

# Atomic Force Microscope Probe based Controlled Pushing for Nano-Tribological Characterization

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**Abstract**— Using an Atomic Force Microscope (AFM) probe as a nano-manipulator, micrometer- and nanometer-sized objects, especially particles, are pushed on substrates for characterizing the object-substrate friction parameters and behavior in various environments, e.g. air, liquid and vacuum. Two possible nano-tribological characterization methods are proposed in this paper: (1) Sliding the micro/nano-object on the substrate while it is attached to an AFM probe, (2) Nano-robotic pushing of the micro/nano-object with the sharp tip of an AFM probe. Modeling of these methods are realized and experiments are conducted for the latter method using a piezoresistive AFM probe as a 1-D force sensor and nano-manipulator. In the experiments, 500 nm radius gold-coated latex particles are pushed on a silicon substrate. Preliminary results show that different frictional behavior such as sliding, rolling, and rotation could be observed, and shear stresses and frictional behavior could be estimated using these techniques at the nano-scale.

**Index Terms**— Nano-manipulation, nano-tribology, Atomic Force Microscopy, micro/nano-forces, nano-mechanics.

## I. INTRODUCTION

**I**MAGING of any type of micrometer- and nanometer-sized object in any type of environment down to atomic and molecular resolution has become possible by the invention of Atomic Force Microscope (AFM). On the other hand, an AFM probe has been recently utilized as a simple nano-manipulator for pushing based positioning of nanometer-sized objects [1], [2], cutting [3], nano-lithography applications, etc. Hence, it can be changed from a passive observation tool to an active manipulation tool. Beside of these applications, there is another new emerging application of AFM-based pushing such that micro/nano-objects can be pushed on substrates in order to understand their tribological properties [4], [5], [6], [7], [8], [9], [10]. Understanding the nano-tribological behavior can help in designing new solid lubricants, e.g. for hard disk storage technology, new polishing materials for Chemical Mechanical Polishing technology, repeatable and reliable manipulation of micro/nano-objects on surfaces, etc.

The nano-scale friction differs from the macro-scale friction and henceforth objects are almost wearless, adhesional friction dominates at low loads [11], and friction becomes an intrinsic property of the particular interface [12]. There has been many works on nano-tribology of AFM probe tips on different surfaces [13], [14]. In these studies, AFM tip is contacted and moved on a surface and frictional forces are measured by torsional bending of the probe. However, these studies are limited to specific tip materials, and cannot characterize different type of motions of objects on substrates such as

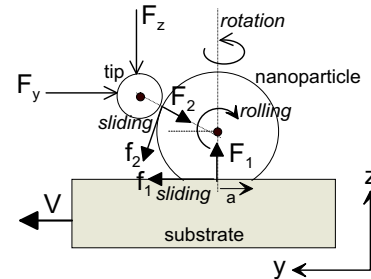


Fig. 1. AFM tip-driven motions of a nano-particle on the moving substrate: sliding, stick-slip, rolling, sticking, and rotation.

rolling, rotation, etc. Therefore, this paper contributes to the literature by introducing an autonomous nano-robotic manipulation system for nanometer-sized object frictional parameter and behavior characterization in any environment and any mode of motion. Two possible characterization methods are proposed in the paper: (1) Sliding the micro/nano-object on the substrate while it is bonded to an AFM probe, (2) Nano-robotic pushing of the micro/nano-object on the substrate with the sharp tip of an AFM probe. Quasi-static motion equations of these methods are derived, and experiments are conducted for the latter method using a piezoresistive AFM probe as a 1-D force sensor and nano-manipulator. In the experiments, 500 nm radius gold-coated latex particles are pushed on a silicon substrate, and frictional parameters and behavior are estimated using the proposed models and experimental pushing force data.

The paper is organized as follows. In Section 2, the main concept of the nano-tribological characterization system is defined with assumptions, and sliding, rolling, and spinning frictional models are given. In Section 3, two possible methods are introduced, and the automatic pushing control scheme for the second method is explained. Section 4 describes the latex particle pushing experiments and results, and conclusions with future directions are reported finally.

## II. PROBLEM DEFINITION

The aim is to develop methods for measuring the friction between a micro/nano-object and the substrate while observing the object motion behavior, e.g. sliding, stick-slip, rolling, and spinning. As a possible method, the object-substrate motion scheme in Figure 1 is proposed. Here, the micro/nano-object is fixed to an AFM probe base or pushed by an AFM probe tip, and, by moving the substrate with a controlled constant speed,  $F_y$  and  $F_z$  forces are measured in real-time to predict the object motion behavior and object-substrate frictional parameters. Here, the following assumptions are made:

