

# 증발 코팅법으로 증착된 광유와 실리콘 오일 윤활제의 마찰저감 특성

## Friction Reduction Properties of Evaporation Coated Petroleum and Silicone Oil Lubricants

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*As the size of mechanical components decreases, capillary forces and surface tension become increasingly significant. A major problem in maintaining high reliability of these small components is that of large frictional forces due to capillary action and surface tension. Unlike the situation with macro-scale systems, liquid lubrication cannot be used to reduce friction of micro-scale components because of the excessive capillary and drag forces. In this work, the feasibility of using evaporation to coat a thin film of organic lubricant on a solid surface was investigated with the aim of reducing friction. Petroleum and silicone oils were used as lubricants to coat a silicon substrate. It was found that friction could be significantly reduced and, furthermore, that the effectiveness of this method was strongly dependent on the coating conditions.*

Key Words: Friction Reduction (마찰 저감), Petroleum Oil Lubricant (광유 오일 윤활제), Silicone Oil Lubricant (실리콘 오일 윤활제), Evaporation Coating Method (증발 코팅 방법)

### 1. Introduction

Miniaturization of mechanical components can lead to many advantages such as higher precision, less energy consumption, less material consumption and lower material cost. Therefore, efforts to develop micro-scale devices are continuously expanding. As the size of mechanical devices becomes smaller, the inertia force decreases and the surfaces forces become important in dictating the behavior of the components. For example, when the system scale is below 1  $\mu\text{m}$ , the inertia force becomes less important, and instead the surface forces become important in predicting the behavior of the

system.<sup>1</sup> Such phenomena sometimes can lead to benefits in developing novel devices such as a climbing robot based on Gecko and scratch drive actuator.<sup>2,3</sup> However, the high surface forces can cause serious problems in micro-scale devices with moving components, because high adhesive force due to van der Waals and capillary forces may reduce the durability and reliability of the system.

To succeed commercialization of movable micro-scale devices, the problems associated with high friction between two contacting components should be overcome.<sup>4</sup> In this regard, numerous researches have been conducted.<sup>5,6</sup> In macro-scale systems liquid

lubricants are extremely effective in reducing friction. However, in micro-scale systems liquid lubricants cannot be used because of relatively high capillary and drag forces. Thus, other types of lubrication methods must be used for these systems. Instead of liquid type lubricant, various lubrication techniques for micro-scale devices have been researched. Thin coating materials including metallic solid lubricants, self-assembled monolayer (SAM), and carbon-based materials have been proposed.<sup>7,8</sup> Another method is the vapor phase lubrication technique.<sup>9,10</sup> In this technique, the lubricant is transformed into vapor and coated on the surface of the components to be lubricated. Typically, since the environmental temperature is below the boiling point of the lubricant, the vapor phase readily returns to the liquid phase as it cools on the surface. The amount of liquid is extremely small such that the capillary force effect can be reduced. Though this method of lubrication has shown to be effective in certain tribological systems, there needs to be further investigation in order to apply this method to a wider range of systems.

In this work, the effectiveness of evaporation coated petroleum and silicone oils in reducing the friction of silicon (Si) wafer was investigated. The motivation of this work was to develop a lubrication technique for silicon-based materials that uses a minimal amount of lubricant. Thus, rather than supplying the lubricant continuously, it was coated on the Si surface by evaporation. This method of lubricant coating was used since preliminary studies showed that spin coating or dip coating methods were not effective in producing a very thin layer of lubricating film on the Si surface. Evaporation of the lubricant was performed at temperatures below the boiling point of the lubricant oil. Friction tests were carried out using specimens prepared under different evaporation conditions. The experimental methods and results are presented in the following sections.

