

# Effective Icephobicity of Silicone Oil-Infused Oleamide–Polydimethylsiloxane with Enhanced Lubrication Lifetime

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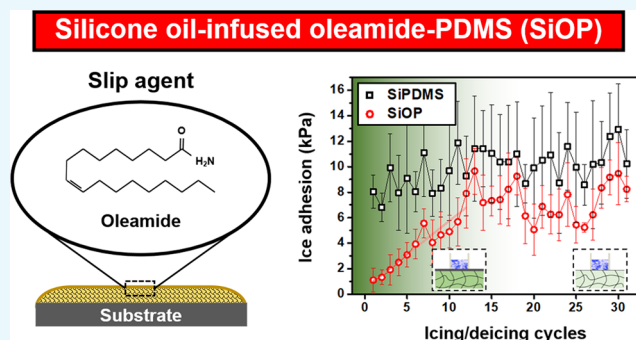


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Supporting Information

**ABSTRACT:** Icing and freezing phenomena in cold weather cause serious damage and economic losses. Thus, the development of a new effective icephobic surface with low ice adhesion strength ( $\tau_{ice}$ ) that can easily remove ice by wind or gravity force is essentially required. In this study, we propose a silicone oil-infused oleamide–polydimethylsiloxane (SiOP) by a facile fabrication method to achieve the effective icephobic performance with enhanced lubrication lifetime. The proposed SiOP is composed of a composite containing oleamide and polydimethylsiloxane (PDMS) and silicone oil impregnated into the polymeric networks of the composite. Oleamide has been used as a slip agent in industries to reduce the skin friction of polymer films. The weight of the oil impregnated in SiOP is approximately three times higher than that of silicone oil-infused PDMS (SiPDMS). Different from the SiPDMS surface on which oil dries easily, a slippery oil layer is stably formed on the SiOP surface. The fabricated SiOP surfaces have very low  $\tau_{ice}$  values of approximately 1 kPa, which is much smaller than that of the SiPDMS surface. The SiOP with an oleamide content of 5 wt % exhibits the smallest  $\tau_{ice}$  value of 0.88 kPa. The fabricated SiOP surfaces maintain their superior icephobicity for more than 30 icing/deicing cycles, demonstrating their enhanced lubrication lifetime. In addition, the ice freezing time of a water droplet of 7  $\mu\text{L}$  in volume is significantly delayed on the SiOP surface compared with that on the SiPDMS surface. The present results demonstrate that the proposed SiOP surface can help provide superior icephobic performance with the aid of the incorporation of oleamide into the conventional SiPDMS. The developed icephobic SiOP can be utilized to satisfactorily resolve the lubricant drought problem of conventional icephobic surfaces by employing oleamide as a complementary slip agent.



## 1. INTRODUCTION

Icing and freezing of machines, such as airplanes, ships, turbines, power lines, or home appliances, caused by cold weather lead to their failures, inducing serious human casualties and economic losses.<sup>1–3</sup> Accordingly, various methods, including mechanical, chemical, and thermal treatments, have been used to remove the ice accumulated on the solid surfaces.<sup>4–6</sup> However, these methods require a large amount of input energy, and most chemical treatments are not environmentally friendly.<sup>7</sup> Therefore, great efforts have been made to develop effective icephobic surfaces to passively prevent the formation of ice by using natural wind or gravity force without external energy consumption. To accomplish the passive removal of ice, the effective icephobic surfaces should have an extremely low ice adhesion strength ( $\tau_{ice}$ ) less than  $\sim 20$  kPa, which is the force capable of removing ice by a strong breeze.<sup>8,9</sup> Furthermore, the formation of ice can be inhibited by falling off the water on the surface before it freezes. The superhydrophobic (SHPo) surface, which is one of the typical icephobic surfaces, could easily remove the water with the aid of strong water repellency of the surface.<sup>1,10–12</sup> Given that the SHPo surface with micro/nano structures has a low contact

area and low sliding angle for water, the icing time of water on the SHPo surface is delayed, and the water on its surface easily falls off before the freezing. However, these conventional icephobic surfaces have a challenging problem in their practical utilization at high humidity and pressure conditions under which water or ice can be adhered to their micro/nano structures, thereby eventually worsening their icephobic performance compared with a smooth surface.<sup>13,14</sup>