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- Friction is taken to be mainly adhesional without wear and ploughing effects by assuming low load, smooth and unreactive surfaces, and high adhesional forces.
- The substrate is moving with a constant speed  $V$  to neglect the inertial effects of the substrate and the positioning stage. Also,  $V$  is chosen to be high so that the stick-slip behavior is not observed.
- Micro/nano-particles with radius  $R$  are selected as the object. However, the same characterization methods can be directly applied to any micro/nano-object such as carbon nano-tubes, nano-wires, nano-crystals, DNA, RNA, cell, etc. by generalizing the contact mechanics models.
- The AFM probe tip shape is assumed to be spherical with radius  $R_t$ .  $R_t$  is selected to be much smaller than  $R$  in order to have relatively lower tip-particle adhesion than the particle-substrate interface during their detachment. Thus, the tip-particle sticking problem after pushing is minimized.
- The tip and substrate are chosen to be the same material for simplicity and less uncertainty.
- The substrate is selected as a conductor or semiconductor material, and it is electrically grounded for preventing the electrostatic adhesion accumulation due to triboelectrification.
- Ambient environmental conditions are assumed with 40–60% relative humidity and 23 °C temperature. Therefore, a water layer could exist on the surface depending on their hydrophilicity and humidity level which would change the tribological behavior [15]. However, the same method is valid for any other environment such as vacuum or liquid.
- All surfaces are assumed to be not contaminated and very smooth. Thus, the roughness effect on adhesion and friction is neglected.

During the AFM probe tip based pushing method, possible particle motions are sticking, sliding, stick-slip, rotation, and rolling. Therefore, nano-scale models for the sliding and rotational friction forces are required. Sliding is the most general case in AFM based lateral frictional force microscopy (stick-slip is also observed frequently in atomic scale imaging and slow speeds [14]). Since the friction is mainly adhesional with the given assumptions, sliding friction becomes as [21], [22]

$$\begin{aligned} f_1 &= \tau A \\ f_2 &= \tau A_2 \end{aligned} \quad (1)$$

where  $\tau$  is the shear stress of the particle and substrate contact and the tip and particle contact points, and  $A = \pi a^2$  and  $A_2 = \pi a_2^2$  are the real contact areas and  $a$  and  $a_2$  are the real contact radii for the particle-substrate and tip-particle interfaces respectively. In general,  $\tau$  is a function of contact area and pressure [23] and the first-order approximation is given by

$$\tau = \tau_0 + c(P + P_0) \quad (2)$$

where  $P = L/A$  is the pressure for a given contact area  $A$  and a normal load  $L$ ,  $P_0$  is the capillary pressure in the case of a liquid layer, and  $c$  is the proportionality constant. For a

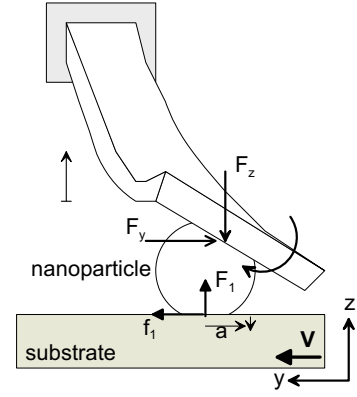


Fig. 2. The sliding motion of a nano-particle, which is bonded to the rectangular AFM probe (front view).

bulk material,  $\tau$  is expected to be  $\tau \approx G/29$  [22] theoretically, where  $G$  is the shear modulus.

Rolling has been addressed in a few nano-related works. Butt *et al.* [16] modeled the rolling friction force  $f_r$  between two identical micro-particles as

$$f_r = 6\pi\gamma_p d \quad (3)$$

where  $\gamma_p$  is the particle surface energy and  $d$  is the critical rolling displacement. Note that  $\sigma \leq d \leq a$  is assumed, where  $\sigma$  is the mean distance between neighboring atoms and  $a$  is the contact radius. Another group [17] has reported the rolling friction moment model based upon Amonton's law, where the rolling friction moment  $M_r$  is given by

$$M_r = \mu_r(L + F_a) \quad (4)$$

with  $F_a$  is the adhesion force, and  $\mu_r$  is the rotational friction coefficient. However, since the adhesion based friction is assumed to be dominant, the particle-substrate rolling friction is modeled as

$$f_1^\psi = \tau_\psi A \quad (5)$$

where  $\tau_\psi$  is the rotational friction coefficient. For most materials,  $f_1^\psi$  is much less than  $f_1$ . If this would be the case at the nano-scale, then mostly rolling would start before sliding depending on the applied pushing load.

### III. METHODS

For a particle-substrate interface friction characterization, the following two methods are proposed in this paper:

#### A. Method I

Let a nano-particle be attached to the bottom end of a rectangular AFM probe as shown in Figure 2. The AFM probe attached with a micro-particle was originally investigated for measuring the pull-off force against the particle-substrate adhesion with well-defined geometries [18]. Adapting it to nano-tribological measurements in this paper, the particle that is attached to the AFM probe base is contacted and moved on the substrate laterally in the  $y$ -direction with a constant speed  $V$ , and the torsional deflection is measured to compute the *direct* particle-substrate friction  $f_1$ . This method can be

applied to frictional characterization of micron or 100s of nanometer size of particles where the object and substrate geometries are well defined. However, for 10s of nanometer sizes of particles, attaching a single nano-particle and sliding control could be very challenging, and this method allows only the characterization of the sliding and stick-slip frictional behaviors since the particle is glued to the nano-probe base.

