Quasi-Static Frictional Test between Silicon Sharp Probes with in-situ TEM Observation of Real Contact Point

Tadashi Ishida, Takaaki Sato, Shinsuke Nabeya and Hiroyuki Fujita

Center for International Research on Micro/Nano Mechatronics, Institute of Industrial Science, University of Tokyo, 4-6-1 Komaba, Meguro, Tokyo 153-8505 JAPAN

tadashii@iis.u-tokyo.ac.jp

Abstract. Frictional deformation of a nano-scaled real contact area between silicon MEMS opposing tips was in-situ visualized by a transmission electron microscope and the force to deform the real contact area was simultaneously measured. In our experiment, the initial diameter of the real contact point was 6.4 nm. During the frictional motion at 0.01 nm/s, the force applied to the contact point was gradually increased. The maximum frictional force was 61 nN when the diameter of the real contact point was 2.2 nm. After the maximum force, the force gradually decreased to zero because the real contact point was retracted and finally fractured. The friction coefficient was 0.9, which is reasonable in comparison with the previous reports. This result suggested that the friction was caused by the deformation of the real contact point.

1. Introduction

According to Coulomb-Amonton’s law, friction is not dependent on apparent contact area at macro scale [1]. This is because the interface between two objects always has small gaps due to many micro/nano asperities on the surfaces. This causes that real contact area is less than the apparent contact area. From this point of view, friction was caused just at the real contact points at micro/nano scale. The real contact area can be assumed by counting the contact points and sum up their contact areas. Some exceptional cases to the law is, however, coming to appear, as nanotechnology developed. In nano- and micro-scaled mechanical devices, which is one of the important application in nanotechnology, have a micro- and nano-scaled contact point [2,3]. At such a small contact point, the interface at the contact point has few or no gaps. This means that the apparent contact area reaches the real contact point. In this condition, friction should depend on the contact area, against the law. Therefore, it is necessary to study nano-scaled tribology.

Additionally, the effect of friction is more significant at the nano scale than at the macro scale [4]. Friction is one of the surface phenomena and proportional to the square of dimension. On the other hand, gravity and inertia are volume phenomena, which are dominant in macroscopic physics. The volume phenomena are proportional to the cubic of dimension. In the miniaturized structure, the surface force becomes more influential than in comparison with the volume force. At the nanoscopic region, friction is one of the most dominant forces to determine the motion of the structure. It is necessary to study nano-scaled tribology for the further development of nano- and micro-scaled mechanical devices [5].
For the study of nano-scaled tribology, it is important to visualize the real contact points, the origins of the friction, during the frictional experiment. To fulfill the requirement, Atomic force microscope (AFM) has been installed in a transmission electron microscope (TEM) [6]. A nano-scaled contact point was visualized during the frictional motion with this equipment [7]. There still remains the problem concerning stability because the AFM apparatus easily suffers from external vibration and drift of piezoelectric actuators. For the further study of nano-scaled tribology, an experimental setup, which allows nano-sopic observation and stable actuation, is necessary. In comparison to the piezoelectric actuators, micro electrostatic actuators have better stability and multi-fuctionalization. Hence, we developed microelectromechanical systems (MEMS)-in- transmission electron microscope (TEM) [8] and performed a frictional experiment under the nanoscopic observation. In this experiment, silicon is chosen for the surface material because it is one of the most popular materials in micro/nano mechanical devices and the devices encountered friction and wear problems [2]. The relationship between frictional force at a single silicon real contact point and its realtime deformation was reported.

2. Experimental Setup

For the visualization of nano-scaled deformation at the silicon real contact point, we performed a frictional experiment using MEMS-in-TEM. Silicon MEMS opposing tips with 2 degree-of-freedom (DOF), MEMS-in-TEM system and a force measurement method between MEMS tips are explained in the following subsections.

2.1. Silicon MEMS Opposing Tips

Figure 1 shows a schematic illustration of a MEMS device with silicon opposing tips. The silicon MEMS opposing tips have electrostatic actuators that allow 2 DOF motion; in the approach direction and the frictional direction. One tip approaches another when a voltage, \( V_{DX} \), is applied between a movable electrode and driving electrodes for approach. Another tip also moves in frictional direction when a voltage, \( V_{DY} \), is applied between a movable electrode and driving electrodes for friction. These electrostatic actuators have precise displacement control within sub-nm order and good stability over several hours.

All the electrodes in this MEMS opposing tips were fabricated with bulk micromachining and opposing tips were formed with focused ion beam (FIB) etching. The detailed fabrication process is explained in ref. 7. Damaged layer was introduced into the surface of opposing tips by FIB etching [9]. To remove this damaged layer, silicon opposing tips were beaten each other in the ultra high vacuum (UHV) chamber of TEM. The damaged layer was removed with this mechanical cleaning. The pure silicon crystalline structures appeared on the surface of tips.
Figure 1. Schematic drawing of MEMS device with silicon opposing tips. Electrostatic actuators between movable electrode and driving electrode move silicon opposing tips to approach and frictional directions.