Recently, a slippery liquid-infused porous surface (SLIPS) has been receiving great attention as a promising icephobic surface.<sup>15–18</sup> The SLIPS inspired by the morphological feature of pitcher plant was fabricated by injecting an immiscible lubricant into micro/nano porous structures.<sup>15,19</sup> Given that a smooth lubricating layer is formed on the SLIPS, the surface

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exhibits high water repellency even at high humidity conditions. When water is frozen on SLIPS, an ice–lubricant interface is formed, which can theoretically reduce the  $\tau_{ice}$  to zero.<sup>7</sup> However, the micro/nano structure fabrication process of SLIPS is expensive and complicated, and the injected oil is easily depleted by external physical stimuli (e.g., mobile liquid or ice on the surface).<sup>20,21</sup> To overcome this shortcomings of SLIPS, a liquid-infused polymer (LIP) was developed by integrating silicone oil into the polymeric network as a liquid lubricant.<sup>7,9,22–24</sup> Different from SLIPS, in which lubricant is injected into a void space, the polymeric network of LIP holds lubricant oil inside itself. LIP can be an economical choice from a practical point of view, thanks to its facile fabrication process and scalability. However, it is difficult for conventional LIP to efficiently utilize the oil stored in its polymeric network, thereby the oil on the surface dries up within a short period of time due to migration or evaporation.<sup>7</sup> This implies that the replenishment of oil from the internal polymeric network to the LIP surface is essential to extend the lubrication lifetime.<sup>25,26</sup>

To resolve this limit of LIP, we newly add oleamide to PDMS as an additional lubricant. Oleamide, a natural material extracted from plants or animals, has been known to weaken the adhesion of biofoulers and improve the scratch resistance.<sup>27–30</sup> This material has been used as a slip agent in industries to reduce the skin friction of polymers or plastic polyethylene films. Furthermore, oleamide is an economical and ecofriendly material.

In this study, we propose a silicone oil-infused oleamide–polydimethylsiloxane (SiOP) coating as an effective icephobic surface with enhanced lubrication lifetime. SiOP contains more oil than silicone oil-infused polydimethylsiloxane (SiPDMS), and the absorbed oil is sustainably replenished on the surface to form a thin lubricating layer. This indicates the effective oil storage and oil management ability of SiOP. Even after the SiOP surface eventually dries out, it maintains lower  $\tau_{ice}$  than SiPDMS, thanks to its lower elastic modulus and the slip effect of oleamide. In addition, oleamide slows down the time elapsed for freezing water droplets on the SiOP at low surface temperatures by decreasing the thermal conductivity of the polymer, supporting the potential for water to slide off before freezing.

## 2. RESULTS AND DISCUSSION

**2.1. Surface Characteristics.** The fabrication process of SiOP is schematically illustrated in Figure 1. At first, oleamide, the PDMS precursor, and the cross-linking agent are well

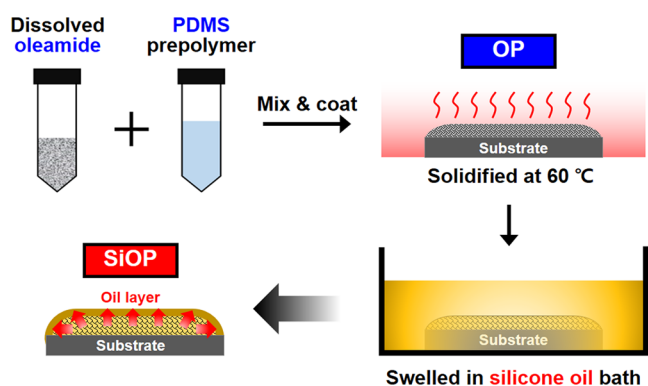


Figure 1. Schematic illustration of the fabrication process of SiOP.

mixed, and this mixture is coated on a substrate. The mixture coated on the substrate is placed in an oven to fabricate oleamide–PDMS (OP). The fabricated OP is then immersed in an oil bath. The polymer network is impregnated with silicone oil to fabricate SiOP. The following four test surfaces were prepared to investigate the surface characteristics and icephobic performance of SiOP: PDMS, OP, SiPDMS, and SiOP. Water contact angles (WCAs) of PDMS and OPs mixed at four different oleamide contents were compared by placing a water droplet of 5  $\mu\text{L}$  on each surface (Figure 2a). The surface of pristine PDMS has the highest WCA of 103° among the samples. This notion indicates that pristine PDMS is a hydrophobic surface. In comparison with pristine PDMS, the WCAs of OPs have slightly lower values due to the presence of the incorporated oleamide. However, all OP surfaces are still hydrophobic with WCAs of over 90°.

