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NANOWEAR OF ATOMIC FORCE MICROSCOPE TIPS: MODELING AND EXPERIMENTS

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Abstract. In this paper, a simple analytical model to predict the nanowear of atomic force microscope tips is presented and experimentally validated. The model is based on the assumption that the energy consumed to remove the unit volume is a material/structural (i.e. size-dependent) parameter. Nanoscratch tests show that this hypothesis is plausible and, more importantly, that the specific energy is close to the material strength at the considered size scale, i.e. the theoretical material strength at the nanoscale. With our approach, predictions on nanowear, e.g. for an optimal nanomachine design, can be made *a priori*.

1. INTRODUCTION

Understanding the wear of atomic force microscope (AFM) tips is of primary importance, since it can strongly affect the related experimental results, such as hardness measurements during AFM nanoindentation [1,2]. Several tribological models have been presented so far, as summarized in a few recent review books by Bhushan [3-5]. While all these models are certainly valid for their range of applicability, without contradicting them, here we present a wear model based on energetic assumptions, that we found applicable at the nanoscale, suited for the tip-substrate interaction of AFM probes.

At macroscale, wear rates have usually been quantified based on Archard's approach, in which the friction coefficient is assumed constant and the dissipated energy is proportional to the product of normal load, sliding distance, and friction coefficient. For example, Ramalho *et al.* [6] performed experiments using a sliding tribometer to study the wear of different steel alloys against high-speed steel. They asserted that the friction energy is dissipated mainly though three processes: rise in temperature, wear particle generation and entropic changes associated with material transformation. Their energy-based model was then based on the assumption that the energy dissipated is divided in such a way that relative amounts of different main parts of energy consumption remain constant. Similarly, Colaco *et al.* [7] performed pin-on disk tribological tests on ultra high molecular weight polyethylene and their results showed that the energy dissipation is mainly caused by elasto-plastic deformation of the polymer and wear of the polymer. All these models which have been relating "dissipated energy" with wear rates seem to be applicable at the macroscale; however, this does not guarantee their applicability at nanoscale as many other factors are involved.

In particular, at the nanoscale, van der Waal's forces are relevant, leading to adhesion between the surfaces in contact [8]. However, if the contact forces

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are high relative to the adhesive forces, it can still be assumed that the dissipated energy is consumed in removal of material. Accordingly, a theoretical model is formulated in the next section and validated through experiments.

2. NANOWEAR THEORY

During wear, the relation between the energy dissipated W and the corresponding volume V of removed material [9] is $W=kV^{d/3}$, where k is a (size-independent) constant and d is the physical dimension of the domain related to the generated fractal nanodust, usually between 2 and 3 (as a consequence of the competition between surface and volume in the predominant mechanisms, e.g. wear and adhesion). The previous equation simply states that the energy consumed is not proportional to the volume nor to the surface but in general to an intermediate fractal domain with dimension d. A fractal exponent is implicitly a sign of a statistical approach, so the conclusions drawn from this analysis may not apply directly to individual phenomena (such as the detachment of a particular nanoparticle from the tip), but frames the general tendency. The previous formula can be re-written as, $W=cV$ [10], where $c=kV^{(d-3)/3}$ is a size-dependent parameter describing the nominal wear strength of the material, i.e. the specific energy needed to remove the material (the size-effect is a consequence of the competition between surface and volume). Thus, the model predicts that "smaller is harder/stronger" ($d<3$). At large size scales $c \approx \sigma_c$ [11], i.e. such a parameter is coincident with the material macro-strength σ_c. Similarly, at the nanometer scale, one could conjecture that $c \approx \sigma_{th}$ (the theoretical strength of the material), i.e. it becomes coincident with the material nanostrength, expected to be close to the theoretical material strength. This hypothesis has to be verified and, if confirmed, could lead to a new simple and powerful tool for *a priori* nanowear predictions, which are fundamental for an optimal nanomachine design.

