Vol 18 No 1, January 2009
1674-1056/2009/18(01)/0231-07

Chinese Physics B

© 2009 Chin. Phys. Soc.
and IOP Publishing Ltd

Effect of micro-dimple patterns on capillary pull-off force and friction force of silicon surface*

Zhang Xiang-Jun(张向军)[a)b)†], Dong Yun-Kai (董云开)[a)], Liu Yong-He(刘永合)[b)], and J. A. Schaefer[b)]

[a)] *State Key Laboratory of Tribology, Tsinghua University, Beijing* 100084, *China*
[b)] *Institute of Physics and Center of Micro/nano Technology, Technich University of Ilmenau, PF* 100565, *Germany*

(Received 19 May 2008; revised manuscript received 13 August 2008)

A microtribometer is used to measure and compare pull-off forces and friction forces exerted on (a) micro-dimpled silicon surfaces, (b) bare silicon surfaces, and (c) octadecyltrichlorosilane (OTS) treated silicon surfaces at different relative humidity (RH) levels separately. It is found that above a critical RH level, the capillary pull-off force increases abruptly and that the micro-dimple textured surface has a lower critical RH value as well as a higher pull-off force value than the other two surfaces. A micro topography parameter, namely sidewall area ratio, is found to play a major role in controlling the capillary pull-off force. Furthermore, micro-dimpled silicon surface is also proved to be not sensitive to variation in RH level, and can realize a stable and decreased friction coefficient compared with un-textured silicon surfaces. The reservoir-like function of micro dimples is considered to weaken or avoid the breakage effect of liquid bridges at different RH levels, thereby maintaining a stable frictional behaviour.

Keywords: micro-dimpled surface, capillary pull-off force, frictional property, relative humidity
PACC: 4630P, 6810

1. Introduction

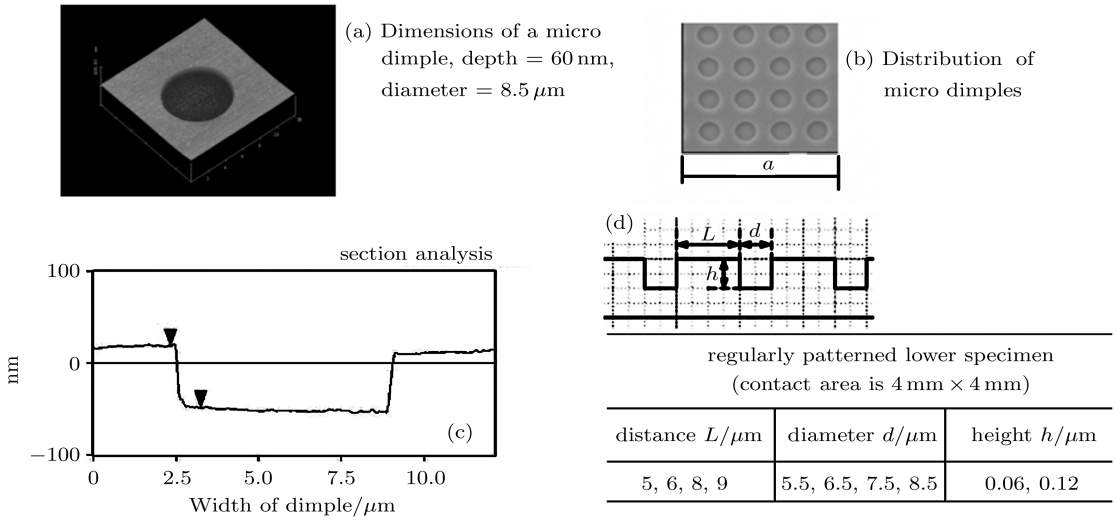
The capillary effect caused by water condensation due to ambient humidity may result in a stiction failure and high friction in hard disk data storage or in the operation of microelectromechanical system (MEMS).[1−4] Stiction occurs when restoring force of micro structure is unable to overcome the interfacial attractive force (named pull-off force), which generally results from the capillary effect of condensed water film due to ambient humidity. Surface texturing proves to be a simplest method to avoid the stiction failure and obtain stable friction property.[5−9] Many theoretical and experimental researches have been performed to investigate the effects of surface texturing or pattern geometry on capillary pull-off force and friction force at different levels of relative humidity (RH). Textured surfaces with micro/nano patterns such as ditches or asperities have been found to be effective to reduce the capillary pull-off force[2] and the friction force.[5−8] The pattern geometry parameters, such as roughness,[3,10,11] depth and width of texturing ditch,[5] as well as aspect ratio[12] are all considered as important design parameters to tailor the capillary pull-off force and frictional behaviours of textured surfaces involved in MEMS and head-disk systems. It has been demonstrated that the stiction possibility due to RH in an MEMS increases as the surface roughness decreases.[2] However, experimental data at micro scale are still scarce to reveal the effects of shape, size, and distribution of micro patterns on capillary pull-off force and frictional property at different RH levels. In this paper, we focus on micro-dimpled surfaces and their capillary effects and frictional properties. Micro-dimpled Si surfaces with various widths, depths and distributions are investigated and compared with smooth un-textured Si surfaces and hydrophobic films deposited on silicon surfaces at different RH levels. We aim to test the possibility of obtaining controllable capillary pull-off forces and stable frictional properties on micro patterned surfaces and applying them as working surfaces in MEMS or micro devices.