After impregnating silicone oil, all SiOP surfaces became hydrophilic with WCAs of less than 90° (Figure 2b). Moreover, they have the same WCAs of 87°, while OPs have different WCAs depending on the oleamide content. These same WCAs are attributed to the presence of the oil layer on the SiOP surface. A previous study reported that the contact angle of a water droplet of 5  $\mu\text{L}$  on the surface of 100 cSt silicone oil layer is approximately 86.4°, which matched that of SiOP impregnated with 100 cSt silicone oil.<sup>16</sup> Meanwhile, the WCAs of PDMS and SiPDMS are 103°. Specifically, the WCA does not change even after the impregnation of oil into PDMS. This result implies that the SiPDMS surface does not have an oil layer on its surface, unlike SiOPs. The presence of an oil layer on SiPDMS and SiOP was clearly confirmed by placing a blotting paper on the surface (Figure S1).

To form an oil layer on a polymer surface, such as like SiOPs, the polymer needs to contain a sufficient amount of oil.<sup>7,24,26</sup> If the polymer contains more than the critical amount of oil, then the oil molecules infused in the polymer networks are diffused out on the surface to form an oil layer. This notion indicates that the SiOP contains a sufficiently large amount of silicone oil; thus, the oil layer is sustainably formed on the surface. In contrast, SiPDMS does not diffuse oil molecules in the polymer toward the surface because the oil content is below the critical value.

To verify this aspect, we measured the oil contents of SiOP and SiPDMS by immersing PDMS and OP in an oil bath (Figure 3a). For oil content measurements, PDMS and OP were used after stripping from the substrate to accurately characterize the material properties. The peeled PDMS and OP are not bound to the substrate and thus undergo 3D swelling as the oil is absorbed. Here, the oil content ( $\Phi$ ) is defined as follows

$$\phi = \frac{m_{oil}}{m_0 + m_{oil}} \quad (1)$$

where  $m_{oil}$  is the weight of the oil infused in the polymer, and  $m_0$  is the initial weight of the polymer without oil impregnation. Specifically,  $\Phi$  indicates the ratio of the infused oil weight to the total weight of the polymer.

Figure 3b shows the temporal variations in the amount of impregnated silicone oil (0.65 cSt) of PDMS and OPs mixed at four different ratios. The incorporation of oleamide into PDMS largely increases the absorption of silicone oil. The  $\Phi$  value of PDMS reaches the equilibrium state at  $\sim 40$  min, while those of OPs reach the equilibrium within  $\sim 25$  min. In