For this method, the normal deflection  $\Delta\zeta$  and torsional twisting  $\Delta\theta$  from the initial positions are measured using an optical detection system. Since the substrate moves along the  $y$ -axis only,  $F_x = 0$  here. Thus,

$$\begin{aligned}\Delta\zeta &= \frac{F_z \cos\alpha}{k_z} - \frac{F_z \sin\alpha}{k_{xz}} \\ \Delta\theta &= S_\theta \frac{F_y}{k_y^\theta} \\ k_z &= \frac{Ewt^3}{4l^3} \\ k_{xz} &= \frac{2L_s}{3} k_z \\ k_y^\theta &= \frac{Gwt^3}{3lH^2} \approx \frac{L_s^2}{2} k_z \\ k_y^b &= \frac{Etw^3}{4l^3} = \left(\frac{w}{t}\right)^2 k_z\end{aligned}\quad (6)$$

where  $k_z$ ,  $k_{xz}$ ,  $k_y^\theta$ , and  $k_y^b$  are the spring constants for bending due to the normal force, bending due to the lateral force moment, twisting due to the lateral force, and lateral bending due to the lateral force, respectively. Also,  $E = 2(1 + \nu)G$ ,  $G$ ,  $\nu \approx 0.33$ ,  $l$ ,  $w$ ,  $t$ ,  $L_s = l/H$ , and  $H$  are the Young's modulus, shear modulus, Poisson's ratio, length, width, thickness, structural constant, and tip height of the beam, respectively,  $S_\theta$  is the sensor coefficient for the twisting measurement, and  $\alpha$  is the beam tilt angle from the base guaranteeing the point contact of the particle with the substrate.

$F_y$  twists the probe but also deflects it laterally. However, since  $F_y$  is to be measured from  $\Delta\theta$  twisting only,  $k_y^b \gg k_y^\theta$  condition should be held for possible measurement errors. This means that following condition should be held for the probe:

$$wH \gg lt. \quad (7)$$

Then, by keeping  $k_z$  in a reasonable range,  $t$  and  $l$  should be minimized while  $w$  and  $H$  ( $= 2R$  in this case) are maximized for this method.

Assuming a quasi-static slow motion of the particle on the substrate, at each instant:

$$f_1 = F_y. \quad (8)$$

Thus, by measuring  $\Delta\theta$ ,  $f_1$  is computed. On the other hand,  $\Delta\zeta$  measurement gives normal load  $F_z$ .  $F_z$  would deform the particle with a contact area  $A = \pi a^2$  and indentation depth  $\delta$  using the Johnson-Kendall-Roberts (JKR) model [19] such that

$$\begin{aligned}a^3 &= R/K(F_z + 3\pi R\Delta\gamma + \sqrt{6\pi R\Delta\gamma F_z + (3\pi R\Delta\gamma)^2}) \\ \delta &= a^2/R - 2/3\sqrt{3\pi\Delta\gamma a/K}.\end{aligned}\quad (9)$$

Here,  $1/K = (3/4)[(1 - \nu_p^2)/E_p + (1 - \nu_s^2)/E_s]$  is the reduced elastic modulus for the particle-substrate system,  $E_p$

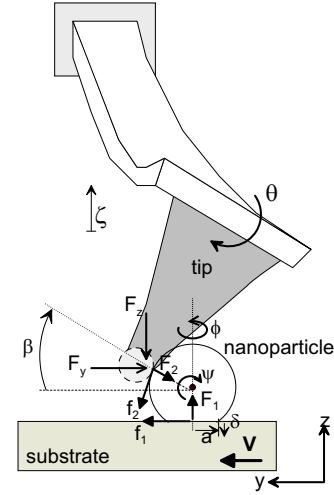


Fig. 3. Nano-tribological measurement technique by controlled AFM probe tip pushing of the micro/nano-object using 2-D force sensing for direct friction measurement.

and  $E_s$  are the Young moduli, and  $\nu_p$  and  $\nu_s$  are the Poisson's coefficients of the particle and substrate, respectively,  $R$  is the particle radius, and  $\Delta\gamma = \gamma_p + \gamma_s - \gamma_{ps} \approx 2\sqrt{\gamma_p\gamma_s}$  [20],  $\gamma_p$ ,  $\gamma_s$ , and  $\gamma_{ps}$  are the particle, substrate, and particle-substrate interface surface energies, respectively.

Measuring  $\Delta\theta$  and  $\Delta\zeta$  and computing  $F_y$  and  $F_z$ ,  $f_1$ ,  $A$  and  $\delta$  are determined. During moving the substrate (or the base of the beam) with a constant speed  $V$ , sliding or stick-slip motion is possible. Assuming only sliding would be considered with a high  $V$ , sliding starts if

$$f_1^* = F_y^* = \tau^s A \quad (10)$$

Thus, the static shear strength  $\tau^s$  could be measured from  $F_y^*$ . In the kinetic friction region,  $f_1 = \tau A$ , and by measuring  $f_1$  and  $F_z$  at this region,  $\tau$  could be also predicted.

## B. Method II

A micro/nano-particle can be pushed on the substrate using a sharp AFM probe tip with a constant speed as in Figure 1. This could be the most *general* method for any material with any size and geometry for any mode of motion (sliding, rolling, rotation, etc.). Depending on the AFM probe deflection measurement technique, this method can be applied in two ways.