*Project supported by the National Natural Science Foundation of China (Grant Nos 50575123 and 50730007), and China Scholarship Council (CSC) and German Research Foundation (DFG).
†Corresponding author. E-mail: xjzhang@tsinghua.edu.cn

2. Experimental details

2.1. Experimental specimen and processing

To make micro/nano patterns on silicon surfaces, different texturing processes can be used[5] including mechanical (polishing, grinding, ultrasonic machining), lithographical or energy beam processes. Within them, surface texturing by using the lithographical process has attracted much interest, due to its high product efficiency, excellent surface finish, and abundant micro scale topographical features. By using this method, regular micro dimples with a variety of dimensions and distributions are etched on a silicon wafer surface (Si(100) surface) as shown in the inset table in Fig.1. The scanning electron microscope (SEM) images of the micro-dimpled specimen are shown in Figs.1(a) and 1(b).



distance L/μm	diameter d/μm	height h/μm
5, 6, 8, 9	5.5, 6.5, 7.5, 8.5	0.06, 0.12

Fig.1. Micro-dimpled silicon surface used as specimen, where panels (a) and (b) show the scanning electron microscope (SEM) images of the micro dimples etched on silicon wafer using lithographical method, panel (c) exhibits the profile of a micro dimple on the Si(100) surface with a surface roughness of less than 1 nm, and panel (d) displays the micro dimples that are different in diameter, depth and interspace.

In our test, the upper specimen is adopted to be a 4 mm×4 mm Si(100) plate with a surface roughness of less than 1 nm. The lower specimen is regularly micro-dimpled silicon surface as shown in Figs.1(a) and 1(b). For comparison, smooth silicon wafers, bare and covered with an octadecyltrichlorosilane (OTS) film are also selected as the lower specimen. The OTS film is deposited on the Si(100) surface that has been immersed into a 10 mmol/L OTS solution for 15 min, and it is used as a kind of hydrophobic surface for investigation. Before each test, the specimens are cleaned in an ultrasonic cleaner using acetone and de-ionized water for 5 min separately. Then they are dried thoroughly by using a heat blower for 2 min.

2.2. Experimental setup and test conditions

A micro tribometer is used in our experiments. Its force sensor is a double-leaf cantilever, and it is schematically shown in Fig.2 (left). The upper specimen is a 4 mm × 4 mm flat surface fixed on the end of the cantilever. The lower substrate is also a flat surface, which is kept parallel to the upper specimen by a specially designed soft spring on the back of the upper specimen holder as shown in the right insert in Fig.2. The force sensor has been calibrated by a laser fibre sensor and has a good linear relationship between its deformation and the applied force. The cantilever is driven by a stepping motor to make the upper specimen move against the lower specimen (which is fixed on the substrate). The end deflection of the cantilever during the relative movement (vertically approaching/detaching or laterally sliding) is measured by the strain gauges glued on it, and then converted into the corresponding normal force or lateral force by using the calibrated spring constants. The force resolution is 7 μN in the normal direction, and 30 μN in the lateral direction.

