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Nanomechanical characteristics at an ultra-small particle-surface contact interface †

In-Ha Sung[1,*], Hung-Gu Han[2] and Hosung Kong[2]

[1]*Department of Mechanical Engineering, Hannam University, Daejeon, 306-791, Korea*
[2]*Energy Mechanics Research Center, Korea Institute of Science and Technology, Seoul, 136-791, Korea*

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Abstract

Atomic force microscopy (AFM) measurements have shown that nanoscale interfaces in sliding contact frequently exhibit atomic lattice stick-slip friction. Using various material surfaces and AFM tips, including colloidal probes, and systematically varying applied load and lever stiffness, it is demonstrated that transitions can be repeatedly observed from smooth sliding to single unit-cell slips and then multiple slips. The behavior is dependent on the interplay between the stiffness of the contact zone, the measurement system (i.e., the AFM cantilever), and the interfacial potential. Atomic lattice stick-slip occurs with colloidal particle tip orders of magnitudes larger than those previously used. Stable atomically corrugated sliding in ambient conditions that cannot be seen elsewhere is reported. The generality of these conditions suggests that atomic-scale stick-slip behavior may be far more prevalent than previously appreciated. In addition, the friction-stiffness maps of various material surfaces in contact with a colloidal particle were reported, and the complex effects of system stiffness and pressure were discussed for chemical-mechanical polishing applications.

Keywords: Atomic force microscopy; Atomic-scale stick-slip friction; Chemical-mechanical polishing (CMP); Contact stiffness

1. Introduction

Interest in ultra-small particles with a size of less than a few micrometer diameters have been tremendously increasing due to their wide applications in various fields such as environmental and medical sciences, as well as engineering [1-3]. As one of their applications, in the chemical-mechanical polishing (CMP) process for silicon wafer planarization, one of the more critical and difficult issues to be solved for performance improvement is the presence of unwanted defects such as scratches, gouging, and corrosion. Much endeavor has been done to understand the machining mechanism from the tribological viewpoint. However, most previous studies on tribology in the CMP process were macro-scale research based on a total CMP system. Understanding nano-scale contact and frictional behavior at particle-surface interfaces is important.

It is generally known that atomistic stick-slip frictional behavior occurs at such ultra-small scale contact interfaces. Great efforts have been made to understand the fundamental mechanisms of stick-slip friction from macro to atomic scale, since stick-slip friction causes system failures and instabilities. By using both simulation and experimental techniques, the effects of various parameters such as scanning direction with respect to lattice structure, contact materials, applied load, and scan speed, as well as theoretical models such as the Tomlinson model have been revealed [4-7]. However, many questions about atomic-scale stick-slip still remain. It is commonly suggested that in a nanometer-sized contact, the tip atoms may be distorted sufficiently by interfacial forces to pull them into at least a partial registry with the sample's lattice. The origin of the behavior, that is, whether the behavior is intrinsic to the interface or it is affected by instrument properties, has not yet been unveiled. The transition in atomic-scale stick-slip friction, which will be discussed in this paper, is also one of the key issues to be examined. However, only a few studies have reported on this [4-7].

Based on the given background, this work aims to understand the frictional behaviors at particle-surface contact interfaces depending on the nanomechanical and material properties of the system under various operating and environmental conditions. In this paper, the effects of system stiffnesses and materials on the atomic-scale frictional behavior at particle-surface contacts will be discussed using several AFM probes with a wide range of lateral cantilever stiffnesses and applied loads.

2. Experimental setup

A commercial AFM (Nanoscope, Digital Instruments Co.) was used. The scanning speed was set to 0.6 μm/s, and all of

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*Corresponding author. Tel.: +82 42 629 8316, Fax.: +82 42 629 8293
E-mail address: isung@hnu.kr

the experiments were performed under ambient atmosphere at room temperature and a humidity level of 35-50% RH.