1) *2-D Force Sensing AFM Probe*: In this method, the particle is pushed in the  $y$ -direction as shown in Figure 3, and  $F_y$  and  $F_z$  are measured simultaneously from the  $\Delta\theta$  and  $\Delta\zeta$  deflection data.

In this case, beam deflection equations are same as Eq.(6). Quasi-static equilibrium equations are:

$$\begin{aligned}f_1 &= F_y = F_z \cos\beta - f_2 \sin\beta \\ f_2 &= F_z \cos\beta - F_y \sin\beta \\ F_z &= F_1\end{aligned}\quad (11)$$

$F_z$  deforms the particle as in Eq. (9). As possible modes of motion:

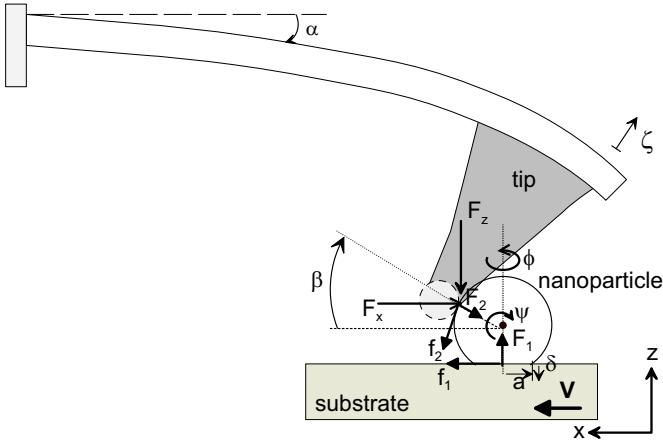


Fig. 4. Nano-tribological measurement technique by controlled AFM probe tip pushing using 1-D force sensing for indirect measurement.

- *Sticking*: If  $F_y = f_1 < \tau^s A$  and  $f_2 < \tau^s A_2$ , the particle would stick to the substrate and tip.
- *Sliding*: If  $F_y = f_1 \geq \tau^s A$  and  $f_2 < \tau^s A_2$ , the particle would slide while stuck to the tip.
- *Rolling*: If  $(f_1 - f_2)R \geq f_1^{\psi}$  and  $f_2 \geq \tau^s A_2$ , i.e.  $f_2 = \tau A_2$ , the particle would start to roll while sliding from the tip. The same equality can be written as

$$[F_z \cos \beta - (1 + \sin \beta) F_y] R \geq \tau_{\psi} A. \quad (12)$$

- *Rotation (Spinning)*: If there is an offset of  $x_0$  along the  $x$ -axis, a spinning could occur along the  $z$ -axis when  $F_y x_0 \geq \tau_{\phi} A$  where  $\tau_{\phi}$  is the rotational friction coefficient.

2) *1-D Force Sensing AFM Probe*: Using 1-D optical or piezoresistive deflection data, only  $\Delta \zeta$  is measured as shown in Figure 4. This kind of simple setup would indirectly measure the frictional force  $F_x$  by pushing the particle in the  $x$ -direction.

The normal deflection of the probe is given by

$$\Delta \zeta = \frac{F_z \cos \alpha - F_x \sin \alpha}{k_z} - \frac{F_z \sin \alpha + F_x \cos \alpha}{k_{xz}} \quad (13)$$

Here,  $\Delta \zeta$  is the only measured parameter which depends both on  $F_x$  and  $F_z$ . Assuming  $F_z$  is relatively very small with respect to  $F_x$  by setting  $\beta \approx 0$ , and the bending due to  $F_x$  is maximized by selecting  $\alpha$  tilt angle large and an AFM probe with a large tip height and short probe length,  $F_x$  can be extracted from the deflection data.

2-D force sensing case sticking, sliding, rolling, and rotation behaviors are the same in this method by just replacing  $F_y = F_x$ .

### C. Automatic Pushing Scheme

This tribological characterization system is supposed to operate automatically for future industrial applications. The aim is to scatter the micro/nano-objects to be characterized on a substrate in a semi-fixed way, and then automatically (or by a user-defined interface) find a single object, and push and record the pushing force data. The data is then analyzed using

the given models. The basic pushing control steps [21] for a micro/nano-object is as follows:

- 1) Scan the substrate with semi-fixed micro/nano-objects on it, and get the 3-D tapping mode AFM image.
- 2) Detect a separate single micro/nano-object with an expected geometry and size using an unsupervised clustering object segmentation algorithm proposed in [24].
- 3) Position the nano-probe with a pre-determined XYZ distance to the single micro/nano-object.
- 4) Get a small window AFM tapping mode scan over the micro/nano-object again to correct any positional error.
- 5) Automatically detect the peak height along the window, and compute the pushing line passing through the object center.
- 6) Move the XYZ positioner along the pushing-line in 1-D with constant height and speed  $V$ , push the object for a defined distance, and record the pushing force data.

