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Slippery Behavior and Stability of Lubricating Fluid Coated Smooth Hydrophilic Surfaces

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Abstract

In recent years, many research groups have studied slippery characteristics on rough or porous hydrophobic surfaces infused with lubricating fluid. Alternatively, we present a simple method to fabricate stable slippery surfaces on hydrophilic samples which are more common and widely used. At room temperature, silicone oil lubricated hydrophilic samples exhibit non-slippery behavior for water drops as they sink into the oil layer due to the inherently hydrophilic surface. Subsequent annealing at higher temperature provides covalent bonding of silicone molecules at silicon surface making it hydrophobic. At optimized annealing temperature and time, the surfaces show excellent slippery behavior with negligible contact angle hysteresis and low sliding angle. Water drops on the slippery surfaces are found

cloaked with a thin layer of the lubricating oil to minimize the oil-water interfacial energy. Upon slipping, the oil cloaked water drops slowly remove lubricating oil which results in the degradation of slippery behavior. This degradation can be prevented by using large volume water drops or continuous water flow.

Introduction

Inspired by *Nepenthes* pitcher plants, lubricating fluid infused slippery surfaces are emerging as a hot topic for fundamental surface science as well enormous practical applications.¹⁻⁴ Various research groups have attempted to fabricate the slippery surfaces using different lubricating fluids infused on rough or porous substrates.⁵⁻¹⁸ Upon coating a suitable lubricating fluid on rough or porous substrates, test liquid drops can slip very effectively on them with very low friction. Due to the lubricating behavior, these slippery surfaces are found very useful for various applications ranging from drag reduction in liquid transportation, anti-ice and anti-frost performances with optical transparency, anti-biofouling, enhanced condensation and fog harvesting, Syringe siliconization to name a few.^{13, 19-27} Slippery surfaces are found more stable if underlying substrates are rough or porous as it provides larger surface area and capillary adhesion for the lubricating fluid.^{8, 15} Therefore rough or porous substrates are preferred while fabricating the slippery surfaces. Also the lubricant has to cover the entire surface therefore the substrates have to be completely wetting for the lubricating fluid. Test liquids, which slip as drops on the lubricating infused slippery surfaces, should be immiscible with the lubricating fluid. Also the test liquid should be non-wetting to the substrate surface.^{12, 15} That is why hydrophobic substrates are always used in slippery surfaces for aqueous drops. Aizenberg et al. observed the disruption of lubricating film on non-silanized (hydrophilic) epoxy based substrates whereas silanized (hydrophobic) epoxy showed stable lubricating film and good slippery behavior.¹⁵ Quéré et al. also indicated about

two possible situations for test liquid drops: floating or sinking, which is determined by surface tension of the liquids and surface energy of solid substrates.²⁸ Different fabrication techniques are used by various research groups to fabricate the lubricating fluid infused slippery surfaces. Aizenberg et al. used porous Teflon nanofibrous membranes to infuse perfluorinated lubricating fluid which shows excellent slippery behavior for various test liquids e.g. water, glycerol, ethylene glycol, alkanes, biofluids, crude oil etc.¹⁵ These surfaces maintained their slippery characteristics up to 700 atmospheric pressure. They also demonstrated that the slippery surfaces lower the nucleation temperature of super-cooled water thus provide an alternative anti-ice and anti-frost surfaces.²⁹ Alternatively, they also fabricated slippery surfaces through layer-by-layer deposition of silica nanoparticles followed by silane functionalization and fluorinated lubricant coating.³⁰ These surfaces also show excellent slippery behavior for variety of test liquids and are found very robust against damage. Varanasi et al. used lithographically patterned hydrophobic silicon substrates infused with either silicone oil or ionic liquid as lubricating fluid to slip aqueous drops.¹² Depending on the interfacial energy, they observed that the aqueous drops may get cloaked with a thin layer of lubricating fluid. Probing trajectories of immersed coffee particles in aqueous drops, they concluded that the drops actually roll rather than slip. These lubricated surfaces also show enhanced condensation of water droplets compared to dry substrates which can be very useful in heat transfer applications. They also demonstrated that these slippery surfaces reduce around 16% drag in laminar flow.³¹