## 2. Specimen preparation and experimental details

In this work two kinds of lubricants, petroleum and silicone oils, were used for the friction tests. The two lubricants were chosen because of their different chemical and physical properties. The petroleum oil (Tri-

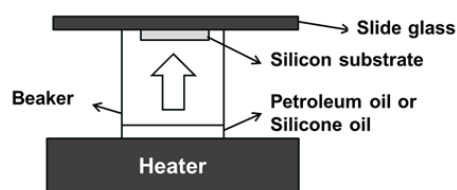
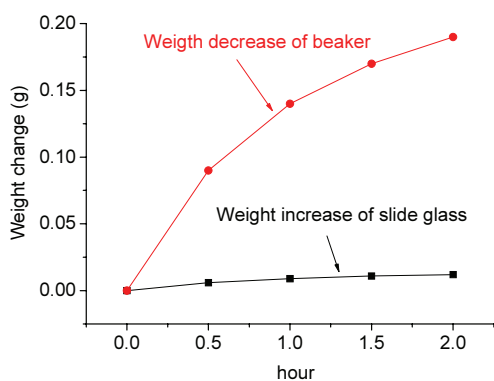


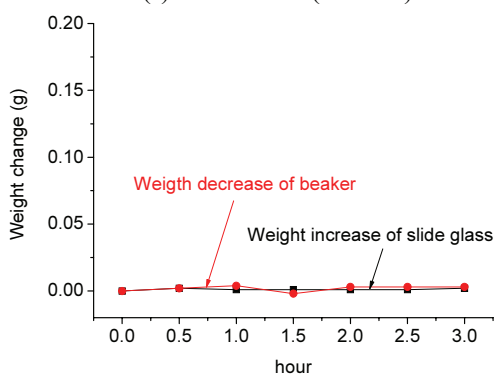
Fig. 1 Schematic of evaporation coating method

flow superior lubricant) is typically used for extreme temperature and humidity. This petroleum oil is basically formulated with polytetrafluoroethylene (PTFE). The working temperature range of the petroleum oil is  $-16^{\circ}\text{C} \sim 246^{\circ}\text{C}$  and the viscosity is 12 cSt at room temperature. The silicone oil (Shinetsu, KF-96-100CS) used in this work is basically formulated with polydimethylsiloxane (PDMS). The viscosity of this silicone oil is 100 cSt at room temperature.

The lubricants were coated on a Si wafer substrate by the evaporation method. Fig. 1 shows the schematic of the evaporation coating method. The petroleum and silicone oils were heated at  $100^{\circ}\text{C}$  and at  $200^{\circ}\text{C}$ , respectively. The heating temperatures were selected based on the thermal properties of the oil. Though petroleum oil evaporated sufficiently at  $100^{\circ}\text{C}$ , silicone oil required a higher temperature of  $200^{\circ}\text{C}$ . Also, considering safety, the silicone oil evaporating temperature was kept to be below  $300^{\circ}\text{C}$ , which is the flash temperature of 100 cSt silicone oil. Fig. 2 shows the weight change of the beaker and the slide glass with the Si wafer substrate attached to it with respect to the evaporation time. The weight decrease of the beaker represented the amount of evaporated oil from the beaker. Similarly, the weight increase of the slide glass/Si wafer represented the amount of oil coated on the slide glass surface including the Si wafer substrate. In the petroleum oil case, 6% of the evaporated oil was coated on the slide glass/Si wafer surface as shown in Fig. 2(a). The Si wafer area was  $1 \text{ cm}^2$  and the oil coated slide glass surface area was  $19 \text{ cm}^2$ . Thus the amount oil coated on the Si wafer was calculated by dividing the weight increase of the slide glass/Si wafer by 19. The calculated results indicated that after 1 minute of evaporation, 0.01 mg of petroleum oil was coated on the Si wafer, and after 2 hours, the weight of coated petroleum oil was 3 mg. In the case of silicone oil, it was difficult to measure the



(a) Petroleum oil (at 100°C)



(b) Silicone oil (at 200°C)

Fig. 2 Weight change of beaker and slide glass with Si wafer substrate attached to it w.r.t. evaporation time

increase of silicone oil weight with respect to evaporation time since the amount was too small to be resolved by the weight difference (Fig. 2(b)). After 3 hours of evaporation of evaporation time, the weight change of the slide glass/Si wafer was only 0.2 mg. This led to the calculation that about 0.01 mg silicone oil was coated on the Si wafer after 3 hours. By assuming linear behavior for the coating rate of the silicone oil during evaporation, it could be roughly estimated that about 0.003 mg of silicone oil was coated on the Si wafer after 1 hour of evaporation time.