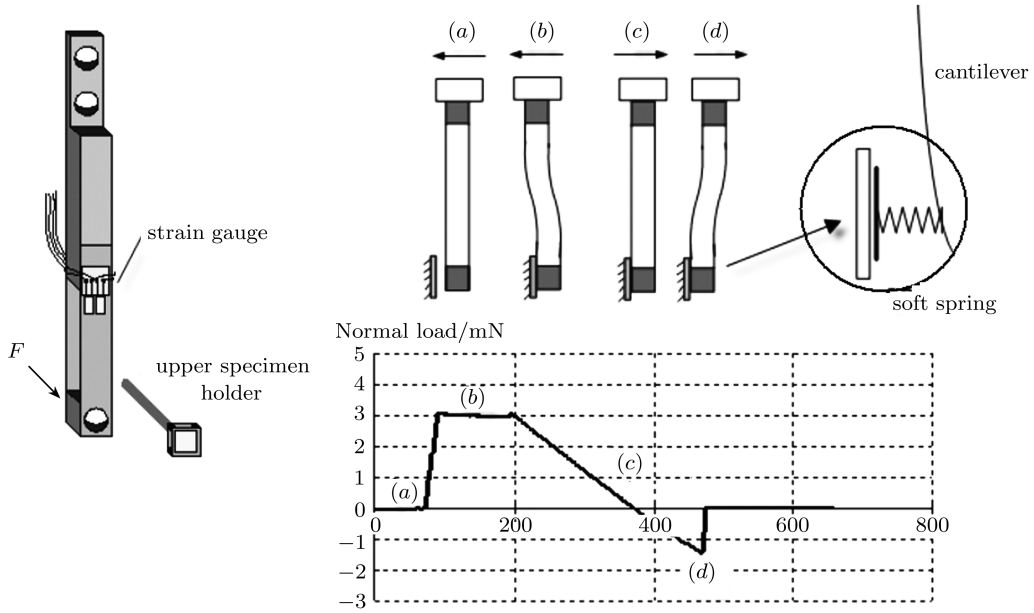


Fig.2. Force sensor and typical force curve of the micro tribometer setup. A double-leaf cantilever is used as the force sensor, with a holder of upper specimen fixed at its end.

A typical force curve is shown in Fig.2 (right bottom) when the cantilever vertically approaches to and then is detached from the substrate. Its x-axis indicates time step, and y-axis shows the normal force exerted on the interface. Points from (a) to (b) indicate that the upper specimen approaches close to and then contacts the lower specimen surface, resulting in a normal load increasing from nearly zero to 3 mN. The normal load can be kept constant by a feedback circuit. When the two specimens are separated from each other, the force curve varies from (b), along (c), then to (d). Point (d) indicates a negative normal load, suggesting there is a detected attractive force still exerted on the interface before their complete separation. The maximum attractive force is referred to as pull-off force, which indicates that the maximum interfacial force that needs to be overcome to avoid any stiction failure involved in MEMS and micro systems. The whole setup is sealed in a closed box with a controlled relative humidity, and it is believed that the capillary effect is dominant in the measured pull-off force, since interfacial dry adhesion force is negligible compared with the capillary adhesion force. Furthermore, when the double-leaf cantilever with the attached upper specimen is driven to slide on the lower specimen in the lateral direction, the interfacial friction force can also be measured.

In our experiments, to investigate the effect of surface patterns on capillary pull-off force and friction force at different RH levels, the inside humidity is controlled to increase step by step through a humidifier. Each measurement is carried out after a 10-min stabilization of the inside humidity. From a sleeve, the lower specimen can be changed easily. During our test, the contact normal load for pull-off force test is kept at 3 mN, and then is held for 100 s. Approaching and detaching speeds both are kept at 5 μm/s. The environment temperature is 21°C.

3. Experimental results and discussion

3.1. Influence of micro dimples on capillary pull-off force

Using the same upper specimen, i.e. a 4 mm × 4 mm smooth silicon wafer, capillary pull-off forces on micro-dimpled Si surfaces, un-textured Si surfaces and OTS film are investigated at RH levels ranging from 15% to about 95%. Results are shown in Fig.3. From Fig.3, a critical level of RH is observed for all the specimens studied. Above the critical RH value, the capillary pull-off force increases abruptly for all the specimen, from a nearly un-detectable value to several hundred μNs. Meanwhile, for the micro-dimpled surface a low critical RH value (about 45%) is observed compared with that for an un-textured surface, in the cases of bare silicon surface (about 65%) and OTS film covered silicon surface (higher than about