Let us apply these concepts to the wear of the AFM tips. The power supplied is $\dot{W}=\mu F v$, where μ is the friction coefficient, F the normal contact force and v the relative sliding velocity. This power is spent in removing the material from the substrate (subscript 1) and from the tip (subscript 2) [12,13]; accordingly, the power balance for the tip is $\eta_2 \mu F v = c_2 \dot{V}_2$, where η_2 is the power fraction absorbed by the tip. Analogously, $\eta_1 \mu F v = c_1 \dot{V}_1$ denotes the energy balance for the substrate, where η_1 is the power fraction dissipated on the substrate and $1-\eta_1-\eta_2$ is the power fraction dissipated through other mechanisms, e.g. thermal dissipation from the tip/substrate system. Thus, we calculate the following wear rates:

$$\dot{V}_i \approx \frac{\eta_i \mu F v}{c_i}, \; i = 1,2 . \tag{1}$$

3. NANO-WEAR EXPERIMENTS

To validate this simple formulation for calculating wear rates, wear tests were performed by scanning Silicon Nitride AFM probes (DNP NP Series – Veeco Instruments) on an Ultra Nano Crystalline Diamond (UNCD) surface [14] using an AFM (Digital Instruments AFM – Nanoscope Dimension 3100). The experiments involved the measurement of cantilever stiffness (i), friction coefficient between probe and substrate (ii), and volume removed during scratching (iii).

(i) The AFM probes used were commercially available Silicon Nitride probes with a specified cantilever stiffness of 0.58 N/m. However, each of the cantilevers used for wear tests were individually characterized for stiffness by deflecting them against a reference cantilever of known stiffness. The set up is schematically shown in Fig. 1. The reference cantilever used for these measurements was 400 mm x 3 μm x 2.49 μm in dimension, made of silicon, with stiffness of 0.289 N/m (Force Calibration Probes from Veeco Probes). Given the stiffness of the reference cantilever, the stiffness of the unknown cantilever can be deduced from the force-deflection response using the following formula [15]:

$$K_1 = K_0 \left[\frac{DC}{N} - 1 \right], \tag{2}$$

where K_0 is the stiffness of the reference cantilever, D is the total deflection of the two cantilevers and N/C is the deflection of the unknown cantilever, C being the deflection sensitivity of the instrument set during the experiment and N being the deflection signal in Volts. Details of this method can be found elsewhere [15]. The deflection was repeated 10 times, retracting the tip between each trial, to obtain a statistical average of the cantilever stiffness. Stiffness values were measured for each of the probes used (see Table 1).

(ii) The friction coefficient between the substrate and AFM probes was measured to be 0.047±0.008, which falls within the range of values (0.04-0.05) reported in the literature [16]. As the direction of frictional force changes during trace and retrace, making the cantilever twist in opposite directions, this

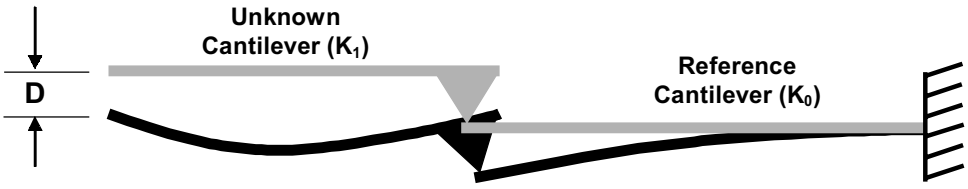

Fig. 1. Schematic diagram illustrating the cantilever stiffness measurement. Grey and black represent the geometries before and after deflection respectively.

measurement was based on the difference between trace and retrace responses during scanning. The coefficient of friction can be calculated as [16]:

$$\mu = 2h\frac{\Delta H_1 + \Delta H_2}{H_0 L}, \qquad (3)$$

where H_0, ΔH_1 and ΔH_2 are shown schematically in Fig. 2, h is the height of the pyramidal tip and L is the length of the cantilever on which the tip is mounted. ΔH_1 and ΔH_2 are the measured differences in tip height from the un-deflected position H_0 due to the additional cantilever bending caused by the frictional forces acting in opposite directions during trace and retrace. One of the probes used for these experiments was characterized under Scaning Electrone Microscopy (SEM) to measure the actual values of h and L.