The Si wafer substrates coated with evaporated lubricants were used for the friction tests. The tests were performed by a pin-on-reciprocating type of a tribotester (CETR UMT-2) as shown in Fig. 3. A stainless steel ball with a diameter of 1.6 mm was used as the pin. The ball was pressed against the Si wafer specimen with a size of

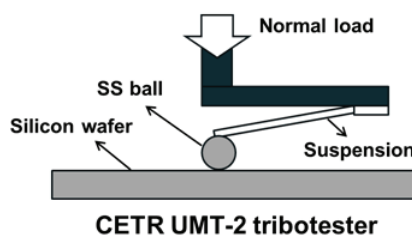


Fig. 3 Schematic of friction test performed using a pin-on-reciprocator type of a tribotester

10 mm by 10 mm. After contact, the stage was moved in a reciprocating motion to create sliding between the ball and the Si wafer. A normal load of 20 mN was used for all the tests. For this load, the calculated the maximum Hertzian contact pressure was 0.265 GPa and the radius of the contact area was 6 μm. The speed of the reciprocating motion was set to 0.1 mm/s and the stroke was 10 mm. The tests were performed for a period of 10 minutes. The frictional force between the two components was monitored during the test using a load cell attached to the ball holder. Each test condition was repeated in triplicate to verify the repeatability of the results. For all the tests the temperature was ~22°C and the relative humidity was ~22%.

### 3. Results of experiment

#### 3.1 Friction behavior

Fig. 4 shows the frictional force data obtained during the reciprocating test performed under 20 mN normal force. The three lines in the figure represent the data for three repeated tests conducted under identical conditions. The square-shaped data is representative of the reciprocating sliding motion. Thus, the negative frictional force was due to the reversed sliding motion. Fig. 4(a) shows the frictional force data of bare Si wafer specimen without lubricant. This data was used as the reference for the lubricated specimens. Fig. 4(b) shows the frictional force data of petroleum oil coated on Si wafer specimen for 2 hours. Surprisingly, this specimen showed higher frictional force than the bare Si wafer specimen. Finally, Fig. 4(c) shows the frictional force data of silicone oil coated Si wafer specimen for 3 hours. The frictional force of this specimen showed significantly lower frictional force than the bare Si wafer specimen. In all cases, the

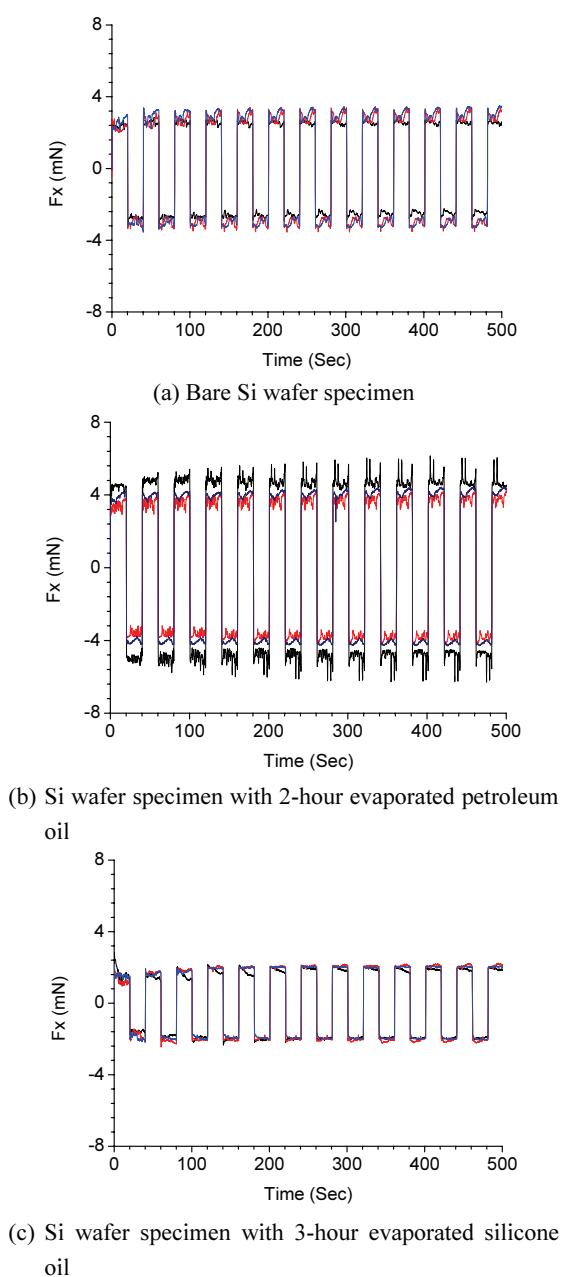


Fig. 4 Frictional force data w.r.t. time (each experiment was repeated three times, indicated by three lines in the figure)

frictional force was relatively uniform over the three repeated tests. Also, the force was quite steady during the entire duration of the test time.