85%). Above the critical RH value, the capillary pull-off force on micro-dimpled surface is obviously larger than those on the bare silicon surface and the hydrophobic surface. The different critical RH values suggest that the micro-dimpled surface begins to be sensitive to ambient humidity early from 45% RH, much earlier than the OTS film surface that is not sensitive to ambient humidity until 85%RH. The critical RH value indicates the potential working ambient humidity for micro devices like head-disk systems and MEMS. A higher critical RH value of the working surface implies that the micro system can operate smoothly with a higher humidity tolerance and with a lower possibility of adhesive failure. From this point of view, when selected as a working surface in an MEMS or a head-disk system, micro-dimpled surface will narrow down the working environment of the micro system due to its relatively high possibility of structure stiction compared with un-textured or hydrophobic surfaces.

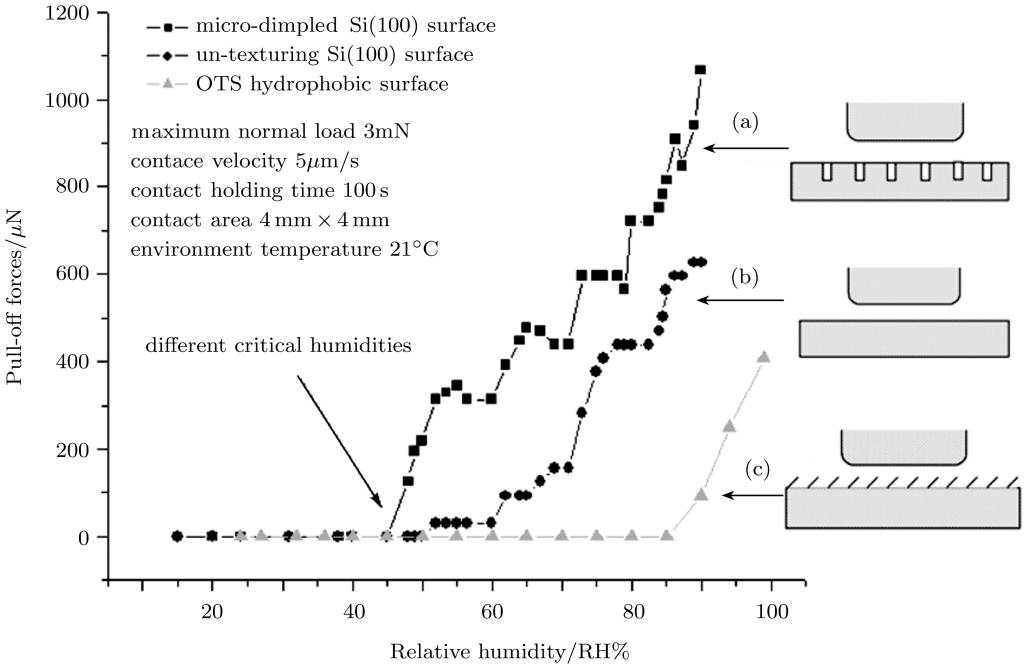


Fig.3. Capillary pull-off forces on (a) micro patterned surface, (b) un-textured surface and (c) OTS film surface. Micro-dimpled Si surface has a low critical RH value and high capillary force compared with others.

Our results on micro-dimpled surfaces are a little different from previous experimental results given in Refs.[3, 10, 11]. It was generally considered that[1−3] a rough surface has a high critical RH value compared with a smooth surface, for surface roughness can reduce the real contact area, and so reduce the capillary adhesion force. Experimental results from Rabinovich *et al*[3] also show that (in Fig.4(a)) critical RH value increases from 20 to 60% as surface roughness increases from 0.2 to 0.7 nm, meanwhile pull-off force decreases obviously. So they suggested that a patterned rough surface was suited to be used as a working surface in micro systems to reduce the capillary effect and avoid stiction failure.