For the cantilever, in order to simulate particle-surface contact, colloidal probes which had a colloidal particle attached in place of the AFM tip were used. Microspheres of silica and vitreous (glassy) carbon with less 10 μm diameters were used for the colloidal probe. The normal stiffnesses of the colloidal probes were in the range of 0.06-0.09 N/m. Each of these cantilevers had different dimensions and thus different normal and lateral stiffnesses. The surfaces for sliding contact were composed of a highly oriented pyrolitic graphite (HOPG, Grade SPI-1), copper, aluminum, nickel, poly-silicon, and P-TEOS, all of which are currently used in the real CMP process as semiconductor materials.

Lateral force calibration and the measurements of normal and lateral stiffnesses of the cantilevers were performed using established methods [8-10]. Further, the lateral contact stiffness between a cantilever and a material surface was measured by AFM in which the scanner moves laterally within the range where no tip slip-ping occurs. The lateral deflection signal of the cantilever was then detected according to the applied load.

3. Results and discussion

3.1 Stick-slip friction at particle-surface contact

It is generally thought that atomic stick-slip friction can occur only when an ultra-sharp and well-shaped tip slides against an atomically flat surface. In reality, almost all previous studies on the stick-slip and atomic lattice resolution imaging have been performed using sharp tips with a nanometer-sized radius. However, the results presented in Fig. 1 show that such a general assumption is not the case in this work.

Fig. 1 shows the images of the vertical and torsional deflection (friction) of the cantilever (5 x 5 nm^2 size) with respect to the scan direction on an HOPG surface. These were obtained simultaneously using the colloidal probe with a glassy carbon particle that is 7 μm in diameter (normal stiffness~0.057 N/m). The line traces were extracted from each of the images. A longitudinal scan means that each line scan was taken by moving the sample parallel to the long axis of the cantilever, while a perpendicular scan means that each line scan was taken by moving the sample perpendicular to the long axis of the cantilever. The stick-slip frictional behaviors that are varied with the scan direction due to the anisotropic lattice structure of graphite can be shown in the images clearly. Such changes in the shape and periodicity of the stick-slip friction signal correspond quite well to the behaviors that can be predicted from previous studies, which mainly focused on the effects of lattice periodicity and anisotropy [4, 11].

In this contact situation, the contact area estimated simply by using the Hertzian theory and the material properties of HOPG and glassy carbon reported elsewhere [12, 13] is about 1300 nm^2. Consequently, more than 6800 unit cells of graphite are involved in the contact area. Despite the fact that this is obviously affected by the roughness of the particle, the results

