

# Study of Nano-tribology using Scanning Force Microscope

Tsunetaka Sumomogi, Toshiro Endo and Shinichi Matsuo

Faculty of Engineering, Hiroshima Kokusai Gakuin University

6-20-1 Nakano, Aki-ku, Hiroshima 739-0321, Japan

## Introduction

The scanning force microscope (SFM) is a unique tool for nanometer-scale modification [1,2] and tribology [3]. When a tip scans on a sample surface in contact mode, there is not only a normal force but also a lateral force acting on the tip. Stick-slip motion with the periodicity of the lattice constant was detected between a tungsten tip and a mica surface. [4]. In ambient air, the presence of physisorbed or chemisorbed molecules could affect the nanotribological properties between the tip and the sample. The effects should be significant for high-resolution operation [2]. The influence of adsorbed water molecules and the capillary condensation on nanotribological phenomena has been characterized by means of SFM friction experiments on silicon wafers [5]. It was reported that friction and adhesive forces were affected by the relative humidity of ambient air, but the behavior of these forces in atomic scale imaging was not mentioned.

In the present experiment, the SFM is placed in an atmosphere control apparatus and the relative humidity around the tip and the sample is controlled at 30% or 70%. The lateral force images corresponding to atomic scale topography images are obtained on the cleaved surface of mica. The nanotribological effects of adsorbed water molecules and the capillary condensation of water is examined.

## Experiment

The schematic of the main part of the SFM used for the present study is shown in Fig. 1. The lateral component  $F_L$  and the normal component  $F_N$  of the force acting on the tip are independently measured by twisting angle and bending inclination of the cantilever, respectively. We use a triangular microcantilever with 25 nm tip-radius, which is made of  $\text{Si}_3\text{N}_4$ .

Its spring constants of twisting and bending are 250 N/m and 0.58 N/m, respectively. The SFM unit is placed into a glove box connected to a precise air control apparatus and the atmosphere around the tip and the sample is controlled to a relative humidity (RH) of 30% or 70%, and a temperature of 30\_ [6]. The normal

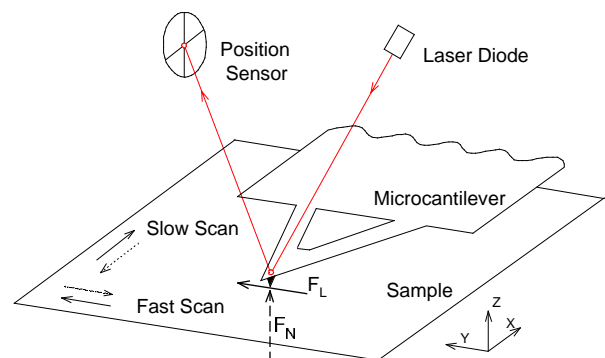


Fig. 1. Schematic of the main part of the SFM.

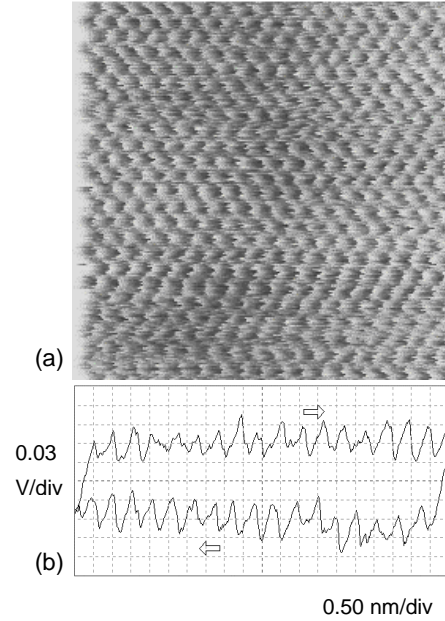
force is 0-200 nN; here the zero force is defined as the force when the tip jumps up from the sample surface during retraction of the tip. With raster scanning of 5-10 nm width under the constant normal force, the topography image and the lateral force image are obtained simultaneously. The fast scan rate is 30 Hz or 60 Hz, so the scan speed is 0.6  $\mu\text{m/s}$  or 1.2  $\mu\text{m/s}$  when the scan width is 10 nm.