## IV. EXPERIMENTS

Using a custom-made AFM system [25] with a piezoresistive AFM nano-probe (ThermoMicroscopes Inc., non-contact piezolever), gold-coated latex particles (JEOL Inc.) are pushed on a silicon substrate using Method II with 1-D force sensing approach and automatic pushing control with a user interface [4]. Experiments are realized in laboratory environment with 23 °C temperature and 50 – 60% relative humidity. Gold-coated latex particles with 500 nm radii are pushed with the probe automatically, and their top-view high-resolution ( $\times 5000$  magnification and 90 nm/pixel) optical microscope (Olympus Inc.) images are used for calculating their positions before and after the pushing operation. A closed-loop piezoelectric XYZ positioner with 15-20 nm accuracy enables the constant speed and height trajectory along the pushing line. The speed of the stage is set to 1.6  $\mu\text{m/s}$  for all experiments. Cantilever deflection is measured through a Wheatstone bridge which gives the voltage difference due to the resistance change of the cantilever by the applied tip forces. Calibrating the normal bending sensitivity of the probe  $S_z$  as 34.4 nm/V using a hard silicon surface,  $\Delta \zeta$  is computed at each instant.

Piezoresistive nano-probe parameters are given as: the tip radius of  $R_t \approx 30$  nm, the length of  $l = 155$   $\mu\text{m}$ , the width of  $w = 50$   $\mu\text{m}$ , the thickness of  $t = 3$   $\mu\text{m}$ , the tip height of  $H = 3$   $\mu\text{m}$ ,  $L_s = l/H = 51.7$ , normal bending stiffness of  $k_z = 8$  N/m, and the measured resonant frequency of 135.3 KHz. Using these parameter values,  $k_{xz} = 276$  N/m,  $k_y^{\theta} = 11225$  N/m, and  $k_y^b = 2222$  N/m are computed. Moreover,  $\alpha = 15^\circ$  and  $\beta \approx 0^\circ$  are taken. Measured  $\Delta \zeta$  corresponds to

$$\Delta \zeta = 0.12 F_z - 0.036 F_x. \quad (14)$$

For calculating the contact area,  $\Delta \gamma = 0.248$  J/m<sup>2</sup> is computed for  $\text{SiO}_2$  substrate (few nanometers thick natural  $\text{SiO}_2$  layer exists on silicon substrate in ambient conditions) and the gold layer of the particle with a possible water layer in between. Here,  $\gamma_{\text{SiO}_2} = 160$  mJ/m<sup>2</sup> [26],  $\gamma_{\text{Au}} = 1.5$  J/m<sup>2</sup> and  $\gamma_{\text{H}_2\text{O}} = 73$  mJ/m<sup>2</sup> are taken. Since  $\beta \approx 0^\circ$ ,  $F_z = f_2$  and  $A_2$  is the tip-particle contact area due to the contact load  $F_x$ .

For no contact load case ( $F_x = 0$ ),  $A_2 \approx 1.5 \times 10^{-16} \text{ m}^2$  using Eq. (9) with  $E_p = 3.8 \text{ GPa}$ ,  $E_{SiO_2} = 73 \text{ GPa}$ ,  $\nu_p = 0.4$ ,  $\nu_{SiO_2} = 0.17$ , and  $R = 500 \text{ nm}$ . On the other hand, the particle-substrate contact area  $A$  is determined by  $F_z$ , and  $A = 109 \times 10^{-16} \text{ m}^2$  for  $F_z = 0$ . Then, from Eq. (14),  $F_x \approx -30\Delta\zeta$ . Using  $S_z$ ,  $\Delta\zeta$  voltage data is transformed to  $nN$  frictional forces by

$$f_1 = -1032\Delta\zeta(V) \text{ nN} . \quad (15)$$

During the pushing experiments, the following modes of motion are observed:

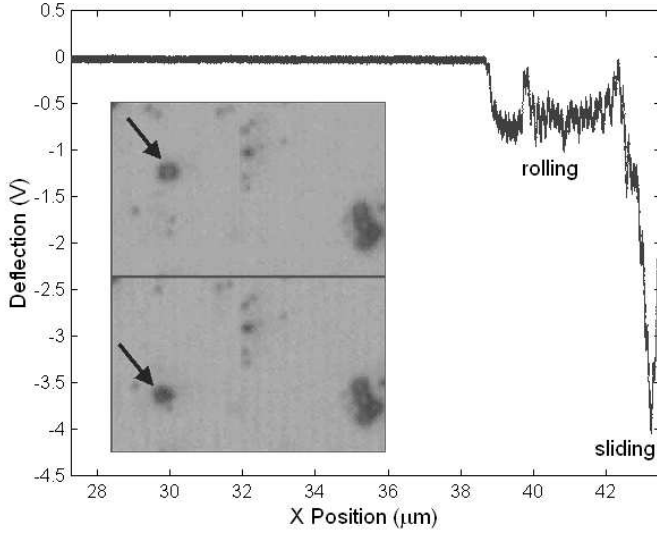


Fig. 5. Initially rolling and then sliding behaviors for a  $1 \mu\text{m}$  gold-coated latex particle pushing case where the particle is pushed around  $4 \mu\text{m}$ : Inner images are the before (upper image) and after (lower image) the pushing operation top-view optical microscope images, and the arrow indicates the pushed particle (deflection (V)  $\times 1032 =$  nanoforce (nN) in the y-axis).