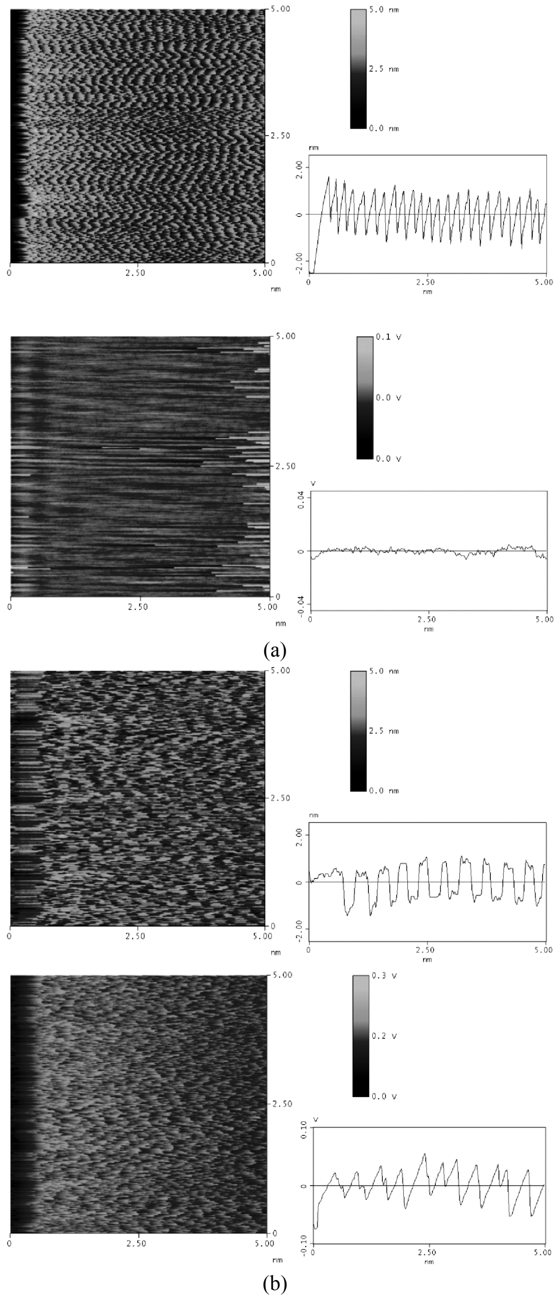
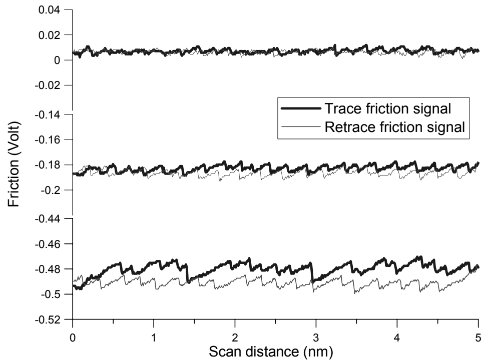


Fig. 1. Stick-slip behaviors with respect to scan direction on an HOPG surface using a colloidal probe with a glassy carbon particle that is 7 μm in diameter. Applied normal load – 56.2 nN. (a) Longitudinal scan, (b) Perpendicular scan. (In each set of images: Upper- vertical deflection images, Lower – torsional deflection (friction) images. The line traces were extracted from each of the images.)

clearly indicate that atomic stick-slip friction can occur even when there is a relatively large contact area, and the tips lack a well-shaped apex. As mentioned above, it was generally thought that atomic stick-slips only occur for nano-scale tips. Therefore, one important conclusion is that atomic stick-slip friction may be a more common phenomenon than is currently thought.

Another important point to note is that double or multiple



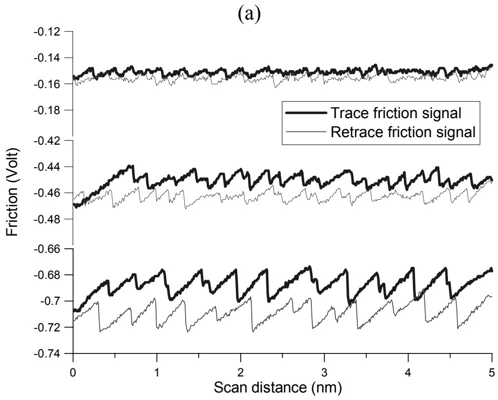


Fig. 2. Friction loops at different loads (low, medium, and high loads) from the contact between a silica colloidal probe and (a) HOPG (Top: -20.2 nN, middle: 108.0 nN, bottom: 278.0 nN), (b) copper (Top: -16.0 nN, middle: 54.5 nN, bottom: 285.2 nN).

slips are shown in the friction image and the line trace in the case of perpendicular scans (Fig. 1(b)), unlike the expected single slip behaviors. In other words, the tip jumps one or several stick points abnormally, and consequently, slips occur for two or more lattice spacings. From the experiments, it was found that even in the case of using the cantilevers which belong to the same type, some of them showed a single slip, some showed multiple slips, and others presented mixed behaviors. Such results could be explained by the lateral stiffnesses of the cantilever, as well as the applied normal load.

