A COMPARISON OF LATERAL CALIBRATION TECHNIQUES FOR QUANTITATIVE
FRICITION FORCE MICROSCOPY

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ABSTRACT

A comparison of two lateral force calibration techniques for friction force microscopy is presented. We used methods developed by Ogletree et.al. [1] and Ruan and Bhushan [2] to measure the friction response between the atomic force microscope (AFM) probe and a silicon sample and to obtain lateral force calibration factors. The factors were used to characterize the friction behavior and interfacial shear strength of a silicon nitride (Si₃N₄) probe-ultra high molecular weight polyethylene (UHMWPE) interface.

INTRODUCTION

Although numerous friction force microscopy studies reporting quantitative friction values on the nanoscale exist, they often differ in the technique used for lateral (friction) force calibration. Widely used lateral force calibration techniques include those developed by Ogletree et.al. (OG) [1], Ruan and Bhushan (RB) [2], Cain et.al. [3] and Liu et.al.[4]. Here, we compare two of these methods to obtain quantitative friction measurements on silicon and ultra-high molecular weight polyethylene (UHMWPE) using standard V-shaped silicon nitride cantilevers. UHMWPE is a popular material used in total joint replacements (TJR). The tribological behavior of the interface between a polyethylene cup and the metallic femoral head in TJRs is of critical importance to joint reliability and durability. Nanotribological techniques provide a viable alternative to macroscale tests in evaluating the tribological performance of materials for TJRs.

EXPERIMENTAL

Friction force microscopy was performed using a Dimension 3100 AFM (Nanoscope IV, Digital Instruments Veeco Metrology) with commercially available Si₃N₄ cantilevers with integrated probes. Prerequisite for quantitative friction force microscopy are evaluation of normal spring constant and probe radius. Normal spring constants were evaluated using the calibrated lever technique [5]. For our cantilever, we obtained a normal spring constant of 0.32 N/m (quoted value was 0.58 N/m). The probe radius was evaluated to be 40 nm using a TGT01 sample (Mikromasch, USA) and SPIP software (Image Metrology, www.imagemet.com).

LATERNAL FORCE CALIBRATION

OG’s method [1] aims at determining the response of a cantilever to lateral forces in terms of its normal force response. Briefly, in this approach, the tip is slid over a surface with two known slopes (TGG01 silicon calibration grid from Mikromasch) and the force calibration is done by measuring the lateral force signal as a function of applied normal load. The TGG01 sample was aligned such that the ridges were perpendicular to the lateral scanning direction. The friction response is monitored as a function of normal applied load on the upward and downward slopes in terms of the half-width of the friction loop and lateral offset of the friction loop from the mean. The data so obtained is plotted in Fig. 1. The slopes of the plots from Fig. 1 are used in the equations presented in [1] to arrive at a lateral calibration factor of 2.16 µN/V.

Briefly, in RB’s method [2], the tip is first scanned across the surface in a direction parallel to the long axis of the cantilever over a range of normal loads. The coefficient of friction (COF) is obtained from the slope of a plot (Fig. 2 a) of profile separation (‘Trace-Retrace’, TMR value at each scan location) as a function of average piezo center and probe/cantilever geometry. Next, scans perpendicular to the long axis of the cantilever beam are performed at various normal loads and the lateral deflection response is plotted as shown in Fig. 2b. By equating the slope of this plot to the previously obtained coefficient of friction, a calibration factor is obtained. We obtained 15.6 nN/V for our setup on silicon.

Table 1 summarizes the results of the two methods. There is an order of magnitude difference in the COF and calibration
factor between the two methods on the same sample. The probe shape and size was verified to be predominantly unchanged throughout our experiments (from 40 to 42 nm). Assumptions and precautions associated with each technique were carefully complied with. A more detailed analysis to identify reasons for this discrepancy is being carried out.

Table 1: Comparison of data: lateral calibration methods

<table>
<thead>
<tr>
<th>Method</th>
<th>Coefficient of friction on Si</th>
<th>Calibration factor (nN/mV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ruan and Bhushan</td>
<td>0.017</td>
<td>0.0156</td>
</tr>
<tr>
<td>Ogletree et al.</td>
<td>0.307</td>
<td>2.288 ± 0.054</td>
</tr>
</tbody>
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We note that Cain’s method [3] utilizes the initial stick portion of a friction loop to get a lateral calibration factor. This method is suitable only for stiff cantilevers and colloidal probes. For our cantilever and load regime, the effect of the lateral contact stiffness was significant and a single value of lateral calibration factor could not be obtained.

Figure 1: OG method – friction response as a function of normal response

The calibrated cantilever setup was used to measure friction on an UHMWPE (K-Mac Plastics, Michigan) sample. The uncalibrated friction data is shown in Fig. 3

Figure 3: Friction force vs applied load – Si₃N₄ cantilever on a UHMWPE sample

The friction force was calibrated using the calibration factor obtained with RB’s method [2]. The interfacial shear strength of the material pair was calculated from the friction force data by fitting a Johnson-Kendall-Roberts model to the data. The value thus obtained was 5.85 MPa. The value obtained using OG’s method resulted in a shear strength of 0.86 GPa. Macroscale measurements report a value of 6.95 MPa [6]. The results shown in this paper are specific only to commercially available v-shaped Si₃N₄ cantilevers.

ACKNOWLEDGMENTS

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REFERENCES