- **Sliding:** In Figure 5, it can be seen that the particle is pushed on the substrate for around  $4 \mu\text{m}$  distance. At first, the particle starts to roll, and after some distance it started to slide with a large peak. The large peak corresponds to the static sliding friction, and after this peak, kinetic friction is observed with a smaller friction. From the peak, around  $4.04 \text{ V}$  is measured. Using Eq. (15),  $f_1^* = 4.17 \mu\text{N}$  is computed. Since  $F_z \approx 0$ ,  $A \approx 109 \times 10^{-16} \text{ m}^2$  gives the static shear strength as  $\tau^s = 382 \text{ MPa}$ . In the kinetic friction region,  $f_1 = 2.58 \mu\text{N}$ ,  $\tau = 237 \text{ MPa}$  is held. Using the theoretical shear modulus and Poisson's ratio values for the  $SiO_2$  and gold interfaces,  $\tau \approx G/29 \approx 311 \text{ MPa}$  is computed using  $G = [(2 - \nu_{Au})/G_{Au} + (2 - \nu_{SiO_2})/G_{SiO_2}]^{-1} = 9 \text{ GPa}$  for  $G_{SiO_2} = 31.4 \text{ GPa}$ ,  $G_{Au} = 30 \text{ GPa}$ , and  $\nu_{Au} = 0.42$ . For a thick water layer case between the particle and substrate,  $\tau = 144 \text{ MPa}$  [27]. Thus, measured shear strength values are close to the theoretical values.
- **Rolling:** Rolling behavior could be observed in Figure 5, 6, and 7 by the periodic oscillation behavior in the force deflection data. After an initial static frictional phase, cantilever deflects almost with a periodic motion of  $500$

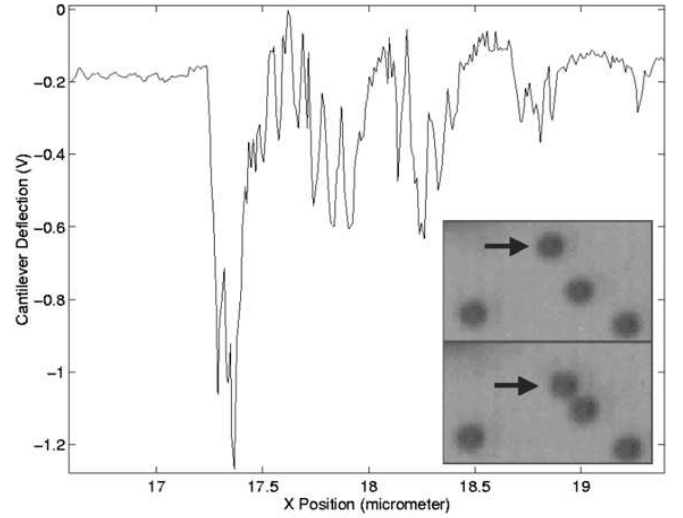


Fig. 6. Rolling case where the particle is pushed around  $1 \mu\text{m}$  as shown in two high-resolution optical microscope images (before and after pushing images).

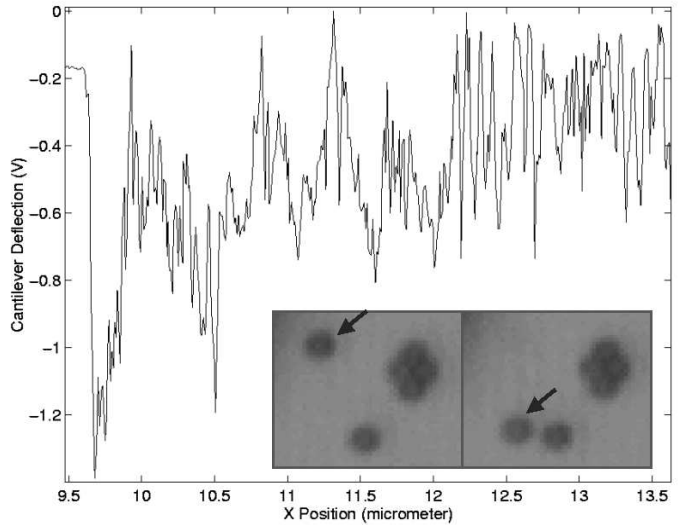


Fig. 7. Rolling case where the particle is pushed around  $3 \mu\text{m}$ .

$\text{nm}$  intervals which is the radius of the particle. In this region,  $\Delta\zeta = 0.7 \text{ V}$  is approximated in average, and this corresponds to  $f_1^\psi = 722 \text{ nN}$  frictional force. Using  $A \approx 109 \times 10^{-16} \text{ m}^2$ ,  $\tau_\psi = f_1^\psi R/A = 33 \text{ N/m}$  rolling friction coefficient is predicted.

- **Rotation:** Rotation of the particle occurred when there is a significant  $x_0$  offset during pushing. In Figure 8, optical microscope image shows a  $90^\circ$  particle rotation around the  $z$ -axis taking the small black dot attached on the particle as the reference. From the deflection data, after the second peak, the tip loses contact with the particle after around  $500 \text{ nm}$  displacement. The rotational friction is predicted from the second peak as  $f_1 = 155 \text{ nN}$  which gives  $\tau_\phi = f_1 x_0/A \approx 4 \text{ N/m}$  rotational friction coefficient with  $x_0 \approx 300 \text{ nm}$ .



