| Algorithm | RMSE | MAE | $R^2$ |
|---|---|---|---|
| **RF** | 1.21 | 0.554 | 0.81 |
| **MLP** | 1.78 | 1.53 | 0.85 |
| **SVR** | 1.39 | 0.927 | **0.90** |

## 4. Results and discussion

The best-performing SVR ML model is finally used to plot the solutions of the predicted nanoscale friction force values $F_f$ within the whole variable range of values of the studied process parameters. The visualization of the surface plots of the thus developed solutions is given in Fig. 5, where in each diagram two process parameters are varied, while the third one is kept

constant.

In Fig 5a the influence of temperature ($\vartheta$) on the nanoscale friction force values shows a sloped parabolic effect, which can be attributed to the complex interplay of the thickness of the adsorbed water layer and the resulting adhesive forces that have a strong effect on $F_f$. What is more, the concurrent effect of sliding velocity ($v$) shows a mild quasi-linear effect on $F_f$, which is consistent with the above preliminary observations (cf. Table 1). The dominant positive effect of the normal load $F_N$ gives rise to a strong quasi-linear influence on $F_f$ in the case of constant temperature, although this effect is concurrently affected by temperature – as it can be appreciated in Fig. 5b, where, for the lowest and highest values of temperature, the effect of normal load varies greatly. All of these observations are confirmed also in Fig. 5c.

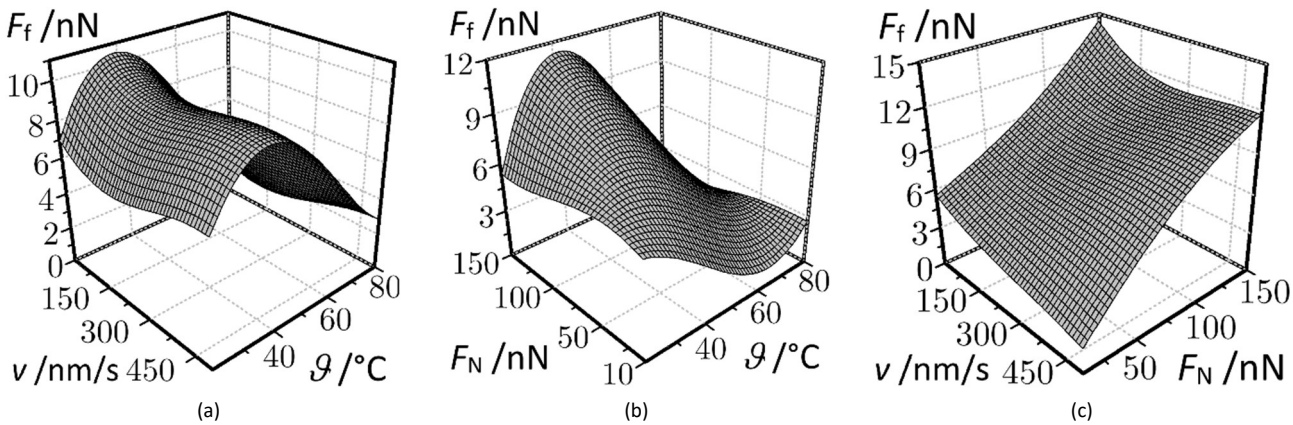


**Figure 5.** Surface plots of the SVR-based ML solutions of the nanoscale friction force values $F_f$ for a constant total normal load ($F_N$ = 100 nN) (a), a constant sliding velocity ($v$ = 250 nm/s) (b), and constant temperature ($\vartheta$ = 80 °C) (c), for the PLD synthesized X39CrMo17-1 steel thin-film samples.

Overall the results corroborate, therefore, the fact that frictional phenomena at the nanoscale (i.e., in the single asperity contact conditions) depend greatly on the conditions in the contact region of the two materials in the tribo-pair.

## 5. Conclusions and outlook

The analysis of the frictional behaviour in the nanometric domain, performed in this work by using the black-box ML models on experimentally acquired data, allows evidencing that it is possible to provide an effective prediction of the influence of multiple process parameter on the value of the friction force with satisfactory levels of accuracy, i.e., with $R^2$ values ranging from 0.81 to 0.9. The developed ML models provide also novel insights into the multi-variable effects on nanoscale frictional phenomena of the X39CrMo17-1 steel thin-film synthesized by using PLD.

The visualizations of models' predictive results clearly shows complex concurrent effects of the considered variable process parameters on the nanoscale friction force, thus justifying further studies at the higher and the lower ranges of scales, i.e., at the molecular and the microscales. The obtained predictive correlations will thus be used for studies comprising frictional scaling effects, but also for predictive control modelling, and other fields of study - all with the goal of enabling a further development of precision positioning mechanical systems and components.


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