LFM CHARACTERIZATION OF TiO_2 FILMS ON A NANOINDENTER

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1. Introduction

Thin oxide films are commonly used as coatings in precision devices, where they are often subjected to sliding contacts and friction whose impact can be identified via experimental techniques [1].

An original structured experimental procedure, based on advanced design-of-experiments (DoE) algorithms, allowing to determine the correlation between multiple process parameters and the resulting nanoscale friction, was recently proposed [2]. Such an approach was applied to study, via nanoindentation measurements in the lateral force microscopy (LFM) mode, the effects of the most important influencing parameters on mesoscale friction [1]. In fact, nanoindentation is a valid method to experimentally characterize friction forces in the μN range [3]. LFM measurements performed on a nanoindenter are hence used in this work to assess the properties of a TiO_2 thin-film deposited on an Si substrate. In the space defined by the experimentally determined ranges of sliding velocities and normal forces, the measurement points are determined via DoE routines [2]. Experimental results allow thus establishing correlation functions that can be used as models of the mesoscale frictional behavior of TiO_2 thin films.

2. Materials and methods

The TiO_2 film is deposited on an Si (100) substrate via atomic layer deposition on a Beneq TFS 200 device at 150 °C. A $TiCl_4$ precursor is used for Ti, whereas H_2O is employed as the oxygen source, with the respective pulsing times of 250 and 180 ms, followed by N_2 purging of 3 and 2 s, respectively.

Nanoindentation and nanoscratch measurements are, in turn, carried out on a Keysight Technologies' G200 Nanoindenter, allowing normal and lateral load resolutions of 50 nN and 2 μN, respectively. A fresh Berkovich tip is used to determine the mechanical properties of the sample, whereas LFM tests are performed with a used tip with a slightly "blunter" end. During all the measurements the temperature in the chamber is kept stable at ~ 28 °C.

The mechanical properties of the Si substrate are determined first. The obtained average modulus of elasticity E is 173.4 ± 1.1 GPa, while the average hardness H is 11.9 ± 0.1 GPa. The properties of the TiO_2 film are determined next via a standardized method that enables compensating the influence of the substrate on the results. The average TiO_2 film E value is hence determined to be 144.4 ± 5.2 GPa, while the average H value is 9.5 ± 0.4 GPa.

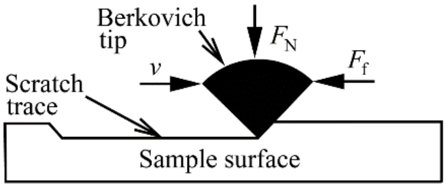

Fig. 1. Scheme of the LFM scratch test.

Fig. 2. Typical F_f vs. scratch distance LFM curve.

The second set of experiments is performed by ramping the applied normal load F_N from 0 to 10 mN so as to determine the maximum F_N value for which the displacement of the indenter tip into the surface does not exceed film's thickness (< 150 nm). The F_N range is thus determined to be from 500 μN to 4.5 mN. According to the experimental findings in [1], the range of sliding velocities v is, in turn, from 2 to 50 μm/s. Based on these values, the parameters' design space is sampled by using the Latinized Centroidal Voronoï Tessellation (LCVT) DoE approach [2]. A set of ten $F_N – v$ measurement pairs is hence determined. The single direction LFM test is used next (Fig. 1) to obtain the values of the friction forces F_f. A single track measurement is repeated 5 times for each $F_N - v$ pair at different locations on the sample, so as to estimate the respective uncertainties. Typical F_f vs. scratch distance graphs are thus obtained (Fig 2).

3. Results and discussion

The mean obtained F_f values for the 10 measurement points are analyzed by using the response surface methodology (RSM), where linear and full quadratic polynomial regression models, whose coefficients are determined iteratively via the Gauss-Newton algorithm, are used [4]. To gain insight into the influence of F_N and v on F_f, the data is statistically analyzed via a correlation matrix (Table 1). It can thus be inferred that F_N has a strong proportional impact on F_f, inducing its growth via tip's penetration into the film (higher plastic deformations). Rising sliding velocities result also in rising F_f values, but the influence of v on F_f is notably smaller than that of F_N.

Table 1. Correlation matrix of experimental data.

	F_N	v	F_f
F_N	1		
v	0.092	1	
F_f	0.964	0.315	1

The derived meta-models have the coefficients of determination R^2 of 0.984 and 0.995, respectively. The linear and full-quadratic meta-models can hence be expressed as [2, 4]:

$$F_f = -0.1055 + 0.1599 \cdot F_N + 0.0032 \cdot v \quad (1)$$

$$F_f = 0.007 + 0.103 \cdot F_N - 0.002 \cdot v + \\ + 0.011 \cdot F_N^2 + 0.0001 \cdot v^2 + 0.0001 \cdot F_N \cdot v \quad (2)$$

Table 2. Average F_f values and respective deviations.

Experimental data				Model predictions			
F_N [mN]	v [µm/s]	Exp. [µN]	St. dev. [%]	Lin. mod. [µN]	St. err. [%]	Full quad mod. [µN]	St. err. [%]
0.6	42	150	8.1	120	14.8	140	1.8
1.1	23	110	5.3	140	30.6	140	24.9
1.4	9	180	2.5	150	20.2	160	11
2	38	330	1.9	340	2.0	310	5.3
2.4	12	310	3.7	320	1.3	310	1.1
2.5	33	370	4.7	400	6.6	370	1.8
3	4	400	1.4	390	4.4	410	0.8
3.5	21	470	6.8	520	11.2	500	7.3
4.1	50	730	1.3	710	2.5	740	1.5
4.2	29	680	7.3	660	3.9	650	4.5

The experimentally and numerically determined F_f values, and the respective deviations, are reported in Table 2. It can be observed that the variance of the experimental F_f values is from 1 to 8 %. When compared to the mean experimental data, the model-derived F_f values induce a standard error e limited to ca. 20 % for the linear and of 10 % for the full quadratic one, except for the second measurement point that shows clearly bigger deviations. Further experimentally obtained F_f values for randomly chosen $F_N - v$ pairs, allow establishing that in all the considered cases e is < 10 % for both models.

4. Conclusions and outlook

The experimentally-derived meta-model of the dependence of mesoscale friction on sliding velocities and normal forces on a TiO_2 thin-film, is described in this work. The measurements, performed by using a nanoindenter in the LFM measurement mode, result in a marked positive correlation of the process parameters on the friction force, although the normal force has clearly a higher impact. The determined polynomial meta-models provide a good fit with the experimental data.

The study will be continued on other thin-film samples (ZnO, MoS_2 and Al). The attained results on the mesoscale shall finally be integrated with performed nano- [2] and macro-scale [5] friction characterization, with the aim of developing a multi-scale friction model to be used in applications aimed at ultra-high accuracy and precision.

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