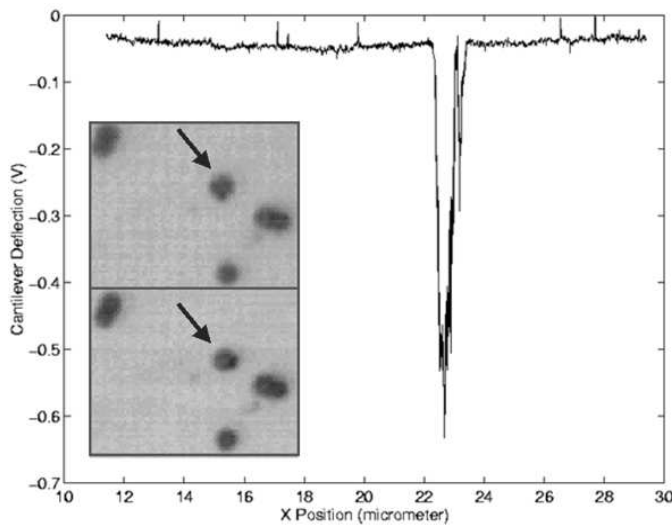


Fig. 8. Rotation case where the particle is rotated around  $90^\circ$  around the vertical  $z$ -axis (a small black dot on the pushed particle in the inner optical microscope images and no positional change of the particle are the proofs to show the approximate  $90^\circ$  spinning behavior).

## V. CONCLUSION

In this paper, using a piezoresistive AFM nano-probe as a nano-manipulator, novel techniques for tribological characterization of nano-scale object-substrate interfaces are proposed. By pushing micro/nano-objects or attaching them on the nano-probe, frictional forces can be directly or indirectly estimated, and sliding, spinning, or rolling frictional parameters can be estimated in any environment. Gold-coated latex particles with  $500\text{ nm}$  radius are pushed on a silicon substrate in the experiments. Preliminary results show that pushing operation can result in sliding, rolling, or rotation behavior depending on the particle-substrate frictional properties, and sliding and rolling frictional coefficients are predicted for gold-silicon interfaces. From the pushing data of the  $500\text{ nm}$  radius gold-coated latex particles on a silicon substrate with a natural silicon oxide layer, static, kinetic, rolling, and spinning shear stresses are estimated as  $382\text{ MPa}$ ,  $237\text{ MPa}$ ,  $33\text{ N/m}$ , and  $4\text{ N/m}$ , respectively. Thus, if there is an offset  $x_0$  from the pushing direction and particle center, the particle would start to spin firstly, and the tip and particle would lose contact after a very short time. In order to prevent this,  $x_0 = 0$  should be precisely controlled. Besides of spinning, rolling or sliding starts first depending on the particle radius,  $\tau^s$ , and  $\tau_\psi$ . For measured  $\tau^s$  and  $\tau_\psi$  values, if the particle radius is smaller than around  $86\text{ nm}$  ( $R < \tau_\psi/\tau^s$  assuming  $f_2 \approx 0$ ), the particle would start to slide first and then roll. However, since  $R = 500\text{ nm}$  in the experiments, particles first started to roll and then slide. On the other hand, sliding and rolling could be observed simultaneously if the friction is higher than the static sliding and rolling frictions.

These methods could be directly applied for other nano-materials, e.g. carbon nano-tubes, nano-crystals, DNA, nano-wires, etc., for their frictional characterization on various substrates with different pushing speeds and environmental conditions. However, modeling of the contact and pushing me-

chanics for these various geometries is needed to be improved. Moreover, using an optical detection system, torsional twisting would be measured, and Method I and II with 2-D force sensing techniques would be also implemented. Future experiments would be also conducted in a vacuum chamber to measure the dry frictional properties at the nano-scale. Besides of characterization applications, these measurements would open new controlled means of understanding nano-scale friction by observing the effects of lattice mismatch between the nano-scale object and substrate, surface roughness, temperature, speed, humidity, contact mechanics, chemical interactions, etc.

