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Nano- to microscale experimental characterisation of the tribological behaviour of Al_2O_3 thin films via lateral force microscopy

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Abstract

Stochastic frictional phenomena in mechanical contacts are an aggravating challenge in controlling precision mechanical systems, highly dependent on the involved physical origins and scales of the applied forces. The fundamental insights into this complex phenomenon can be beneficial for the design and development of new precision equipment or the optimal selection of functional coatings. Novel experimental findings about the scaling effects at the nano- to microscales of normal forces exerted on a single asperity contact are described in this work. The study is conducted via the scanning probe microscope (SPM) in the lateral force microscopy measurement mode, allowing quantitative measurements of single asperity frictional forces between the SPM silicon nitride probe and the surface of Al_2O_3 thin film samples. The measurements are structured to cover normal forces ranging from a few nN to 2.8 μN by using two different SPM probes. To achieve precise quantitative results, the used probes are carefully calibrated.

Nano- and microtribology, lateral force microscopy, scaling effects, thin films, experimental measurements

1. Introduction

Precision positioning systems face a significant design challenge due to the negative effects that occur in the mechanical contacts of sliding bodies. Frictional phenomena as a major disturbance in these systems, are currently being extensively researched. Such studies pose a modelling and prediction challenge due to their inherent stochastic nature, which is influenced by factors such as material type, contact area, normal loads, sliding velocities, temperature, and the complex interplay of other physio-chemical effects and interactions at different scales [1]. To gain insights into such phenomena, tribological experimental measurements are conducted in this work at the nano- to microscale of normal loads with the aim to determine the frictional interactions occurring under single asperity contact conditions.

2. Experimental methodology

The investigation of single asperity frictional phenomena is based in this study on an approach that relies on experimental measurements carried out via 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, while approximating the conditions of a single asperity contact [1, 2] (Figure 1). 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 alumina (Al_2O_3) thin film samples deposited on Si wafer substrates by using the atomic layer deposition (ALD) technique. Al_2O_3 thin films have, in fact, favourable properties as coatings due to their high hardness, wear resistance, non-reactivity, etc. The Si_3N_4 probes apply here 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.

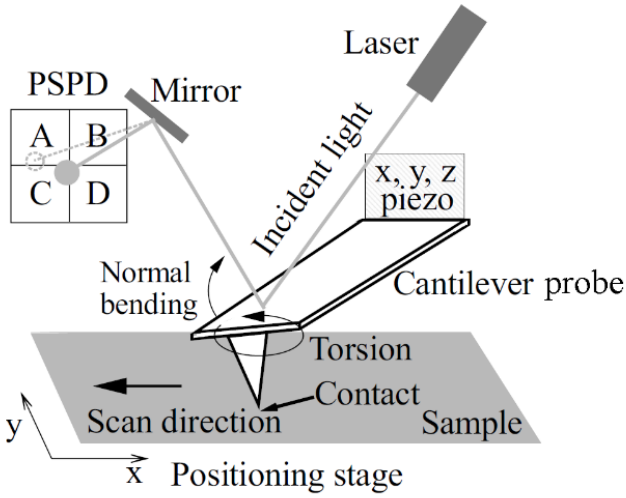

Figure 1. Scheme of the used LFM measurement configuration [3]

To obtain a precise value 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 [3]. To cover the normal force ranges from nano- to microscales, six different probes, each with a different geometry, are selected: Bruker MSNL-10 E & F [4], BudgetSensors AiO-Al A, B & C [5], and Nanosensors PPP-LFMR [6]. The calibration of probes' normal and lateral sensitivity yields, thus, their stiffness and resonant frequencies.

The achievable normal force values F_N for the calibrated probes with respect to the set-point voltage V_{SP} of the z-axis piezoelectric actuator are shown in Figure 2. Based on these results, probes of type AiO-Al A & B are hence selected for the measurements in the nano- and micro-ranges, respectively. In fact, for the AiO-Al A probe the F_N range for nanotribology measurements is from ca. 4 to 470 nN, while the AiO-Al B probe is used in the F_N range from 235 nN to 2.8 μN.

The lateral calibration of the selected probes is carried out at different F_N values by employing the calibration grating TGF11 and using Varenberg's method [7]. The obtained lateral calibration constants for the AiO-Al A & B probes is thus determined to be 0.184 μN/V and 1.192 μN/V, respectively, with a standard deviation of ~ 13 %.

The LFM measurements are conducted next on the 500 nm scan size of the surfaces of the Al_2O_3 thin film samples in a 21 °C temperature-controlled environment with a constant scan rate of 1 Hz, resulting in a 1 μm/s sliding velocity.