To reveal the discrepancy, we focus on the details of the texture patterns of our specimens and those from previous tests.[3] It can be seen that the micro-dimple patterns on our specimens are geometrically concave in nature, while the sample surfaces in Rabinovich *et al*'s experiments are convex generally. The growth mechanisms of the liquid bridge on these kinds of surfaces are quite different, so the concave-patterned surface and the convex-patterned surface may form different interfacial water films. Firstly, water vapour is apt to condense in a narrow interfacial clearance, while a convex-patterned surface is liable to form liquid bridges on tops of the asperities as shown in Fig.4(b), thereby the liquid film is separated into many discrete small bridge necks. As for a concave-patterned surface, e.g. the micro-dimpled surface in our test, the condensed water generally forms a uniform water film on the parallel interfaces, which is not easy to be separated into discrete liquid bridges. Secondly, besides the condensation of water vapour, the

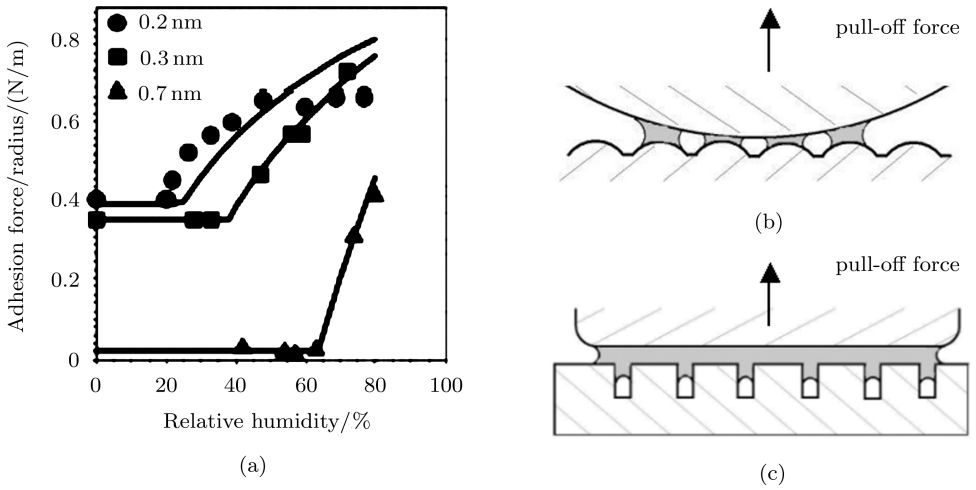


Fig.4. Experimental results given by Rabinovich *et al*[3] (a), contact details obtained from Rabinovich *et al*'s model (b), and contact details of the micro-dimpled surface in our test (c).

thin liquid film on the substrate flowing to the contact zone also influences the formation of liquid bridge, which even becomes a dominant mechanism as the contact time increases.[13] Obviously, the motion of thin liquid film on the substrate as exhibited in Fig.4(c) is easier to form a continuum water film. Additionally, the thin liquid film on the micro-dimpled surface may wet into the micro dimples as shown in Fig.4(c). The wetting liquid may reduce the liquid film thickness and meanwhile it can give rise to an additional separating resistance. Therefore, quite a different capillary mechanism results in a large pull-off force on micro-dimpled surface compared with that on the specimen in Ref.[3].

Furthermore, to investigate the effect of dimple geometry on capillary force, a series of micro-dimpled specimens with different diameters, depths and distributions is tested. The geometry parameters are listed in the inset table in Fig.1. For different dimensions and distributions of micro dimples, the corresponding capillary force curves are quite different. To summarize the geometry effect of the micro dimples, a geometry parameter r, namely a sidewall area ratio, is proposed and adopted in this paper. It is defined as the ratio of the whole sidewall area of the micro-dimples to the nominal contact area of the two flat-flat contact surfaces.

$$r = \frac{\pi dh \times N}{a^2 - \pi(d/2)^2 \times N}, \tag{1}$$

where d and h are the diameter and the depth of the micro dimple respectively; a is the side length of the contact area, i.e. the side length of upper specimen; N is the number of dimples etched on a specimen. The specimen in our test is described by different geometry parameters, and the defined parameter r varies from 0.006 to about 0.03 according to expression (1). In order to indicate the force curves clearly, experimental results on four typical samples are presented in Fig.5. The four samples are labelled as sample 1, 2, 3 and 4 according to the values of parameter r in a decreasing order, i.e. 0.028, 0.018, 0.014 and 0.009 respectively. In Fig.5 we can see that as the value of r decreases from 0.028 for sample1 to 0.009 for sample 4, the critical RH value increases slightly from about 45% to 55%, while the capillary pull-off force decreases largely as indicated by the open square points to the inverse triangle points. Both the critical RH value and capillary pull-off force are shown to be closely dependent on the geometry parameter r, which suggests