## REFERENCES

- [1] D. Schafer, R. Reifengerger, A. Patil, and R. Andres, "Fabrication of two-dimensional arrays of nanometer-size clusters with the Atomic Force Microscope," *Appl. Physics Letters*, vol. 66, pp. 1012–1014, Feb. 1995.
- [2] T. Junno, K. Deppert, L. Montelius, and L. Samuelson, "Controlled manipulation of nanoparticles with an Atomic Force Microscopy," *Appl. Physics Letters*, vol. 66, pp. 3627–3629, June 1995.
- [3] R. W. Stark, S. Thalhammer, J. Wienberg, and W.M. Heckl, "The AFM as a tool for chromosomal dissection - the influence of physical parameters," *Appl. Phys. A*, vol. 66, pp. 579–584, 1998.
- [4] M. Sitti and H. Hashimoto, "Controlled pushing of nanoparticles: Modeling and experiments," *IEEE/ASME Trans. on Mechatronics*, vol. 5, no. 2, pp. 199–211, June 2000.
- [5] M. Sitti and H. Hashimoto, "Tele-nanorobotics using Atomic Force Microscope as a robot and sensor," *Advanced Robotics*, vol. 13, no. 4, pp. 417–436, 1999.
- [6] M. Sitti, B. Aruk, K. Shintani, and H. Hashimoto, "Scaled teleoperation system for nanoscale interaction and manipulation," *Advanced Robotics*, vol. 17, no. 3, pp. 275–291, 2003.
- [7] M. Falvo, R.M. Taylor II, A. Helsen, V. Chi, F. P. Brooks, S. Washburn, and R. Superfine, "Nanometer-scale rolling and sliding of carbon nanotubes," *Nature*, vol. 397, pp. 236–238, 21 Jan. 1999.
- [8] M. Falvo, J. Steele, R.M. Taylor II, and R. Superfine, "Gearlike rolling motion mediated by commensurate contact: Carbon nanotubes on HOPG," *Phys. Review B*, vol. 62, pp. R10665–667, 15 Oct. 2000.
- [9] M. Sitti and H. Hashimoto, "Pushing micro/nanoscale particles on substrates using Atomic Force Microscope probe towards tribological characterization of particle-substrate interfaces," *STM'99 Conference*, pp. 385–386, Korea, July 1999.
- [10] E. Meyer, R. Overney, L. Howald, R. Luthi, J. Frammer, and H.J. Guntherodt, "Friction and wear of Langmuir-Blodgett films observed by Frictional Force Microscopy," *Physical Review Letters*, vol. 69, pp. 1777–80, 1992.
- [11] G. Dedkov, "Experimental and theoretical aspects of the modern nanotribology," *Phys. Stat. Sol.*, vol. 179, no. 3, pp. 3–35, 2000.
- [12] M. Falvo and R. Superfine, "Mechanics and friction at the nanometer scale," *J. of Nanoparticle Research*, vol. 2, pp. 237–248, 2000.
- [13] B. Mailhot, K. Komvopoulos, B. Ward, Y. Tian, and G. Somorjai, "Mechanical and friction properties of thermoplastic polyurethanes determined by Scanning Force Microscopy," *J. of Applied Physics*, vol. 89, pp. 5712–5719, 15 May 2001.
- [14] B. Bhushan, *Handbook of micro/nano tribology*. CRC Press, 2nd ed., 1999.
- [15] T. Bouhacina, B. Desbat, and J. Aime, "FTIR spectroscopy and nanotribological comparative studies: Influence of the absorbed water layers on the tribological behavior," *Tribology Letters*, vol. 9, pp. 11–117, 2000.
- [16] L. Heim, J. Blum, M. Preuss, and H. Butt, "Adhesion and friction forces between spherical micrometer-sized particles," *Phys. Rev. Lett.*, vol. 83, pp. 3328–3331, 18 Oct. 1999.
- [17] N.V. Brilliantov and T. Poschel, "Rolling as a continuing collision," *Eur. Phys. J. B*, vol. 12, pp. 299–301, 1999.
- [18] H. Butt, "A technique for measuring the force between a colloidal particle in water and a bubble," *Journal of Colloid and Interface Science*, vol. 166, pp. 109–117, 1994.
- [19] M. Sitti and H. Hashimoto, "Tele-touch feedback of surfaces at the nanoscale: Modeling and experiments," *IEEE/ASME Trans. on Mechatronics*, vol. 8, no. 2, June 2003 (to appear).
- [20] J. Israelachvili, *Intermolecular and Surface Forces*, 2nd Ed., Academic Press, London, 1992.

- [21] M. Sitti and H. Hashimoto, "Two-dimensional fine particle positioning under optical microscope using a piezoresistive cantilever as a manipulator," *Journal of Micromechatronics*, vol. 1, no. 1, pp. 25–48, 2000.
- [22] G. Dedkov, "Friction on the nanoscale: New physical mechanisms," *Materials Letters*, vol. 38, pp. 360–366, 1999.
- [23] E. Meyer, R.M. Overney, K. Dransfeld, and T. Gyalog, *Nanoscience: Friction and Rheology on the Nanometer Scale*, World Scientific Pub., Singapore, 1998.
- [24] M. Sitti, I. Bozma, and A. Denker, "Visual tracking for moving multiple objects: Integration of vision and control," *IEEE Int. Symp. on Industrial Electronics*, pp. 535–540, July 1995.
- [25] M. Sitti, "Teleoperated 2-D micro/nanomanipulation using Atomic Force Microscope," *Ph.D. Thesis*, Dept. of Electrical Engineering, University of Tokyo, Tokyo, Sept. 1999.
- [26] K. Autumn, M. Sitti, Y. A. Liang, A. Peattie, W. Hansen, S. Sponberg, T. Kenny, R. Fearing, J. Israelachvili, and R. J. Full, "Evidence for van der Waals adhesion in gecko setae", *Proc. of the National Academy of Science*, vol. 99, no. 19, pp. 12252–12256, 17 September 2002.
- [27] C. M. Mate, "Force Microscopy studies of the molecular origin of friction and lubrication," *IBM J. Res. Develop.*, vol. 39, pp. 617–627, Nov. 1995.



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