## Results and discussion

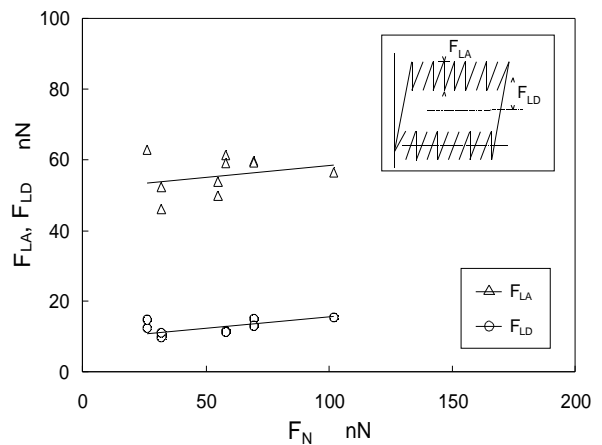
Figure 2 shows an image and a signal profile of the lateral force for mica in 30% RH, with a normal force of 40 nN. The image indicates the periodicity of lattice structure at  $0.52 \pm 0.03$  nm. The lattice constant and the symmetry of the image correspond to the mica (0001) face [4]. The lateral force signal profile indicates hysteresis according to the direction change of the lateral force in trace and retrace and, the occurrence of “stick-slip” processes due to spatially discrete adhesion and jumps with the lattice periodicity.

The amplitude of periodic change  $F_{LA}$  and the average value  $F_{LD}$  of the lateral force are evaluated from the lateral force signal profile, such as that shown in Fig. 2(b). Figure 3 shows  $F_{LA}$  and  $F_{LD}$  on lateral force signal profiles for mica in 30% RH as a function of normal load  $F_N$ .  $F_{LA}$  directly contributes to the contrast of the atomically resolved lateral force images. While  $F_{LA}$  is slightly dependent on  $F_N$ ,  $F_{LD}$  is almost constant with the change of  $F_N$ .

In order to compare the lateral force behaviors under 30% RH and 70% RH,  $F_{LA}$  on the lateral force signal profiles in these conditions is shown in fig. 4 as a function of  $F_N$ . The straight lines are obtained by a least-squares method. It is recognized that the atmosphere around the tip and the sample affects the dependence of the lateral force magnitude on the normal load. At low relative humidity (30%), the lateral force magnitude in atomically resolved images is almost



**Fig. 2.** An image and a lateral force signal profile for mica at 30% RH.



**Fig. 3.**  $F_{LA}$  and  $F_{LD}$  on lateral force signal profiles for mica at 30% RH as a function of  $F_N$ .

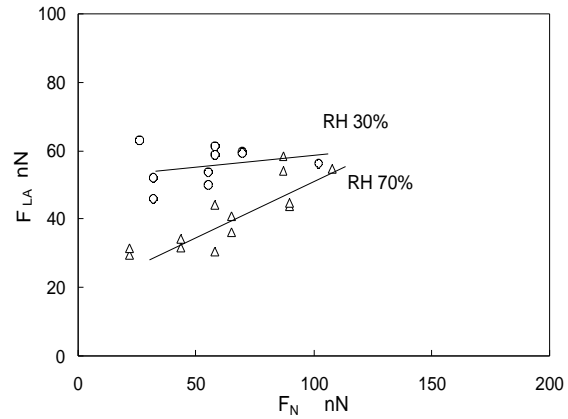
independent of the normal load. We can conclude that the adhesive force, independent of the normal force, is superior to the friction force, dependent on the normal force. At high relative humidity (70%), the lateral force magnitude is smaller than at 30% RH and exhibits a dependence on the normal load. We can conclude that the adhesive force is weaker than at 30% RH.

The relative humidity of ambient air in this experiment, 30-70%, is high enough to cause water molecules and films to be adsorbed on mica surfaces [7]. Therefore, it is necessary to consider that the adhesive force due to the presence of the

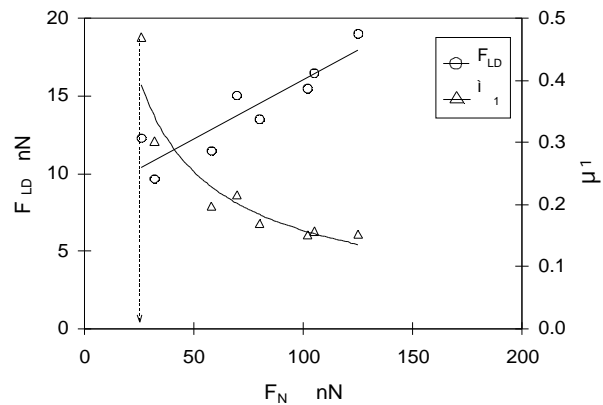
water in the tip-sample gap has a large influence upon the lateral force [8]. The adhesive force acting on the tip is considered to consist of the meniscus force and the interaction forces between the tip and the sample, where water is present in the gaps around the contacting asperities. We discuss the dependence of the adhesive force magnitude on the relative humidity of ambient air.