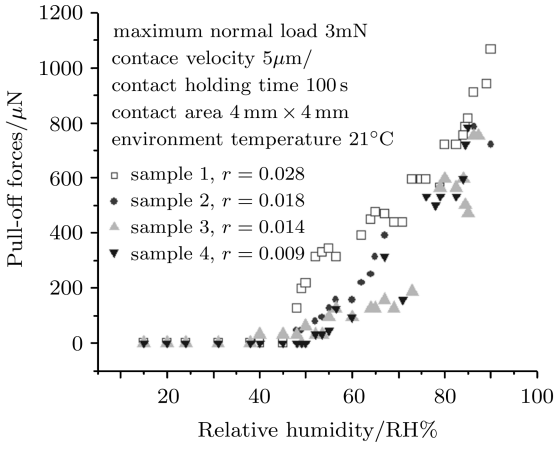


Fig.5. Curves for the capillary pull-off force versus RH value on micro-dimple surface with different geometry dimensions.

that the ratio r may be an effective geometry parameter of micro-dimpled surfaces to tailor its capillary pull-off force.

Actually, as we mentioned above, the continuum liquid thin film and the behaviour of its wetting into the micro dimples play an important role on the pull-off force curve of micro-dimpled surface. Obviously, as the wetting depth of the liquid increases, the interfacial pull-off force will increase due to the de-wetting resistance inside the micro dimples. So it would be more reasonable that the pull-off force is positively related with liquid wetting depth instead of the dimple depth itself, because when the dimple depth increase to a value above a limit, the wetting depth will not increase any more. A further analysis of the capillary pull-off force, with the wetting effect on micro dimple taken into consideration, is under way.

3.2. Influence of contact geometry on frictional property

Furthermore, friction tests are carried out on the specimens at different RH values. The normal load is kept constantly at 314 μN, sliding speed is 2.7 μm/s, and sliding stroke is 100 μm. The sliding speed is selected to be not so high to avoid any hydrodynamic lubrication effect. For each test, the coefficient of friction (COF) is calculated by dividing the average value of the detected friction force by the normal load. Results are summarized in Fig.6. In Fig.6(a), the COF of the un-textured sample surface is initially 0.6 followed by a slight decrease to a relatively stable value of around 0.4, and finally increases abruptly to more than 2.2 after 85%RH. The high COF after 85%RH is observed to be accompanied with an obvious stick-slip behaviour, which makes the frictional property quite unstable. In contrast, the micro-dimpled sample surface owns a lower, more stable friction coefficient of around 0.3 throughout the test, except a peak value of about 0.7 at 80%RH as shown in Fig.6(b). For comparison, frictional force on OTS film is also investigated. As shown in Fig.6(c), the hydrophobic surface has quite a stable and low COF, less than 0.2, at different RH values, which means that it is quite insensitive to RH value. Focusing onto the un-textured Si surface and the micro-dimpled Si surface, we can see that a reduction in COF on micro-dimpled surface is obvious, especially after 85%RH where the COF of un-textured surface increases abruptly. The difference