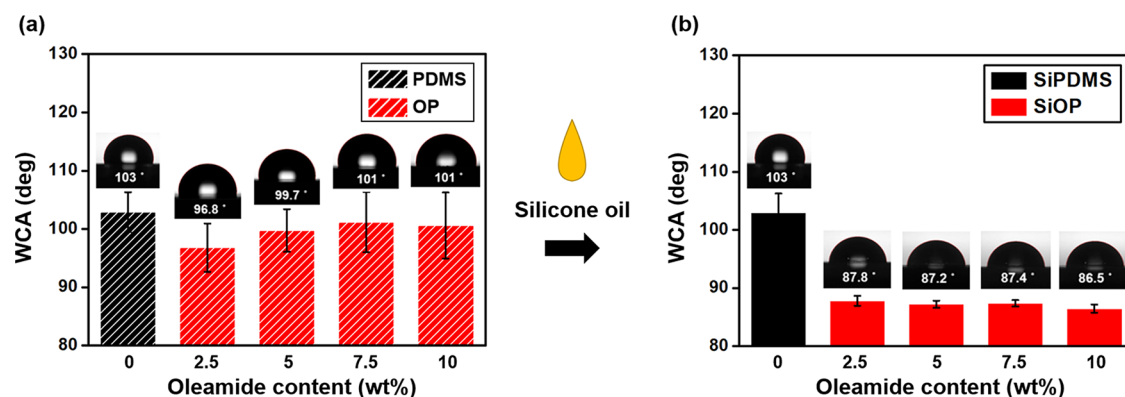


Figure 2. (a) WCAs of PDMS and OPs surfaces before oil impregnation. (b) WCAs of SiPDMS and SiOP surfaces after oil impregnation.  $N = 16$ .

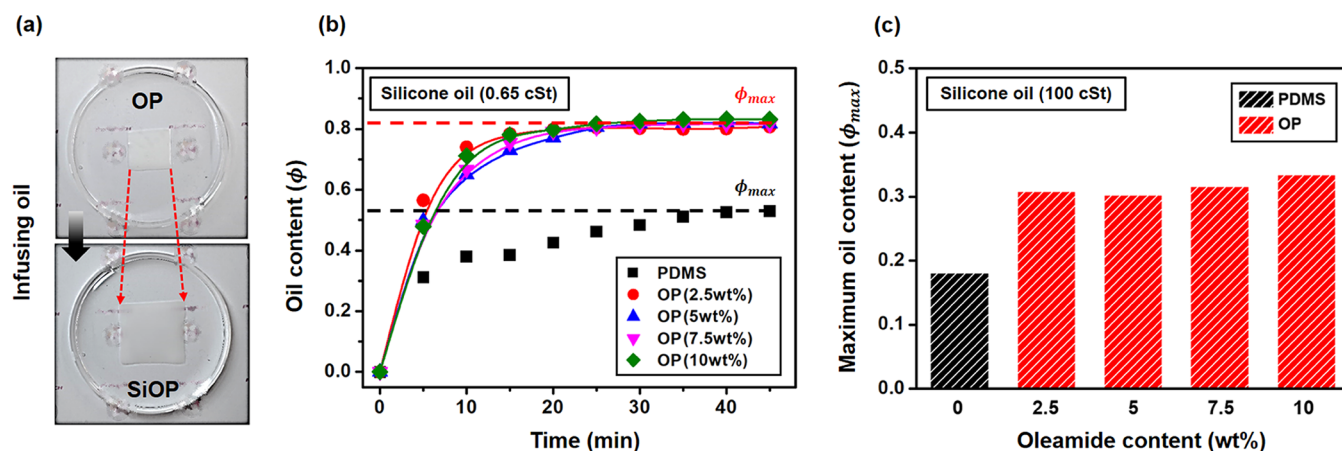


Figure 3. (a) Impregnation of silicone oil into OP to fabricate SiOP. (b) Temporal variations in the amount of impregnated silicone oil (0.65 cSt) in the fabricated PDMS and OPs. (c) Comparison of the maximum silicone oil (100 cSt) contents in PDMS and OPs.