The adhesive force  $F_{ad}$  due to the meniscus between a spherically shaped tip and a flat surface is expressed as  $F_{ad} = Rh\gamma/r$  ( $h \ll R$ ), where  $R$  is the tip radius,  $h$  is the meniscus height,  $\gamma$  is the surface tension of water, and  $r$  is the radius of curvature of the meniscus [9]. Here, the effect of the vapor pressure on the meniscus radius is expressed with the Kelvin equation,  $\ln(p/p_0) = -2\gamma/r$ , where  $p_0$  is the nominal vapor pressure,  $p$  is the observed vapor pressure, and  $r$  is negative for capillaries [10]. From the above considerations, it is conjectured that the absolute value of the meniscus radius increases, and accordingly the adhesive force due to meniscus decreases, with increasing relative humidity.

We also consider the evaluation method of friction coefficient in order to clear the relationship between  $F_N$  and  $F_{LD}$ . Since the lateral force signal profiles are looked as “stick-slip” process, the kinetic friction coefficient can be given by the magnitude of  $F_{LD}$ . The lateral force  $F_L$  is assumed to consist of the friction force due to the normal force  $F_N$



**Fig. 4.**  $F_{LA}$  on lateral force signal profiles for mica at 30% RH and 70% RH as a function of  $F_N$ .



**Fig. 5.**  $F_{LD}$  and the apparent friction coefficient  $\mu_1$  for mica at 30% RH as a function of  $F_N$ .

and the meniscus force  $F_{ad}$  due to the capillary condensation of water [5]. The friction force is the product of the friction coefficient  $\mu_0$  and  $F_N$ , the lateral force is expressed as  $F_L = \mu_0 F_N + F_{ad}$ . The apparent friction coefficient  $\mu_1$  is given by  $\mu_1 = F_L / F_N = \mu_0 + F_{ad} / F_N$ . The value of  $\mu_1$  becomes to be larger than  $\mu_0$  with decreasing  $F_N$ , because the meniscus force is superior to the friction force. In the present examination, we discuss in the sight of the apparent friction coefficient  $\mu_1$ . Figure 5 shows  $F_{LD}$  and the apparent friction coefficient  $\mu_1$  for mica at 30% RH as a function of  $F_N$ . The value of  $\mu_1$  is considerably large in low  $F_N$  near to the adhesive force ( $F_{ad}=32\text{nN}$ ) and becomes to be 0.1-0.2 in high  $F_N$ . It is obvious that the adhesive force due to adsorbed water molecules and the capillary condensation of water has a great influence upon the lateral force properties in low-load measurements.

## Conclusion

In order to discuss the nanotribological effects of adsorbed water molecules and the capillary condensation of water, the lateral force images are examined quantitatively as a function of the normal load on the sample surface. The dependence of lateral force magnitude on the normal load is found to be affected by the atmosphere around the tip and the sample. The adhesive force due to the presence of water in the tip-sample gap has a large influence upon the lateral force properties in low-load measurements.

## References

1. T. A. Jung, A. Moser, H. J. Hug, D. Brodbeck, R. Hofer, H. R. Hidber, and U. D. Schwarz, *Ultramicroscopy* **42-44**, 1446 (1992).
2. T. Sumomogi, T. Endo, K. Kuwahara, and R. Kaneko, *J. Vac. Sci. Technol.* **B13**, 1257 (1995).
3. R. Kaneko and E. Hamada, *Wear* **162-164**, 370 (1993).
4. R. Erlandsson, G. Hadziioannou, C. M. Mate, G. M. McClelland, and S. Chiang, *J. Chem. Phys.* **89**, 5190 (1988).
5. M. Binggeli and C.M. Mate, *J. Vac. Sci. Technol.* **B13**, 1312 (1995).
6. T. Sumomogi, K. Hieda, T. Endo, and K. Kuwahara, *Appl. Phys.* **A 66**, S299 (1998)
7. H. K. Christenson, *J. Colloid Interface Sci.* **121**, 170 (1988).
8. D. A. Bonnell ed., *Scanning Tunneling Microscopy and Spectroscopy* (VCH Publishers, New York 1993), p.239.
9. J. N. Israelachvili, *Intermolecular and Surface Forces*, 2nd edn. (Academic Press, London 1985), p.330.
10. Y. Inoue, *J. Jpn. Soc. Tribologists (in Japanese)* **36**, 296 (1991).