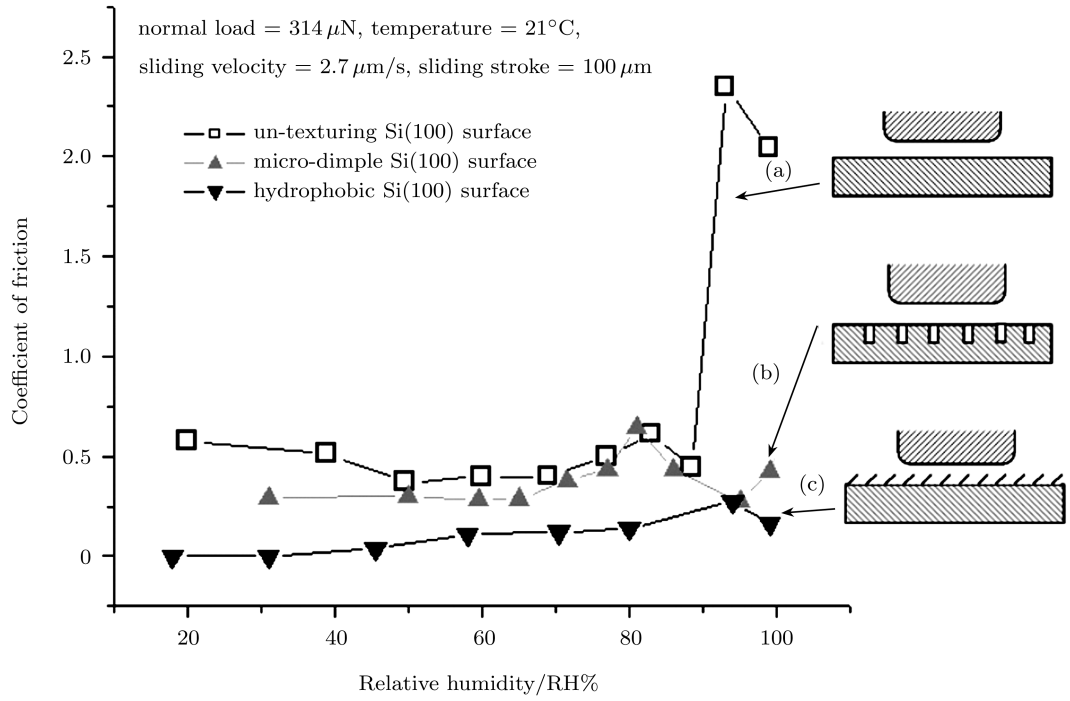


Fig.6. Coefficients of friction on an un-textured Si surface (a), a micro-dimpled Si surface (b), and an OTS film surface (c).

mainly may be due to the breakage of the liquid bridges, which can give rise to fluctuation on lateral resistance force, and the micro dimples on surface, which may act as reservoirs to store and supply water during relative sliding to avoid sudden breakage of liquid bridges, which eventually produces a relative stable friction force. This implies that the appropriate texturing of micro-dimpled surface may succeed in friction control and friction reduction compared with un-textured surfaces.

4. Conclusions

The capillary pull-off forces and the frictional forces on micro-dimpled silicon surface and un-textured silicon surfaces, bare and OTS film covered, are investigated by using a micro tribometer at different levels of relative humidity. The findings are summarized as follows.

(1) A critical RH value exists for all the tested sample surfaces, including hydrophobic surface, un-textured surface and regular patterned surface. When ambient humidity is above the critical value, capillary pull-off force increases abruptly with RH.

(2) A regularly patterned micro-dimpled surface has a low critical RH value and high capillary fore compared with an un-textured surface and a hydrophobic surface. A geometry parameter, namely sidewall area ratio, is found to be an effective parameter to tailor capillary pull-off force on a micro-dimpled surface.

(3) The COF for micro-dimpled silicon surface is found to be not so sensitive to the variation in RH level compared with that for un-textured silicon surface. The reservoir-like function of micro dimples is suggested to explain the stable frictional behaviour observed.

In summary, micro patterned surface is a promising option as working surface involved in MEMS and micro systems. Some aspects of the dominant parameters and potential mechanism in micro-dimpled surface are studied. Further investigations are still needed to reveal the mechanism and control strategy for other micro patterned surfaces.

Acknowledgments

The authors sincerely thank S. I.-U. Ahmed and W. Hild for fruitful discussions.

References

[1] Burton Z and Bhushan B 2005 *Nano Letters* **5** 1607
[2] Spengen W M van, Puers R and Wolf I D 2002 *J. Micromech. Microeng.* **12** 702
[3] Rabinovich Y I, Adler J J, Ata A, Singh R K and Moudgil B M 2000 *Journal of Colloid and Interface Science* **232** 10
[4] Guo X Y, Li X, Hu Y Z and Wang H 2008 *Chin. Phys.* B **17** 1094
[5] Ando Y 2000 *Wear* **238** 12
[6] Wakuda M, Yamauchi Y, Kanzaki S and Yasuda Y 2003 *Wear* **254** 356
[7] Schreck S and Zum Gahr K H 2005 *Appl. Surf. Sci.* **247** 616
[8] Murthy V S R, Kobayashi H, Tsurekawa S, Tamari N, Watanabe T and Kato K 2004 *Tribology International* **37** 353
[9] Bhushan B and Tian X F 1995 *J. Electron. Pack.* **17** 26
[10] Wei Z and Zhao Y P 2004 *Chin. Phys.* **13** 1320
[11] Cleaver J A S and Tyrrell J W G 2004 *Chemical and Process Engineering* **22** 1
[12] Farshchi-Tabrizi M, Kappl M, Cheng Y J, Gutmann J and Butt H J 2006 *Langmuir* **22** 2171
[13] Wei Z and Zhao Y P 2007 *J. Phys.* D *Appl. Phys.* **40** 4368