In all these studies, either the used substrates were inherently hydrophobic or an additional step of hydrophobic coating was performed before infusing a lubricating fluid. Using hydrophobic substrates limit the choice of surfaces whereas hydrophobization process has its own limitation such as chemical compatibility of substrates, large area fabrication, multi step fabrication process, cost etc. Alternatively, if the hydrophobization of hydrophilic

substrates can be performed while or after coating a lubricating fluid using a convenient process, the overall fabrication procedure remains relatively simple. Here we demonstrate that annealing hydrophilic substrates after silicone oil (lubricating fluid) coating hydrophobizes the substrate surface which subsequently improves the lubricant adhesion as well as slippery characteristics. Optimization of various annealing parameters results in the most effective slippery surface with smallest contact angle hysteresis and largest slip velocity. If the lubricating fluid is washed away after annealing, substrates still show hydrophobicity confirming the hydrophobization of substrate surface. This method can be generalized to all the metal oxide and ceramic surfaces where -OH bonds can be introduced. Slipping water drops are cloaked with thin oil layer which results in the degradation of slippery behavior. The degradation is found to be dependent on size of slipping drops and for continuous flow no degradation is observed at all. Once degraded completely, the slippery behavior can be recovered simply by recoating the lubricating fluid.

Experimental section

Polished silicon (Si) wafers cut into 2cm × 2cm pieces were used as substrates for slippery surfaces. These substrates were cleaned by ultrasonication in ethanol, acetone and toluene for 5 mins each followed by oxygen plasma cleaning for 30 sec which results into hydrophilic surfaces with complete water spreading. Silicone oil (Sigma Aldrich, kinematic viscosity ~ 370 cSt) was used as the lubricating fluid which was spin coated on to the cleaned Si substrates. Subsequently the lubricating fluid coated samples were annealed at different temperature and time to optimize the slippery behavior as well as the stability of the lubricating fluid. Wettability of the lubricating fluid coated slippery surfaces was measured using an optical contact angle goniometer (OCA35, DataPhysics Germany) using sessile drop method. 10 µlit volume drops of de-ionized (DI) water were used as test liquid to investigate

the slippery behavior of the fabricated slippery surfaces. Slippery behavior was quantified by measuring water drop velocity on 10° tilted slippery surfaces and the tilt angle was kept constant for all slippery measurements. To investigate the stability of lubricating fluid, 50 ml volume step of water was dispensed drop-wise (10 μ lit drop volume) on the slippery surfaces, followed by measuring slip velocity using 10 μ lit volume water drop. Later, we also investigated the effect of dispensed drop volume on the stability behavior.

Results and Discussions

Cleaned silicon substrates, having a thin native SiO_2 top layer, are inherently hydrophilic as well as oleophilic with water and oil contact angles as 10° and 0° respectively. Spin coating silicone oil with varying *rpm* provides lubricating films with different initial thickness. Water drops on the as-coated silicone oil films are found unstable as they sink in the oil due to underlying hydrophilic silicon substrate. Supplementary movie S1 shows the sinking behavior of a water drop on a freshly silicone oil coated substrate. Strong polar interaction between hydrophilic Si surface and water molecules results into the sinking of water drops inside silicone oil making it non-slippery. So the most important requirement in using hydrophilic substrates as slippery surfaces is to first make them hydrophobic. This was achieved by annealing the lubricating fluid coated substrates at different temperatures for different time. Aizenberg et al. found that for stable slippery behavior, the lubricating oil should be wetting (oleophilic) and the test liquid should be non-wetting (hydrophobic) on substrates. In the present case (water and silicone oil as test liquid and lubricating fluid respectively), total energy for three different configurations are calculated as: configuration 1 for water on Si substrate, configuration 2 for oil on Si substrate and configuration 3 for water on silicone oil on Si substrate (cf. Figure 1). Conditions for stable slippery surface can be

written in terms of the energy difference between configurations 2 and 3 with that of configuration 1 as:

$$\Delta E_{12} = E_1 - E_2 = (\gamma_o \cos \theta_o - \gamma_w \cos \theta_w) + (\gamma_w - \gamma_o) \quad (1)$$

$$\Delta E_{13} = E_1 - E_3 = (\gamma_o \cos \theta_o - \gamma_w \cos \theta_w) - \gamma_{wo} \quad (2)$$

where γ_w , γ_o are surface tensions of water, silicone oil and γ_{sw} , γ_{so} and γ_{wo} are interfacial tensions between solid - water, solid - oil and water - oil respectively, θ_o and θ_w are silicone oil and water contact angles on Si substrate.

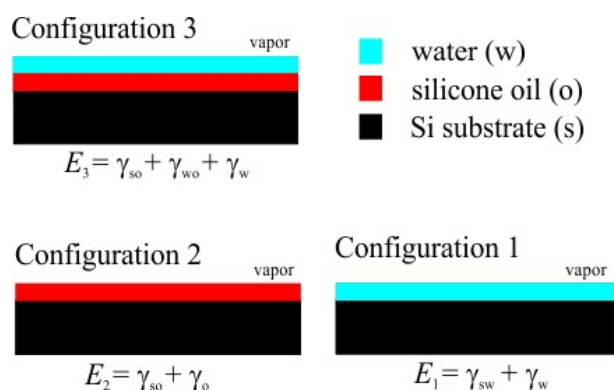


Figure 1: Schematic diagram of configurations 1, 2 and 3 to calculate total energy of the system. Configurations 1, 2 and 3 correspond to water on substrate, oil on substrate and water on oil on substrate respectively.

For stable slippery surfaces, the energy differences ΔE_{12} and ΔE_{13} should be positive. For freshly coated samples, the value of these energy differences are $\Delta E_{12} = 1.1 \text{ mJ/m}^2$ and $\Delta E_{13} = -92.5 \text{ mJ/m}^2$. As a result, test water drops sink into the lubricating oil layer depicting non-slippery behavior. Upon annealing silicone oil coated Si substrates, silicone molecules get covalently bonded to Si surface modifying γ_s , γ_{sw} and θ_w .^{32, 33} Therefore for the annealed samples the energy differences become positive as $\Delta E_{12} = 94.3 \text{ mJ/m}^2$ and $\Delta E_{13} = 0.65 \text{ mJ/m}^2$ thus preventing test water drops from sinking and making the slippery surfaces stable. Subsequently, the effect of annealing temperature and time was investigated to optimize the

annealing parameters. Figure 2 shows the effect of annealing temperature and time on slip velocity of water drops on slippery surfaces. First, Si substrates coated with silicone oil at different *rpm* were annealed at different temperatures for 90 mins to find out the optimum annealing temperature. As the annealing temperature is increased, the slippery behavior is also improved as indicated by increasing water drop velocity in Fig. 2(a). Initially, no improvement in slippery behavior is observed from room temperature till 50°C and further increasing the annealing temperature up to 200°C improves the slippery behavior with increasing drop velocity. Upon annealing at temperatures beyond 200°C, slippery behavior of the samples is destroyed since the temperature approaches the boiling and flash point of silicone oil (200°C & 250°C) and the lubricating film starts dewetting (breaking into drops).