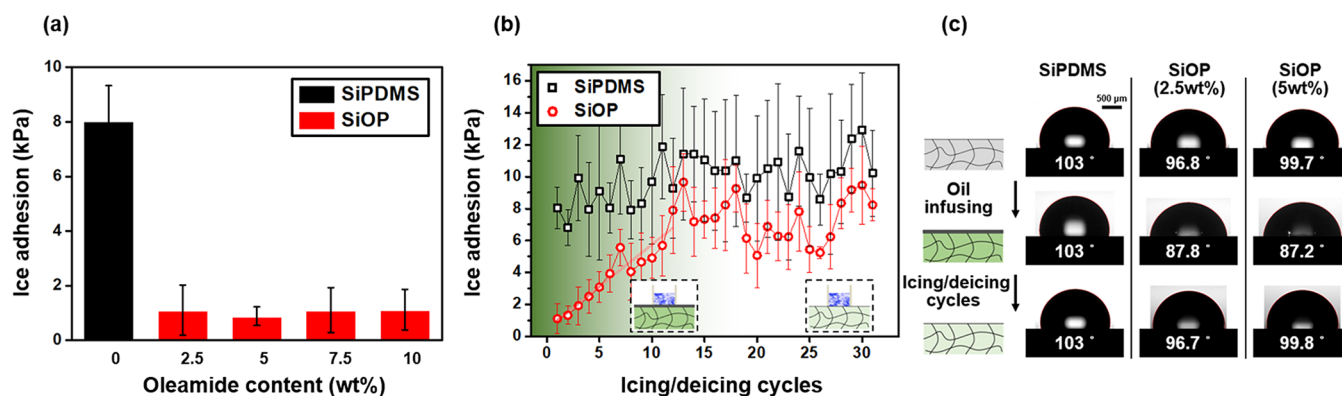


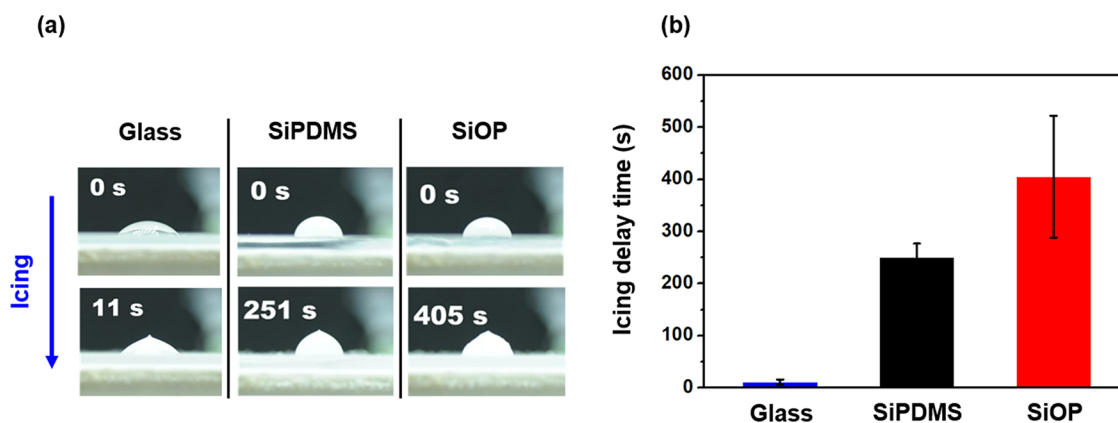
Figure 4. (a) Comparison of ice adhesion strength on SiPDMS and SiOP with different oleamide contents.  $N = 7$ . (b) Variations of ice adhesion strength on SiPDMS and SiOP surfaces according to the number of icing/deicing cycles.  $N = 7$ . (c) Variation of WCAs on SiPDMS and SiOPs according to oil infusion and icing/deicing cycle tests.

addition, the maximum oil content ( $\Phi_{max}$ ) of the four OPs with different mixing ratios is  $\sim 0.82$ , while that of PDMS is  $\sim 0.59$ . In comparison with the amounts of oil absorbed per unit initial mass ( $m_{oil}/m_0$ ), OPs contain approximately 3.3 times more silicone oil (0.65 cSt) than PDMS. Figure 3c compares the  $\Phi_{max}$  values of 100 cSt silicone oil used in the icephobic performance tests of PDMS and OPs. The  $\Phi_{max}$  values of PDMS and OP are approximately 0.18 and 0.32, respectively. The maximum content of 100 cSt oil per unit initial mass ( $m_{oil}/m_0$ ) of OP is approximately 2.3 times higher than that of PDMS.

This result is closely associated with the elastic modulus of the polymer. The shear modulus ( $G$ ) of a polymer can be expressed as follows<sup>31,32</sup>

$$G = \frac{\rho RT}{M_C} \quad (2)$$

where  $\rho$  is the mass density of the polymer,  $R$  is the ideal gas constant,  $T$  is the absolute temperature, and  $M_C$  is the average molecular weight of the polymer chains between cross-links. According to eq 2, the chain length between cross-links is



**Figure 5.** (a) Photograph of a water droplet on glass, SiPDMS, and SiOP before and after its freezing. (b) Comparison of IDT on glass, SiPDMS, and SiOP.  $N = 12$ .

increased with the decrease in the  $G$  of the polymer. This notion implies that more oil is impregnated with the expansion of the polymer network.<sup>32–34</sup> In OPs, the incorporation of oleamide in PDMS decreases the elastic modulus of the polymer; accordingly, the network expands and contains more silicone oil.<sup>28,29,34</sup> The presence of the oil layer on SiOPs can be confirmed by checking the increase in  $\Phi_{\max}$  value due to low elastic modulus of OP. When the  $\Phi_{\max}$  value exceeds the critical oil content, an oil layer is formed on the SiOP surface, unlike SiPDMS.