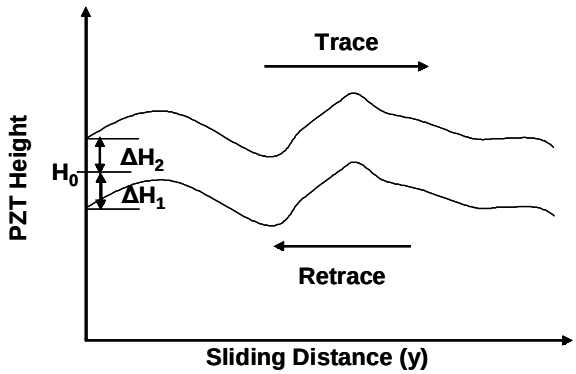

Fig. 2. Schematic diagram showing the difference between the trace and retrace responses, used to calculate the friction coefficient.

(iii) In order to perform the wear test, the AFM probe was brought in contact with the substrate at a specified contact force calculated (based on cantilever stiffness, deflection sensitivity of the AFM and the initial deflection set point) and the scanning was performed at a given speed for a given period of time. Four experiments were done using different AFM probes. In order to measure the material removal during wear, high magnification SEM images of those AFM probes were taken before and after the scanning process. In addition, the substrate was monitored during scanning to check if there was any significant wear on it. The wear in the substrate was found to be negligible (as expected, since in our case $\sigma_{th1} >> \sigma_{th2}$, thus $V_1 >> V_2 = 0$); however, the material removed from the probes was significant. The amount of material removed from the probes was calculated using the SEM images. Figs. 3a and 3b show SEM images of Chip No. 4 before and after the wear test respectively. The edges were extrapolated to complete the pyramidal shape of the tip and the missing volume was calculated by pixel counting. Compensation for tilt of the probes while imaging under SEM was included in the measurements of change in volume, and error was estimated by pixel counting according to the resolution of the image. Table 2 summarizes the scanning parameters used and the wear observed for four different tips. It is assumed that all the material is removed from the tip and this assumption is strengthened by the argument that the tip undergoes cyclic stresses while scanning the substrate, whereas each point on the substrate comes in contact with the tip just once during a scan. Considering $V_2 \approx \eta_2 \mu F v / c_2$ with $\eta_2 \approx 1$ (no wear in the UNCD substrate), $\mu \approx 0.05$ (measured), $c_2 \approx \sigma_{th,2} \approx E_2/30 \approx 10$GPa (see http://www.accuratus.com/silinit.html for an estimation of the Si_3N_4 theoretical strength, E_2 is the Young's

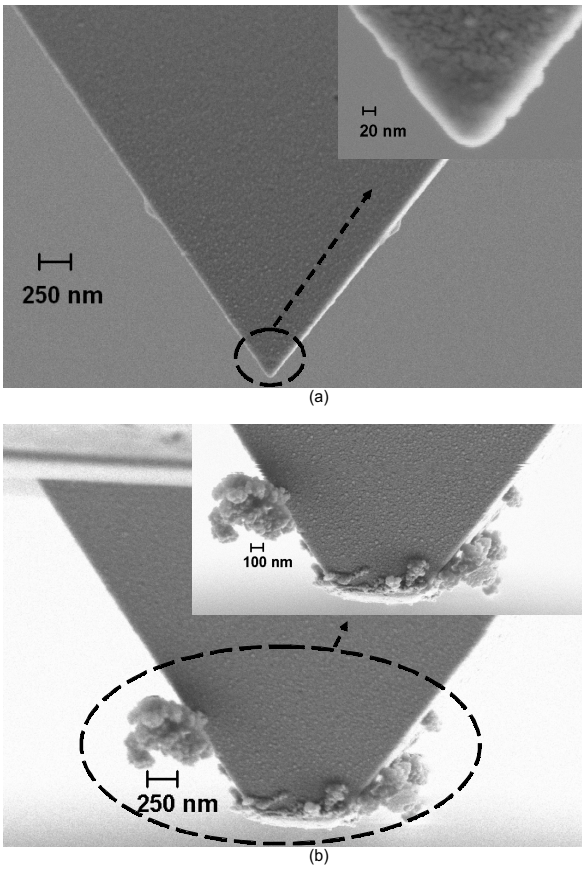

Fig. 3. High Magnification SEM images of a Silicon Nitride AFM probe before (a) and after (b) the wear test.