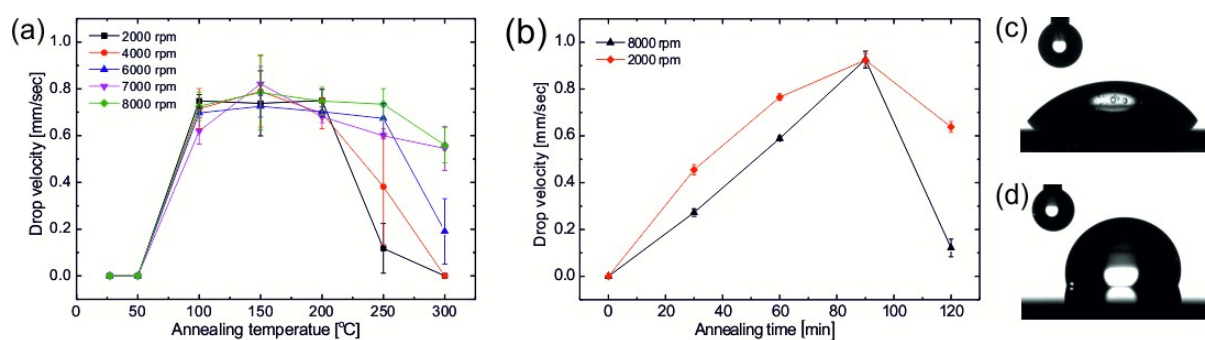


Figure 2: (a) Slip velocity of a test liquid (water) drop as a function of annealing temperature for lubricating films prepared at different rpm (annealing time was kept constant as 90 min), (b) Optimization of annealing time (annealing temperature was kept constant as 150°C), (c) and (d) optical contact angle images of a water drop on non-annealed and annealed samples.

Figure 2(a) suggests that annealing at 150°C provides the optimum slippery behavior as the drop velocity is the highest for this temperature. Figure 2(b) shows the optimization of annealing time for samples annealed at 150°C. As the annealing time is increased, the slip velocity increases improving the slippery behavior. The highest slip velocity is obtained for

samples annealed for 90 mins which provides enough time to silicone molecules to covalently graft with Si surface. Therefore the most efficient slippery surfaces can be fabricated by annealing freshly coated samples at 150°C for 90 mins (see supporting movie S2). Figure 2(c) & (d) shows optical contact angle images of water drops on silicone oil coated non-annealed and annealed samples. In case of non-annealed samples water drops sink inside the lubricating layer showing much lower apparent contact angle ($\sim 53^\circ$). For annealed samples, water drops do not sink and show equilibrium contact angle around 108° which is similar to the contact angle of water drops on monolayer of silicone molecules. Figure 2 also predicts that varying the thickness of silicone oil (prepared by changing spin coating *rpm* from 2000 up to 8000) does not affect the slippery behavior. This is due to the fact that for the annealed samples, the slip velocity is independent of the lubricant thickness in the given thickness range. So the optimized slippery surfaces, for all subsequent experiments, were fabricated by spin coating silicone oil at 8000 rpm and then annealing at 150°C for 90 mins.

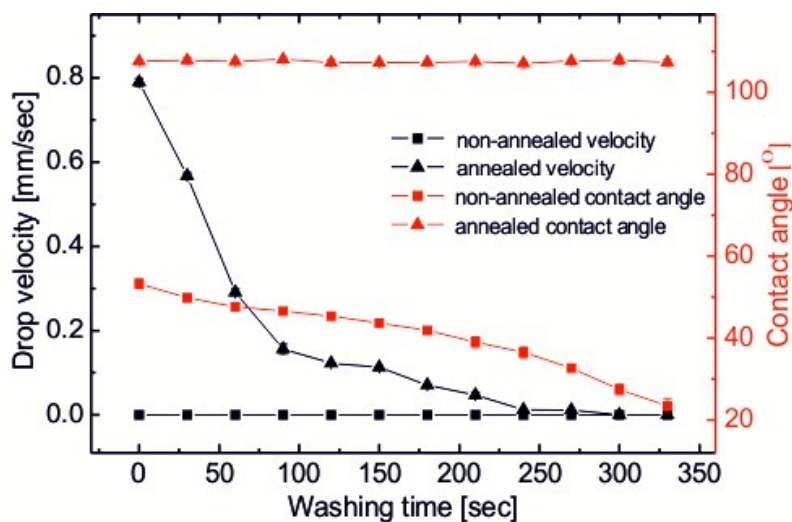


Figure 3: Drop velocities and contact angles of 10 μ lit water drops upon removing (by washing) the lubricant from non-annealed and annealed slippery surfaces.