3.2 Effects of load and material on the transition in friction

Fig. 2 presents the friction loops at different loads obtained by using a silica colloidal probe on HOPG and copper surfaces. The data shows a clear transition from single slips to multiple slips according to the applied load. Each of the friction loops shows clear smooth sliding, a single slip, and multiple slips.

It is also found that such transition occurs differently with the contact material. It is obviously found that the fluctuation in the friction signal (stick-slip magnitude) of copper is larger than that of graphite under a similar load. In other words, the energy dissipation due to friction (the area formed by both trace and retrace lines) is relatively high in contact with copper. It can be thought that this result was caused by the different stiffnesses of contact between the cantilever tip and the material surface [14].

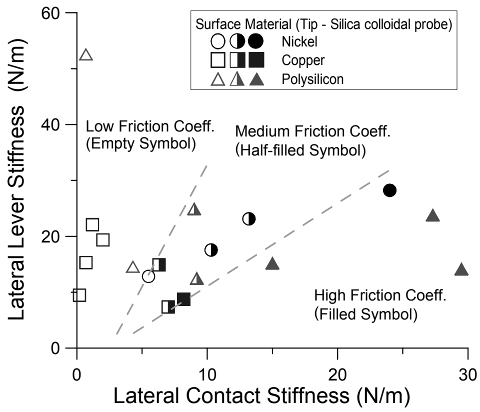


Fig. 3. A friction–stiffness map of various material surfaces in contact with a silica colloidal probe.

The effects of changing the applied load are twofold. On one hand, according to the proposed theoretical models [4-7], increasing the load increases the corrugation of the tip-surface interfacial potential. Therefore, stick-slip is more likely. However, increasing the load can also increase the lateral contact stiffness. This contributes to the system stiffness, and this can make stick-slip less likely. Therefore, in order to observe how the transition shown in Fig. 2 changes according to the stiffnesses of the cantilevers and material contact, a set of experiments considering the lateral stiffnesses of the cantilevers and contact was performed. The result is discussed in the following section.

3.3 Relationship between friction and stiffness

Fig. 3 is a friction-stiffness map of various material surfaces in contact with the silica colloidal probes. As shown in the figure, the friction coefficient could be categorized into three regimes according to its magnitude, that is, low (below 0.2), medium (0.2-0.5), and high (above 0.5) friction coefficient regimes. As a whole, the frictional behaviors of the low, medium, and high friction coefficient regimes showed smooth, single, and multiple slips, respectively.

The results indicate that there is a boundary between each of the friction coefficient regimes. Also, it could be concluded that the transitions in the friction vary largely with the surface material in contact with the colloidal probe, even if the cantilevers have similar lateral stiffness to one another. This is due to the fact that whether or not a single slip or multiple slips occur is a question of the dynamics of the system. According to the theoretical stick-slip models [4-7], multiple jumps can only occur if the cantilever has a relatively low lateral stiffness and relatively large lateral stiffness of the contact, under the condition of insufficient system damping. Therefore, the experimental results are consistent with the prediction from the model. From these results, it can be concluded that in order to have good machine surface integrity, the pressure applied to the particle-surface interface in CMP processes should be properly determined with respect to the material to be machined and the lateral stiffness of the CMP system.

4. Conclusions

An experimental investigation of the atomic/nano-scale frictional behavior at various tip-surface contacts was carried out using atomic force microscope (AFM) cantilevers with different stiffnesses and tips. The experimental results show that stick-slip friction behavior occurs even when the colloidal probes with a particle of a few micrometers in diameter have a relatively large contact area and lack a well-shaped tip end. This indicates that the atomic stick-slip friction may be a more common phenomenon than currently thought.

In addition, systematic transitions from smooth to single to multiple slips in friction at particle-surface contacts were first observed systematically by considering the competition between the stiffness of the interatomic potential across the interface and the elastic stiffnesses of the contacting materials and the cantilever itself. From the observations, a friction map with respect to the lateral contact stiffness was obtained for various semiconductor material surfaces. These are expected to be useful data in applications using ultra-small particles such as CMP processes.