From the raw frictional force data, the friction

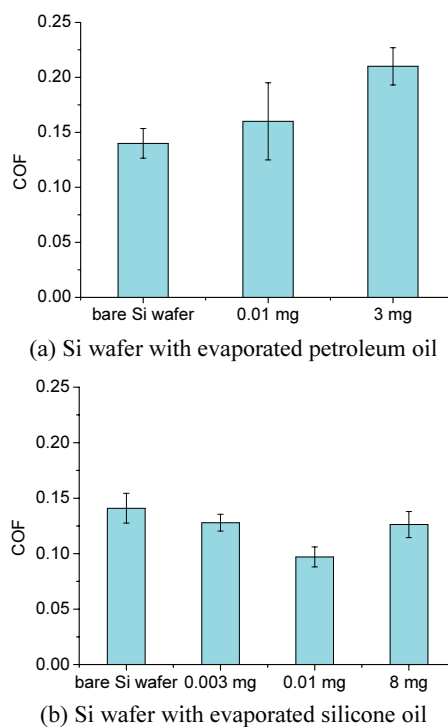


Fig. 5 Friction coefficient w.r.t. amount of lubricant

coefficient behavior with respect to the amount of lubricant coated on the Si wafer was obtained. As mentioned earlier, the amount of oil coated on the Si wafer could be varied by varying the evaporation time. Fig. 5(a) shows the friction coefficient data with respect to the amount of petroleum oil coated on the Si wafer specimen. As the amount of petroleum oil increased the friction coefficient also increased. With 3 mg of petroleum oil coated on the Si wafer, which was achieved by 2 hours of evaporation coating, the friction coefficient was about 0.2. This value was almost 50% higher than the friction coefficient of the bare Si wafer. Such a high friction coefficient was attributed to the viscous effect of the lubricant that played a significant role in the frictional force as the amount increased. Fig. 5(b) shows friction coefficient data with respect to the amount of silicone oil coated on the Si wafer. As the amount of oil increased to 0.01 mg, the friction coefficient decreased to about 0.09. Since it was not efficient to further increase the amount of silicone oil coated on the Si wafer by the evaporation method, a Si wafer specimen with a relatively large amount of lubricant was prepared by dropping the

silicone oil on the Si wafer. In this case, the presence of oil on the Si wafer was clearly visible and the amount was measured to be 8 mg. This was 200 times greater than the amount of silicone oil that could be coated on the Si wafer by the evaporation method in 3 hours. The friction coefficient of the Si wafer with 8 mg of silicone oil was similar to that of the Si wafer coated with 0.003 mg of silicone oil. Thus, it was evident that an appropriate amount of silicone oil is needed to achieve the minimum friction coefficient. Although 0.01 mg of silicone oil coated on the Si wafer was a relatively small amount, it was sufficient to lower the friction coefficient of Si wafer to below 0.1.

**3.2 Contact angle measurement of water droplet**

In order to better understand the state of the Si wafer surface after coating with silicone oil by the evaporation coating method, water contact angle measurements were performed using the coated specimens. Fig. 6 shows the optical image of the DI water droplet formed on the surface with respect to the amount of silicone oil coated on the Si wafer. The contact angle measurement results are showed in Fig. 7. As the amount of silicone oil on the Si wafer increased, the contact angle was also increased. This indicated that the Si wafer surface became more hydrophobic as the amount of silicone oil increased. However, in the case of the Si wafer specimen with 8 mg of silicone oil dropped on the surface, it was not possible to obtain the contact angle. This was because the water droplet was formed on top of the oil droplet, which made it impossible to define the horizontal reference plane needed to measure the contact angle. Nevertheless, the approximate shape of the DI water droplet formed on the silicone oil dropped specimen was similar to that of the 1-hour silicone oil coated specimen.