To verify the chemical modification of Si surfaces upon annealing, which is essential for stable slippery behavior as suggested by the energy calculations (cf. Eq. 1 & 2), lubricant

was slowly removed from non-annealed and annealed samples and water contact angle and drop velocity were measured. Lubricant coated samples were washed in 2 wt% surfactant (Sodium Dodecyl Sulphate) solution in water via ultrasonication and water contact angle and drop velocity were measured as a function of washing time. Figure 3 shows slip velocity and static contact angle of water drops on silicone oil coated non-annealed and annealed samples as a function of washing time. For non-annealed samples, the slip velocity remains zero throughout the lubricant removal time as water drops feel strong polar interaction with hydrophilic Si surface. Static water contact angle on non-annealed samples decreases due to thinning of the silicone oil layer and increasing exposure to hydrophilic Si surface. Upon complete removal of lubricant, water contact angle becomes $\sim 24^\circ$ which is very close to the contact angle of bare Si surface. This confirms that for non-annealed samples, the Si surface remains hydrophilic and all silicone oil molecules can be removed via ultrasonication in surfactant solution in few minutes. On the other hand, for the samples annealed at 150°C for 90 mins, the slip velocity decreases as a function of lubricant washing time. This happens because the surfactant solution slowly removes silicone oil molecules from lubricant layer reducing its thickness thus decreasing the slip velocity. Upon complete washing the lubricating layer, the slip velocity again becomes zero making the surface non-slippery. Initial water contact angle for annealed samples is $108^\circ(\pm 3)$ which does not vary as a function of lubricant washing. Even after complete lubricant removal, the water contact angle remains 108° . This confirms for annealed samples that even after complete removal of lubricant, there is at least a monolayer of silicone molecules at Si surface which cannot be removed. This monolayer of silicone molecules at Si surface can be stable against surfactant washing only if they are covalently bonded with Si surface. That is why the water contact angle decreases on non-annealed samples but does not vary on the annealed ones as a function of lubricant removal. Figure 4 shows detailed XPS spectrum of Si2p, C1s and O1s core lines of bare and

annealed Si samples. Survey scan confirms the presence of Si, O and C in bare and annealed samples. Bare Si substrate shows standard Si bonds: O-Si-O (103.6 eV), Si-OH (102.5 eV), C bonds: C-H (284.6 eV), C-N (286.3 eV), C-O_x (287.1 eV) and O bonds: O-Si-O (532.2 eV), C-O_x (533.6 eV). The annealed samples show Si bonds: O-Si-O (103.6 eV), Si-C (100.3 eV), Si-O-Si (101.9 eV), C bonds: C-Si (284.5 eV), C-H (284.8 eV), C-O_x (286.7 eV) and O bonds: Si-O-Si (531.7 eV), O-Si-O (532.2 eV), C-O_x (533.6 eV). Binding energy of these peaks match with standard references.^{34, 35} Appearance of Si-O-Si and Si-C peaks in the annealed samples confirm the covalent bonding of silicone molecules with bare Si surface upon annealing.