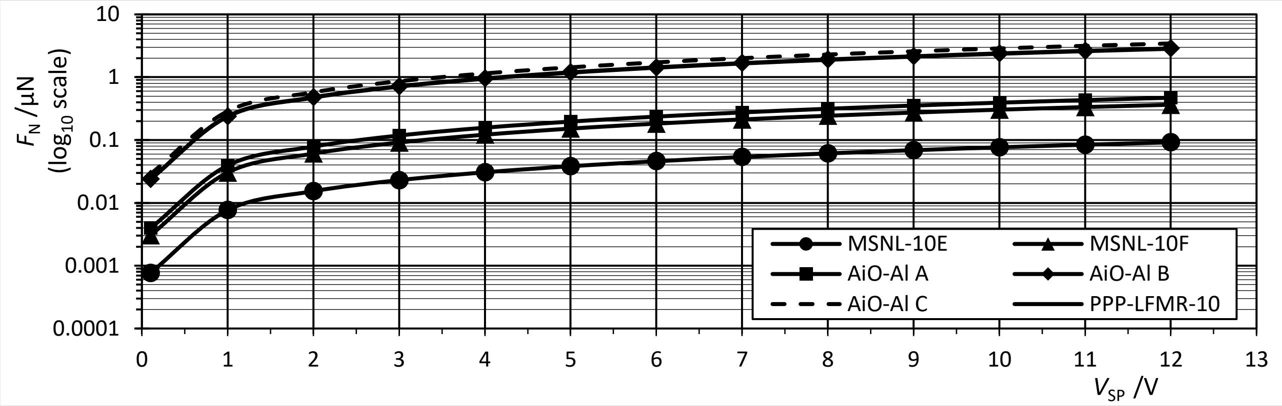

Figure 2. F_N values achieved for each of the studied probes at different set-point voltages V_{SP} of the z-axis piezoactuator

3. Results and discussion

The measured LFM voltages for all F_N values are hence processed with the lateral calibration constants so as to obtain the values of the frictional force F_f in the nN to μN range of normal loads, as shown in Figure 3. The expected quasi-linear trend is visible for both curves, while the transition between the nN and μN ranges shows some discrepancies. What is more, the values in the higher μN range exhibit some non-linearities, which is a sign of an enlargement of the surface area of the tip of the probes due to wear. This effect is tendentially rather high for large normal loads, giving rise to an enlargement of the adhesion force in the contact region between the tip of the probes and the samples' surface.

The instabilities visible for the higher nN and μN F_N ranges can, in turn, be an indication that the deformation limit of the selected probes is reached, implying that future measurements should be carried on with more probes that would enable covering the complete range of the foreseen normal loads.

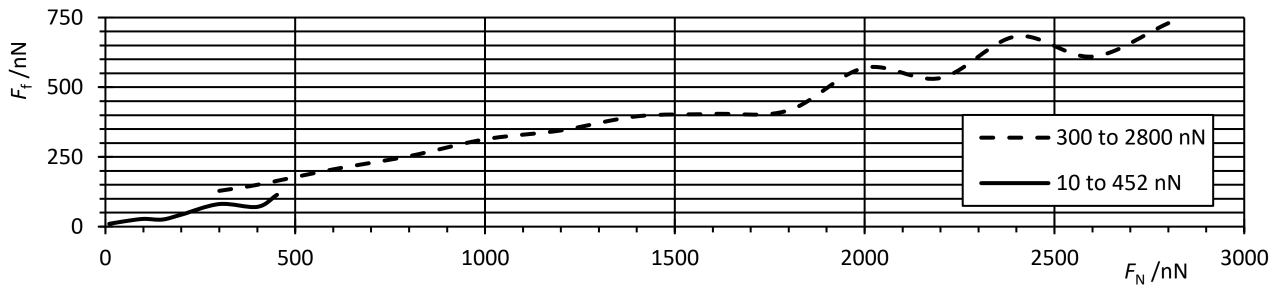

Figure 3. F_f values on the Al_2O_3 thin film samples vs. the variable normal load F_N in the nN to μN range

4. Conclusions and outlook

The performed study into the multiscale tribological properties of the studied Al_2O_3 thin film material allows establishing that, although the general trends of the frictional force variability for normal loads in the nano- to microscales show the expected quasi-linear behaviour in line with the conventional frictional models, the transitional and higher-value ranges of F_N require further in-depth and structured studies to determine all the involved coupled effects. Additional experimental studies of the influence of the probes' tip wear with the resulting adhesion effects, as well as probes' deformation limits are, therefore, needed to fully understand the frictional scaling phenomena, thus providing the necessary insights for the development of predictive tribological models as well as of the corresponding compensation typologies.

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