**2.2. Ice Adhesion Strength and Sustainability.** At a surface temperature of  $-10$  °C, the  $\tau_{\text{ice}}$  values of SiPDMS and SiOPs at four different mixing ratios were measured (Figure 4a). The  $\tau_{\text{ice}}$  value of SiPDMS is approximately 8 kPa. Meanwhile, all SiOPs have an  $\tau_{\text{ice}}$  value of approximately 1 kPa, regardless of the oleamide content. To evaluate the sustainability of the icephobic performance, an icing/deicing cycle test was conducted to repeatedly measure the  $\tau_{\text{ice}}$  values of SiPDMS and SiOP. As shown in Figure 4b, the  $\tau_{\text{ice}}$  values of SiPDMS do not significantly change. However, the  $\tau_{\text{ice}}$  value of SiOP rapidly increases up to approximately 12 cycles and thereafter converges to a value of 6.5 kPa. The  $\tau_{\text{ice}}$  values of SiOP are kept lower than those of SiPDMS up to 30 repeated experiments.

Figure 4c compares the WCAs of SiPDMS and SiOP measured at three stages (before and after the oil impregnation and after the icing/deicing cycle test). The WCA values of SiOPs (2.5 and 5 wt %) after the icing/deicing cycle test are the same as those before the oil impregnation. This result indicates that the oil layer on the SiOP surface disappeared after the icing/deicing cycle test. After the oil impregnation (i.e., before the icing/deicing cycle test), the WCA of both SiOPs is approximately  $87^\circ$ . This result supports the presence of the oil layer on the surface, as mentioned in Section 2.1.

The oil on the surface gradually disappeared with the repetition of the ice removal process because the fabricated SiOP surfaces have an oil layer at the beginning of the cycle test. Accordingly, the  $\tau_{\text{ice}}$  value of the SiOP surface is rapidly increased. When the oil layer disappeared from the surface, the  $\tau_{\text{ice}}$  values converged, and the ice column was in full contact with the elastomer surface. Several factors may influence the lower  $\tau_{\text{ice}}$  of SiOP than SiPDMS even after the oil disappeared from the surface. One of them is the elastic modulus of the coating material, which is strongly related to  $\tau_{\text{ice}}$ .<sup>35–37</sup> The

equation for  $\tau_{\text{ice}}$  of a soft material without oil layer on the surface can be described as follows<sup>38</sup>

$$\tau_{\text{ice}} \sim \sqrt{\frac{GW_a}{t}} \quad (3)$$

where  $W_a$  is the work of adhesion between the ice and the surface; and  $G$  and  $t$  are the shear modulus and thickness of the coating, respectively. According to eq 3,  $\tau_{\text{ice}}$  on the coating surface decreases as  $G$  of the coating material decreases. The reasons for the smaller  $G$  values of SiOP may be as follows: (1) the effect of oleamide incorporation and (2) the effect of oil infusion. In the former case, the incorporation of oleamide into PDMS gives rise to a smaller elastic modulus.<sup>29</sup> In addition, the oil impregnation softens the polymer and decreases the elastic modulus.<sup>7,34</sup> If the  $\Phi$  values of SiOPs are higher than those of SiPDMS after the oil dried on the surfaces, this would contribute to the low  $\tau_{\text{ice}}$  of SiOPs.

Furthermore, fatty acid amides have been known to have a slippery feature that can reduce frictional resistance exerting on solid surfaces.<sup>27–29</sup> The incorporation of a fatty acid amide into a polymer increases the yielding contact strain; accordingly, the friction coefficient is reduced. Thus, the pressure drop measured in a flow channel coated with OP is smaller than that in a channel coated with pristine PDMS.<sup>29</sup> This slippery property of oleamide may contribute to the lower  $\tau_{\text{ice}}$  value of the SiOP surface compared with the SiPDMS surface even after the oil on the surface dries.