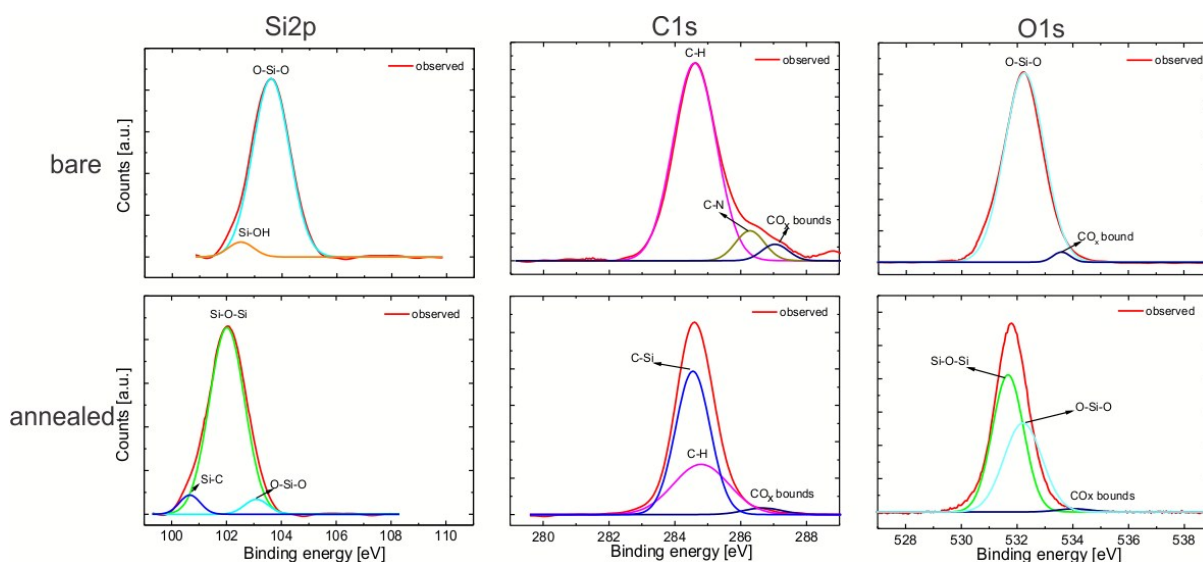


Figure 4: XPS core spectrum of Si2p, C1s and O1s of bare and annealed samples.

These covalently bonded silicone molecules make the Si surface hydrophobic as well as oleophilic which provides the necessary condition for a stable slippery surface. Contact angle hysteresis measurements were also performed on the annealed slippery surfaces. The hysteresis was measured by measuring the advancing and receding contact angles while increasing and decreasing the water drop volume. Due to the slippery characteristic of the surface, three phase contact line of a water drop could move easily upon increasing and

decreasing its volume thus resulting in very low hysteresis ($\Delta\theta \sim 2^\circ$). It confirms that the lubricating fluid surface is homogeneous, defect free and very smooth.

Annealed slippery surfaces show degradation in slippery behavior if very large number of water drops are dispensed through them. Therefore we performed the stability test of the slippery surfaces against water drops dispensing which is summarized in Figure 5. For this, slippery behavior was investigated after every step of 50 ml volume of water (as 10 μ lit volume drops) was dispensed from the slippery surfaces. Contact angle hysteresis was also measured along with slip velocity to predict any change in the surface energy and morphology. Slip velocity decreased monotonously after each step which is an indication of the removal of the lubricant layer responsible for slippery behavior. After dispensing about 800 ml of water, slip velocity becomes zero indicating almost complete removal of lubricant layer.

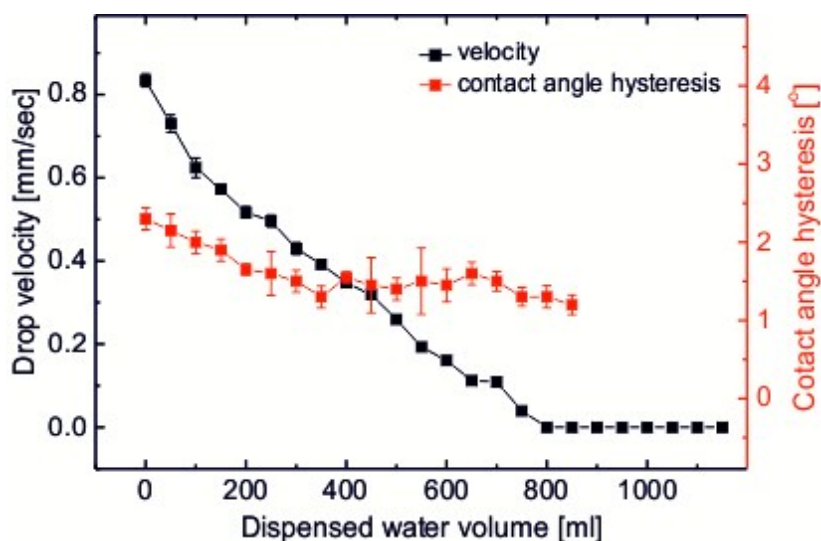


Figure 5: Durability test of slippery surfaces by measuring the slip velocity (black data, left Y-axis) and contact angle hysteresis (red data, right Y-axis) as a function of dispensing water.

