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Cryo-FIB/SEM Investigation of Mechanism of Frost Formation on Lubricant-Impregnated Surfaces

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Frost formation and ice accumulation are major problems affecting variety of industries including transportation, power generation, and agriculture [1, 2]. Currently used active mechanical, thermal, and chemical methods of frost suppression and ice removal are time consuming and costly in operation. Consequentially development of passive methods preventing frost formation and ice accumulation is highly desirable. Hydrophobic surfaces have a high energy barrier for ice nucleation [3], low ice adhesion strength [2], and, if properly textured on the nano- and/or micro-scales, can repel impact of supercooled water droplets [4, 5]. However, hydrophobic as well as superhydrophobic surfaces lose their anti-icing properties due to frost formation [1, 6]. Recently, Kim et al.[7] reported that Lubricant-Impregnated Surfaces (LIS) consisting of nanotextured polymer coatings infused with perfluorinated oil have superior anti-frosting and anti-icing properties as compared to superhydrophobic surfaces. In addition, LIS have also been reported to have omniphobic [8], self-cleaning [9], and self-healing [8] properties.

In this work we used recently developed cryo-FIB/SEM methodology [10] to explore morphology of LIS before and after frosting in high humidity conditions. We first confirmed that LIS prepared using oil drop deposition and gravity induced draining procedure used by Kim et al. [7] contained about 8 µm thick excess oil film. This excess oil layer can easily break up due to perturbations such as air motion and can mask the underlying complexities of physicochemical principles of water drop mobility on LIS [11, 12]. In contrast, our study revealed that LIS prepared using systematic dip-coating procedure [13] have thermodynamically stable oil-solid morphology (see Fig.1 a & b). On such stable LIS, frost formation in high humidity conditions occurred via droplet condensation and subsequent freezing. As in previous reports [10, 11], we showed that drops condensed on top of the oil layer. However, we observed that after droplet freezing the oil fully drained from the substrate's texture (see Fig.1 c & d). We used cryo-FIB/SEM destructive tomography to show that the oil migrated to the surface of the frozen drop and filled-in the voids in-between nucleating nano-icicles (see Fig.1 e). We observed that the oil "cloaking" slowed, but did not prevent further growth of the dendritic nano-icicles. We showed that the frozen drops are in direct contact with the underlying solid substrate. As a result, frosting of cloaking LIS leads to degradation of their ice-repelling properties. Lastly, we used optimized *in situ* Environmental SEM [14, 15] to show that defrosting of the frosted cloaking LIS can lead to depletion of the oil and consequential loss of self-healing characteristics. Thus, further research is necessary to develop lubricant-nano/microtextured substrate pairs, which when properly combined, will provide a truly sustainable passive frost suppression method.

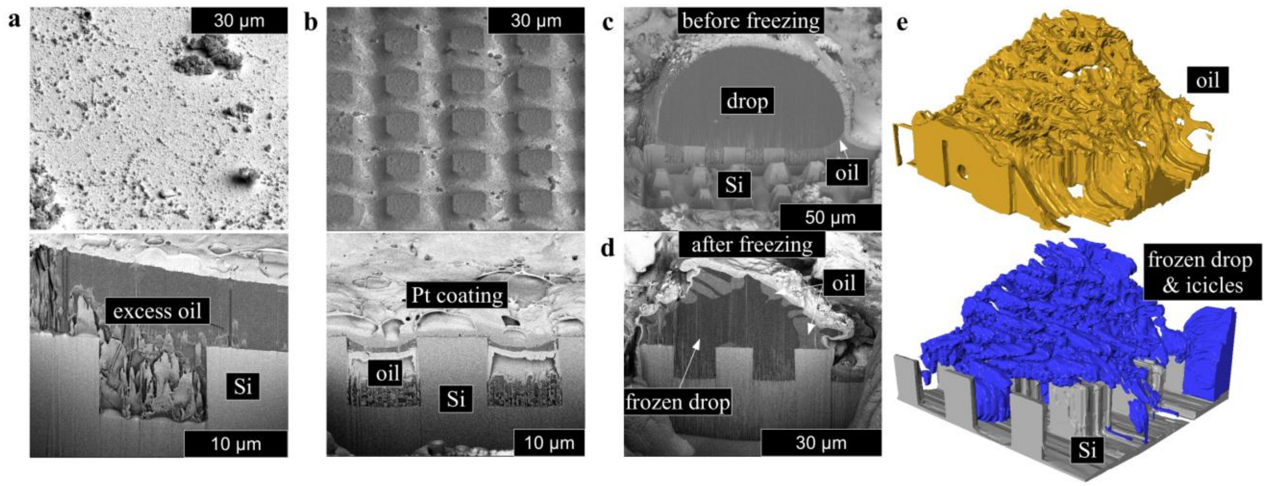

FIG. 1. cryo-FIB/SEM images of LIS prepared using (a) drop deposition and gravity induced draining and (b) dip-coating procedure; drops condensed on LIS (c) before and (d) after freezing, and (e) 3D reconstruction of drop frozen on LIS. The individual phases were segmented and separated to reveal the geometry of the frozen drop and nano-icicle network covered by the oil layer.

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