**2.3. Icing Delay Time.** The freezing times of a water droplet of  $7 \mu\text{L}$  on SiOP and SiPDMS at a surface temperature of  $-10$  °C were measured to evaluate the ability to delay icing time (Figure 5). The freezing time was determined by monitoring the transparency of water droplets. The icing delay time (IDT) of SiOP is approximately 405 s. The delay is 37 times longer than that of a pure glass and 1.6 times longer than that of SiPDMS.

This result can be explained by using the following expression of IDT  $\Delta t$ <sup>39</sup>

$$\Delta t = \frac{\rho_w C_p (T_0 - T_1)}{\Delta Q} \quad (4)$$

where  $\rho_w$  is the density of water,  $C_p$  is the specific heat capacity,  $T_0$  is the initial temperature of the droplet,  $T_1$  is the surface temperature of the test sample, and  $\Delta Q$  is the net heat loss per unit time. The experimental conditions of  $\rho_w$ ,  $C_p$ , and  $T_0$  are

the same for all test samples. The surface temperature was measured by installing four thermocouples on the surface of each sample during the cooling stage. The temporal variations in the surface temperature of aluminum, SiPDMS, and SiOP (10 wt %) are nearly similar without significant difference (Figure S2). Therefore, the different IDT values of the test samples are attributed to the heat loss ( $\Delta Q$ ) of the cold solid surface. This notion supports that the incorporation of oleamide might decrease the thermal conductivity. Accordingly, the heat transfer from the water droplets to the SiOP is largely reduced, and the freezing time of water droplets is eventually delayed. Therefore, water droplets can be easily removed from the surface by inhibiting the freezing of water with prolonged IDT.

### 3. CONCLUSIONS

In this study, a new icephobic SiOP surface with enhanced lubrication lifetime was proposed and fabricated by impregnating silicone oil as a liquid lubricant into the polymeric networks of OP, and its superior icephobic performance was experimentally evaluated. As a result, the maximum oil content in the fabricated SiOPs is approximately three times higher than that of SiPDMS. The proposed SiOP surfaces form a stable oil layer unlike SiPDMS. The  $\tau_{ice}$  values of these surfaces are  $\sim 1$  kPa, which is approximately eight times smaller than that of SiPDMS. The SiOP with an oleamide content of 5 wt % has the smallest  $\tau_{ice}$  of 0.88 kPa. This excellent icephobic performance was sustained for more than 30 icing/deicing cycles. Several reasons for the superior ice adhesion property of SiOP are as follows: first, the oil layer formed on the SiOP surface largely reduces  $\tau_{ice}$  on its surface. However, after numerous repeated clearing processes, the oil layer on the SiOP surface eventually dries out. The incorporation of oleamide leads to the increased elasticity of the polymer and the slip effect at the interface between ice and surface, which decreases  $\tau_{ice}$  on the SiOP surface without the oil layer. In addition, oleamide also delays the freezing of water on the SiOP surface by the decreased thermal conductivity of the embedded polymer. The proposed SiOP surface would be effectively utilized in various engineering applications, which require superior anti-icing and de-icing functions.

### 4. EXPERIMENTAL METHODS

**4.1. Preparation of the SiOP.** A PDMS precursor (SYLGARD 184 base, Dow Inc., USA) and a curing agent (SYLGARD 184, Dow Inc., USA) are mixed at a ratio of 10:1, and then, the mixed solution is poured onto an adhesive (1200 OS Primer, Dow Inc., USA)-bonded substrate to coat PDMS.

To fabricate OP, four different amounts of oleamide corresponding to mixing ratios of 2.5, 5, 7.5, and 10 wt % are prepared. For example, 10 g of PDMS and 0.52632 g of oleamide are used to fabricate the OP with an oleamide content of 5 wt %. The prepared oleamide is immersed in 4 mL of toluene and dissolved by shaking at an ambient temperature of 70 °C for 10 min. Thereafter, the oleamide dissolved in toluene is combined with the mixture of a PDMS precursor and a curing agent. They are mixed evenly for 3 h by using a vortex generator (Vortex-Genie, Scientific Industries Inc., USA). This well-mixed solution is poured onto an adhesive-bonded substrate to coat OP.