Varanasi et al. showed that water drops deposited on a silicone oil coated solid surface are cloaked with a thin layer of the oil film.¹² The condition of the cloaking is defined by positive

spreading parameter of oil on water i.e. $S_{ow} = \gamma_w - \gamma_{wo} - \gamma_o$ which is equal to 8.5 mN/m for water and silicone oil system. Therefore water drops on silicone oil coated slippery surfaces are always cloaked with a thin layer of the oil. As water drops slip from the surface, silicone oil is also removed resulting in the thinning of the lubricating film and reducing the slip velocity. We found that this degradation in slippery behavior depends on the volume of the dispensing water drops. Smaller is the volume of dispensing water drops, larger will be the degradation in the slippery behavior due to large surface to volume ratio of smaller volume drops. Figure 6 shows the effect of dispensed drop volume on the durability of slippery surfaces. Using 10 μlit volume drops, complete degradation of slippery behavior is observed after dispensing total 800 ml of water whereas using 200 μlit volume drops only 10% reduction in drop velocity is observed for the same amount of dispensed water.

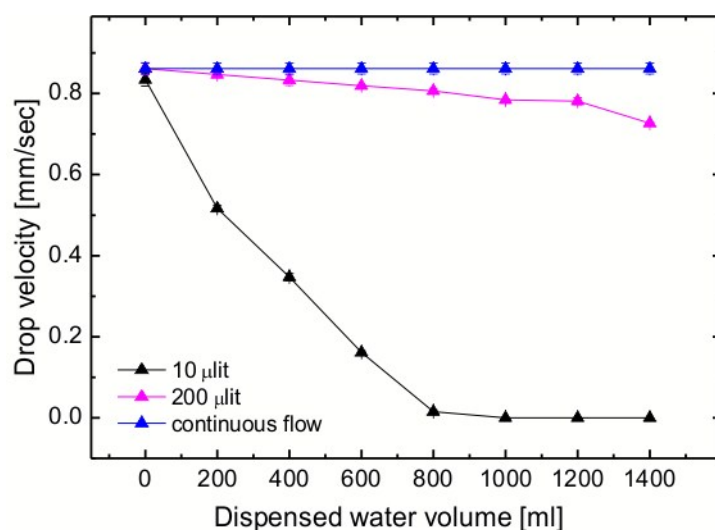


Figure 6: Durability test of slippery surfaces as a function of total dispensed water volume for three different volumes of drops.

So the total degradation would probably require about tens of litres of water. For continuous stream of water, since cloaking cannot occur, the slippery surfaces are found completely unaffected. In case if the slippery behavior is completely or partially deteriorated either due

to water flow or some other reason, it can be quickly recovered upon coating the lubricant again. Since the samples have already been annealed once, there won't be any need for annealing as the hydrophobicity will not be affected. We also checked that these surfaces behave as slippery not only for water but also for other silicone oil immiscible liquids e.g. glycerol, ethanol and ethylene glycol and show different static contact angles and slip velocity.

Conclusions

Hydrophilic Si surfaces coated with silicone oil as lubricating fluid depict very poor slippery behavior as slipping water drops sink inside the lubricating layer immediately. To prevent the sinking of water drops, the Si surface has to be hydrophobic as well as oleophilic which could be obtained by annealing the silicone oil coated Si surfaces. Annealing at 150°C for 90 mins provides the optimized slippery surface in terms of slip velocity, contact angle and contact angle hysteresis. Chemical modification of Si surface upon annealing was confirmed by washing the lubricating layer in a surfactant solution which systematically decreased the slip velocity but didn't affect the contact angle at all. Also, the slippery surfaces are found to be degrading as large number of water drops slip on them. This degradation is due to cloaking of silicone oil around water drops which slowly removes the lubricating oil during slipping. Using large volume drops or continuous stream of water prevents the degradation due to less or no removal of lubricating fluid. In case the slippery behavior is degraded, it can quickly be restored by coating the lubricant again on the surface.

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Lubricating fluid coated stable slippery surface on hydrophilic smooth silicon surfaces.