**3.3 Optical image of coated silicone oil on Si wafer surface**

Further characterization of the silicone oil coating behavior on the Si wafer was conducted by taking optical images of the coated Si wafer specimens. Fig. 8 shows the optical images of the Si wafers coated with silicone oil for 1 hour and 3 hours of evaporation. It was evident that the oil was not coated uniformly on the Si wafer surface. Rather, the oil existed in the form of islands on

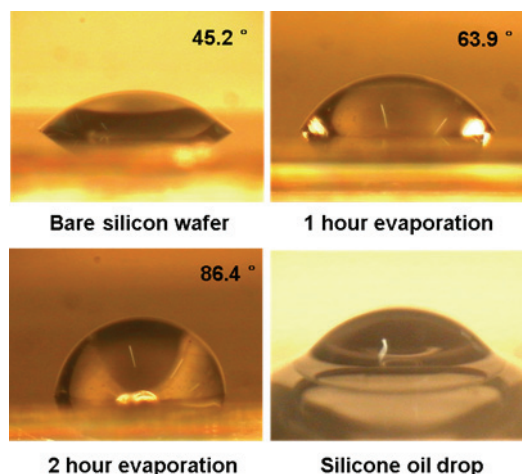


Fig. 6 Optical image of water droplet formed on the surface for contact angle measurement of various specimens

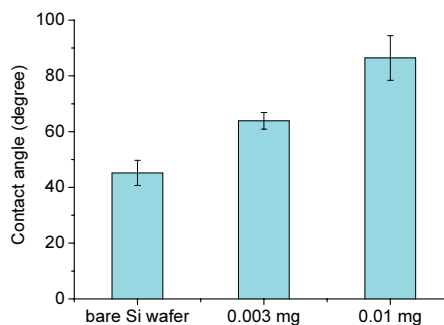


Fig. 7 Contact angle measurement with respect to amount of silicone oil coated on Si wafer

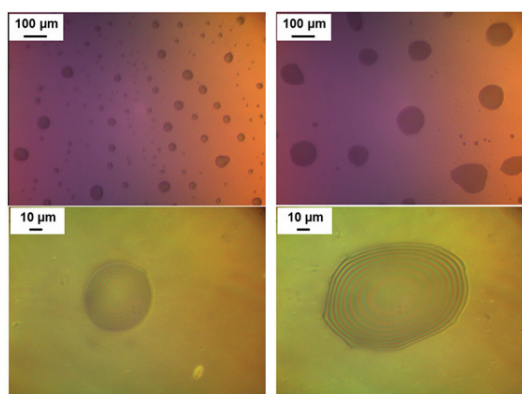


Fig. 8 Optical image of silicone oil coated on Si wafer specimen by (a) 1 hour and (b) 3 hours of evaporation

the wafer surface. Furthermore, it was clear that the size of the oil islands was significantly larger for the Si wafer specimen coated for 3 hours compared to that of the 1-hour specimen. This outcome was attributed to the reason for the lower friction coefficient obtained with the 3-hour coated specimen than the 1-hour coated specimen. Also, greater areal coverage of the oil islands on the Si wafer surface resulted in the higher contact area of the 3-hour coated specimen as shown in Fig. 7. Another important point to note was that a continuous oil film is not necessary to obtain low friction coefficient of Si wafer. These results indicate that evaporation method is effective in lowering the friction coefficient of silicon.

#### 4. Conclusions

In this work, the feasibility of using an evaporation method to coat a thin film of organic lubricant on the solid surface was investigated with the aim to reduce friction. For this purpose, petroleum and silicone oils were used as lubricants to be coated on a Si wafer substrate. Based on the experimental results, the following conclusions may be drawn:

- 1) The friction coefficient of Si wafer coated with petroleum oil by evaporation method was higher than the Si wafer coated by the same method using silicone oil.
- 2) The friction coefficient of Si wafer could be reduced to  $\sim 0.09$  from  $\sim 0.14$  by coating 0.1 mg of silicone oil over an area of 10 mm by 10 mm of Si wafer.
- 3) Excessive amount of silicone oil coated on Si wafer was not effective in reducing the friction coefficient. This was attributed to the increase in the viscous force of the lubricant.
- 4) Optical image analyses of the silicone oil coating behavior on Si wafer showed that the oil existed on the surface in the form of islands. Thus, a continuous oil film was not necessary to obtain low friction.
- 5) With further optimization, it is expected that the evaporation method can be used to coat a small amount of lubricant on solid surfaces to reduce friction.

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