2.2. MEMS-in-TEM system

Figure 2 shows the MEMS-in-TEM experimental setup. The MEMS device with silicon opposing tips were inserted into UHV-TEM specimen chamber using a specially customized sample holder for MEMS device. This TEM sample holder had 9 feedthroughs to apply voltage to the electrostatic actuators. With changing the voltage, the motions of opposing tips were controlled, resulting in the frictional test inside TEM. The spatial resolution was 0.2 nm and the time resolution was 1/30 s. The vacuum of TEM specimen chamber was 3x10^{-8}~1x10^{-7} Pa, which kept the surface of tips clean. An electron beam from a TEM electron gun irradiated silicon opposing tips and projected an high resolution image on its screen. Strong magnetic field over 1 T was applied to the device due to electron lens of TEM. The current density of the TEM electron beam for the observation was 1.6x10^{4} A/m². With this electron beam, the heating effect was less than 1 K, which was enough low to consider that the condition was at room temperature [10].

Figure 2. MEMS-in-TEM system. MEMS opposing tips can be driven inside TEM specimen chamber, applying driving voltages with a TEM special sample folder for MEMS device.

2.3. Force Measurement Method between MEMS Tips

The frictional force can be calculated using displacement difference of the movable tip between with and without a contact at a certain driving voltage. The displacement before the contact is determined by the balance between an electrostatic force and a restoring force of the movable electrode. After the contact, the displacement is determined by the balance among an electrostatic force, a restoring force and a frictional force. The frictional force is always applied to the contact point against the moving direction. Therefore, the displacement becomes smaller by this frictional force. According to the Hook’s law, the frictional force between tips can be calculated by the product between the displacement difference and the spring constant of the movable electrode. The displacement of the movable electrode can be measured by TEM observation. The spring constants of the movable electrodes for approach and for friction are 60 N/m, 23 N/m, respectively. Considering that the spatial resolution of our TEM is 0.2 nm, the force resolution should be 12 nN in approach direction and 4.6 nN in frictional direction.
3. Frictional Experiment between Silicon Tips

Figure 3 shows the experimental procedure of the friction test between silicon tips. The movable tip for approach moves towards the movable tip for friction (Fig. 4a). The tips are brought into contact. After the contact, a silicon nanocontact is formed between tips [12]. The movable tip for friction is driven, resulting in the deformation of the silicon nanocontact with frictional motion (Fig. 4c). The silicon nanocontact is fractured with the frictional motion of the tip (Fig. 4d). These steps are performed in the TEM specimen chamber to observe its deformation during the experiment.

(a) approach

(b) formation

(c) friction

(d) fracture & round

Figure 3. Experimental procedure of frictional test between silicon opposing tips. (a) approach, (b) formation of silicon nanocontact, (c) frictional deformation of the silicon nanocontact, (d) fracture of the silicon nanocnatact and rounding of the corner.

4. Deformation of Silicon Nanocontact and its Frictional Property

Figure 4 shows the nanometer scaled deformation of the silicon nanocontact during the experiment. The silicon nanocontact of 1.3 nm in diameter was formed between tips at the moment that the movable tip for approach pushed the tip for friction by 1.1 nm. The silicon nanocontact grew in diameter by a silicon atomic surface diffusion, while the actuator position was kept stationary. After 30 min from the contact, the diameter of the silicon nanocontact was 6.4 nm (Fig. 4a).

The silicon nanocontact formed between tips was plastically deformed with frictional motion of the tip. The displacement speed of the tip for friction was 0.01 nm/s in average during the frictional motion. The diameter of the silicon nanocontact gradually decreased from 6.4 nm to 3.6 nm before the displacement was 5.8 nm. At 8.7 nm in displacement, the diameter of the silicon nanocontact was 2.4 nm (Fig. 4b). The silicon nanocontact was finally fractured at the displacement of 16.2 nm (Fig. 4c).
The corners were rounded from 0.6 nm to 1.1 nm in radius of curvature in 5 min (Fig. 4d). During this frictional experiment, the displacement with the silicon nanocontact was smaller in comparison to that without the silicon nanocontact.

Figure 4. TEM images of each experimental step. (a) formation, (b) frictional deformation, (c) fracture, (d) rounding of the silicon nanocontact.

5. Discussion
From the observation of the displacement difference of the tip, the force applied to the silicon nanocontact was calculated. The force did not increase so much from the initial position to 5.8 nm in displacement. After the displacement exceeded 5.8 nm, the force suddenly increased. The force reached the maximum value of 61 nN with 8.7 nm in displacement. After the maximum value, the force gradually decreased to zero. At the moment when force became zero, the silicon nanocontact was fractured.

In this experiment, the normal force at the contact point was 66 nN. On the other hand, the maximum force applied to the silicon nanocontact during the frictional motion was 61 nN. From these values, the frictional coefficient was calculated to 0.9 using the equation, \( \mu = F/N \), where \( \mu \) is friction coefficient, \( F \) is friction force and \( N \) is normal force. According to the previous experimental reports, the static friction coefficient this experiment was comparable value [13-16]. This result suggested that the deformation of the silicon nanocontact caused the friction between tips.

6. Conclusions
The silicon nanocontact was plastically deformed caused by the frictional motion using silicon MEMS opposing tips under TEM observation. From the observation, it was confirmed that the frictional force was caused by the deformation of the real contact point during the frictional motion. This MEMS-in-TEM system will be a powerful tool to study nano-scaled tribology.

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