The PDMS and OP-coated substrates are placed in an oven at 60 °C overnight to solidify the coatings. SiPDMS and SiOP

are finally fabricated by immersing the solidified coatings in a silicone oil bath to impregnate the oil.

**4.2. Sample Characterization.** The test samples (SiPDMS and SiOPs) taken out from the oil bath were covered with residual oils on these surfaces. The residual oils were removed by flowing water at a flow rate of about 4 m/s for 45 s over the sample surfaces just before the main experiments.

A deionized water droplet of 5  $\mu$ L was placed on the test surfaces to evaluate the wettability of the fabricated SiPDMS and SiOP surfaces, and WCA was measured with a contact angle measurement device (SmartDrop, Femtofab, Korea). The WCA was obtained by averaging a total of 16 contact angle values by repeatedly measuring four times at different four points on each test surface. The oil content in the impregnated polymer was measured by using an electronic balance. The test samples used for oil content measurements were stripped from the substrate to accurately characterize the material properties.

**4.3. Ice Adhesion Strength Measurements.** Icephobic performance tests were conducted in a room with a maintained humidity of 60% and a temperature of 26 °C. A home-made adhesion tester was used to measure the  $\tau_{ice}$  (Figure S3). First, an acrylic cylinder was placed on the aluminum plate (60 mm  $\times$  60 mm  $\times$  1 mm) coated with a test sample. Then, 10 mL of deionized water was poured into the cylinder of 36 mm in inner diameter and frozen for more than 12 h. The coated surface on which an ice column was attached was tightly fixed onto the cooling stage (LHP-800CP, TECA Co., USA) maintaining a surface temperature of  $-10$  °C. A force gauge (FB-N, IMADA Co., Japan) was fixed on a moving stage (LSP02, Longer Precision Pump Co., China). The device pushed the ice column at a speed of 10 mm/min. The force at which the ice column starts to be detached from the surface was measured as the  $\tau_{ice}$ . Additionally, the procedure of  $\tau_{ice}$  measurement was consecutively repeated once a day for icing/de-icing cycle tests.

**4.4. IDT Measurements.** The surface temperature of each sample was fixed to  $-10$  °C to measure the IDT. Then, a deionized water droplet of 7  $\mu$ L was placed on the sample surface. Subsequently, the freezing process of the deionized water droplet was photographed with a CCD camera. The IDT for water droplet freezing was determined by monitoring the transparency of the deionized water droplets with ImageJ program.

The surface temperature of a test sample was measured by installing thermocouples at four points on each sample surface (Figure S2). The aluminum plate coated with a test sample was placed on the cooling stage. The initial surface temperature of a test sample was fixed at 0 °C, and the temperature measurement started with a decreasing temperature of the cooling stage to  $-10$  °C at a constant rate. The temperature of each sample was measured twice, and eight data were averaged to obtain the surface temperature.

### ■ ASSOCIATED CONTENT

#### Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.2c01956>.

Optical image of SiOP and SiPDMS before and after placing blotting papers on the surface; schematic of the experimental setup for the surface temperature measure-

ment; temporal variations of the surface temperature of aluminum, SiPDMS, and SiOP (10 wt %); schematic of the experimental setup for measuring the ice adhesion strength; and optical image of the experimental setup used for ice adhesion strength measurements (PDF)

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### Author Contributions

All of the experimental concept design, method development, surface fabrication, characterization, data analysis, writing the manuscript, and theory consolidation were developed by S.J.L., and G.D.P. assisted the experimental setup, data analysis, and writing the manuscript. All authors assisted the experimental setup for icephobic performance tests, and S.J.L. supervised the whole project.

### Notes

The authors declare no competing financial interest.

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## ABBREVIATIONS

PDMS, polydimethylsiloxane; OP, oleamide–PDMS; SiPDMS, silicone oil-infused PDMS; SiOP, silicone oil-infused oleamide–PDMS; SHPo, superhydrophobic; SLIPS, slippery liquid-infused porous surface; LIP, liquid-infused polymer; FAA, fatty acid amide; WCA, water contact angle; IDT, icing delay time

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