Table 1. Measured cantilever stiffness.

Probe No.	Expected Stiffness [N/m]	Measured Stiffness [N/m]
1	0.58	0.5674±0.0794
2	0.58	0.5782±0.0834
3	0.58	0.5610±0.0870
4	0.58	0.5769±0.0763

modulus) we calculate the material removals reported in Table 2. A relevant agreement between predictions and measurements, without invoking any best-fit parameter, is observed (with the exception of the Chip No. 1, approximately three times harder than the other chips, perhaps a consequence of a slightly modified material structure or some other mechanism involved, like breaking off of the tip).

4. CONCLUSIONS

Summarizing, we have presented a simple model to predict nanowear, confirmed by *ad hoc* nano-scale measurements on AFM tips. In particular, we have found that the classical macroscopic hypothesis of material removal proportional to energy consumption is still nominally applicable at the nanoscale (i); but the nominal constant of proportionality (specific energy) is strongly size-dependent (ii); and,

Table 2. Experimental results and theoretical predictions. Each sub-cell correspond to a different scanning condition.

Chip No.	Contact Force [nN]	Scanning Speed [μm/s]	Scanned Distance [mm]	Material removal observed during wear tests [μm^3]	Material removal predicted [μm^3]	
1	110.6 +/- 15.5	10.0	10.24	0.020991 +/- 0.000134	0.00581	0.069719
	221.3 +/- 31.0	50.9	51.20		0.058099	
	110.6 +/- 15.5	10.0	10.24		0.00581	
2	116.2 +/- 16.8	10.0	10.24	0.084823 +/- 0.001350	0.005974	0.101291
	348.7 +/- 50.3	50.9	51.20		0.089344	
	116.2 +/- 16.8	10.0	10.24		0.005974	
3	255.3 +/- 39.6	76.3	61.44	0.065061 +/- 0.000508	0.078029	
4	285.6 +/- 37.8	40.0	61.44	0.088581 +/- 0.000498	0.088166	

more importantly, it is surprisingly close to the material strength, thus to its theoretical value at the nanoscale (iii). With our approach, predictions on nanowear, e.g. for an optimal nanomachine design, can thus be made *a priori*.

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REFERENCES

[1] L. Calabri, N. Pugno, A. Rota, D. Marchetto and S. Valeri // *Journal of Physics- Condensed Matter* **19** (2007) 395002.
[2] N. Pugno // *Acta Materialia* **55** (2006) 1947.
[3] B. Bhushan, *Introduction to Tribology* (John Wiley, New York, 2002).
[4] B. Bhushan, *Nanotribology and Nanomechanics: An Introduction* (Springer, Berlin, 2005).
[5] B. Bhushan, *Handbook of Nanotechnology, Second ed*. (Springer, Berlin Heidelberg, 2007).
[6] A. Ramalho and J.C. Miranda // *Wear* **260** (2006) 361.
[7] R. Colaco, M.P. Gispert, A.P. Serro and B. Saramago // *Tribology Letters* **26** (2007) 119.
[8] E. Rabinowicz, *Friction and Wear of Materials, Second Edition* (John Wiley and Sons, Inc., 1995).
[9] A. Carpinteri and N. Pugno // *Journal of Applied Mechanics* **69** (2002) 854.
[10] A. Carpinteri and N. Pugno // *International Journal of Numerical and Analytical methods in Geomechanics* **26** (2002) 499.
[11] N. Pugno // *Journal of Applied Mechanics* **73** (2006) 697.
[12] A. Carpinteri and N. Pugno // *Journal of mechanics and control* **5** (2004) 57.
[13] A. Carpinteri and N. Pugno // *Wear* **257** (2004) 408.
[14] D. M. Gruen, S. Liu, A. R. Krauss and X. Pan // *Journal of Applied Physics* **75** (1994) 1758.
[15] C.T. Gibson, S. Myhra and G.S. Watson // *Nanotechnology* **7** (1996) 259.
[16] J.-A. Ruan and B. Bhushan // *Transactions of the ASME* **116** (1994) 378.