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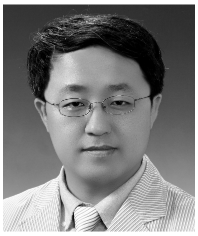
References

[1] G. M. Whitesides and B. Grzybowski, Self-assembly at all scales, *Science*, 295 (2002) 2418-2121.
[2] A. Terray, J. Oakey and D. W. M. Marr, Microfluidic control using colloidal devices, *Science*, 296 (2002) 1841-1844.
[3] J. -M. Park and H-. D. Jeong, A study on the fabrication of micro groove on Si wafer using chemical mechanical machining, *Journal of Mechanical Science and Technology*, 19 (2005) 2096-2104.
[4] S. Morita, S. Fujisawa and Y. Sugawara, Spatially quantized friction with a lattice periodicity, *Surface Science Report*, 23 (1996) 1-41.
[5] K. L. Johnson and J. Woodhouse, Stick-slip motion in the atomic force microscope, *Tribology Letters*, 5 (1998) 155-160.
[6] G. A. Tomlinson, A molecular theory of friction, *Philosophical Magazine*, 7 (1929) 905-939.
[7] S. N. Medyanik, I. -H. Sung, W. K. Liu and R. W. Carpick, Predictions and observations of multiple slip modes in atomic-sale friction, *Physical Review Letters,* 97 (2006) 136106.
[8] J. E. Sader, J. W. M. Chon and P. Mulvaney, Calibration of rectangular atomic force microscope cantilevers. *Review of Scientific Instruments*, 70 (1999) 3967-3969.
[9] J. P. Cleveland, S. Manne, D. Bocek and P. K. Hansma, A nondestructive method for determining the spring constant of cantilevers for scanning force microscopy, *Review of Scientific Instruments*, 64 (1993) 403-405.
[10] D. F. Ogletree, R. W. Carpick and M. Salmeron, Calibration of frictional forces in atomic force microscopy, *Review of Scientific Instruments*, 67 (1996) 3298-3306 .
[11] A. Schirmeisen, L. Jansen and H. Fuchs, Tip-jump statistics of stick-slip friction, *Physical Review B*, 71 (2005) 245403.
[12] J. P. Lu, Elastic properties of carbon nanotubes and nanoropes, *Physical Review Letters*, 79 (1997) 1297-1300.
[13] J. Robertson, Mechanical properties and coordinations of amorphous carbons. *Physical Review Letters*, 68 (1992) 220-223.
[14] R. W. Carpick, D. F. Ogletree and M. Salmeron, Lateral stiffness: a new nanomechanical measurement for the determination of shear strengths with friction force microscopy, *Applied Physics Letters*, 70 (1997) 1548-1550.



In-Ha Sung received his Ph.D. degree in Mechanical Engineering from Yonsei University, Korea, in 2004. He then worked as a Research Associate at the University of Wisconsin-Madison, USA. Currently, he is an Assistant Professor at the Department of Mechanical Engineering at Hannam University in Daejeon, Korea. He serves as an Associate Editor for the Journal of Mechanical Science and Technology. His research interests include tribology, functional surfaces, and micro/nano-fabrication.

Hung-Gu Han received his Ph.D. degree from V. A. Belyi Metal-Polymer Research Institute of the National Academy of Sciences of Belarus in 2000. Currently, he works as a research scientist at the Energy Mechanics Research Center of the Korea Institute of Science and Technology (KIST). His research interests include lubrication, oil analysis, and surface coatings.

Hosung Kong received his Ph.D. degree from Cambridge University, England in 1992. Currently, he works as a principal research scientist at the Energy Mechanics Research Center of KIST. His research interests include friction and wear of materials, machine condition monitoring, and solid lubrication.