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Pillars or Pancakes? Self-cleaning surfaces without coating

Naureen Akhtar,1,3 Peter J. Thomas,2 Benny Svvardal,2 Stian Almenningen,1 Edwin de Jong,3
Stian Magnussen,4 Patrick R. Onck,3 Martin A. Fernø,1 Bodil Holst1*

1Department of Physics and Technology, University of Bergen, P.O. Box 7803, NO-5020, Bergen, Norway.
2Christian Michelsen Research AS, P.O. Box 6031, NO-5892, Bergen, Norway
3Zernike Institute for Advanced Materials, University of Groningen, Nijenborgh 4, NL-9747AG Groningen, The Netherlands
4ProAnalysis AS, P.O. Box 2619, NO-5836, Bergen, Norway
*Corresponding author: Bodil.Holst@uib.no

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ABSTRACT. Surfaces that stay clean when immersed in water are important for an enormous range of applications from ships and buildings to marine, medical and other equipment. Up till now the main strategy for designing self-cleaning surfaces has been to combine hydrophilic/hydrophobic coatings with high aspect ratio structuring (typically micron scale pillars) to trap a (semi-)static water/air layer for drag and adhesion reduction. However, such coating and structuring can distort optical properties; get damaged in harsh environments; and contamination, i.e. particles, oil droplets and biofouling, can get trapped and aggregate in the
structure. Here we present a radically different strategy for self-cleaning surface design: We show that a surface can be made self-cleaning by structuring with a pattern of very low aspect ratio pillars ("pancakes"). Now the water is not trapped. It can flow freely around the pancakes thus creating a dynamic water layer. We have applied the new pancake design to sapphire windows and made the first surfaces that are self-cleaning through structuring alone without the application of any coating. An offshore installation has now been running continuously with structured windows for more than one year. The previous uptime for unstructured windows was 7 days.

For a surface to be self-cleaning in water it must be underwater oleophobic i.e., the balance between the surface tensions must be such that the surface prefers to be wet by water rather than oil. It is crucial that the surface is underwater oleophobic, because this contributes to self-cleaning, not only for oil contamination, but also for bio-fouling; for example, the extracellular polymeric substances that create the structural integrity in bio-films are natural polymers of high molecular weight. Note that a superhydrophilic surface will be underwater oleophobic because the surface will be wetted by a water layer, which is immiscible with oil.

However, oleophobicity is not enough to ensure self-cleaning. Firstly it does not solve the problem of particle contamination and secondly even if the contact angle between the oil/polymer contaminant and the surface is high, the oil/polymer may still stick. If this is the case, even an oleophobic surface will eventually get contaminated. This is illustrated nicely by previous results on bare sapphire surfaces. It was shown here that a mechanically polished sapphire surface with micro scratches has a contact angle comparable to that of atomically flat. However, oil exposure tests showed that while an atomically flat surface stays clean under water for a few hours, a scratched surface contaminates rapidly: The reason being that the water cannot
flow freely through the scratches and remove the oil droplets, so in the scratches the oil droplets stick and act as nucleation sites.

It is well known that surface structuring may lead to an increase in contact angle by reducing the interface area between water droplets and surface with air in between the structures or, for the underwater case, oil droplets and surface with water layer at the interface; this is referred to as the Cassie-Baxter state.\textsuperscript{10} Up till now the strategy for using this in self-cleaning has been to make micron scale, high aspect ratio surface structures (height/width >1) with additional nano-structuring and/or coating (usually hydrophobic coating for self-cleaning in air and hydrophilic coating for underwater case) on top.\textsuperscript{11-13} Above water this is known as the famous lotus effect. In water, a (semi-)static water layer is trapped in between the structures, which reduces drag. This is essentially the principle of fish scales, where a hydrophilic coating is combined with the rough structure of the scales (height variations in the order of 10 \textmu m).\textsuperscript{12} However, surfaces with high aspect ratio structures pose major challenges to long term self-cleaning underwater due to aggregation of particles and oil droplets over the longer periods of time, loss of optical transparency and reduced robustness.

Recently it was shown that for superhydrophobic surfaces in water, the presence of an air/vapour layer at the solid-water interface leads to an increased drag reduction and hence improved self-cleaning.\textsuperscript{14-16} However, in order to keep the desired low-drag properties, the air/vapour layer needs to be replenished frequently, for example by heating the surface.\textsuperscript{17-18}

Here we present a radically different strategy for creating a self-cleaning surface. Instead of using a high aspect ratio structure, which traps a (semi-)static water layer, we use a low aspect ratio structure, consisting of flat pillars (“pancakes”) whose height has been deliberately reduced so that instead of being trapped between tall pillars, the water can flow easily in-between the flat
pancakes. We call it the ‘dynamic water-layer’ approach. We present experiments on sapphire surfaces. Sapphire (Al₂O₃) is the second hardest material in the world after diamond and widely used for windows in harsh environments. This also makes sapphire surface processing very challenging. Previous efforts on sapphire surface patterning and etching are described in the Supporting Information.

To the best of our knowledge we present here the first demonstration of self-cleaning solely through a structuring of the substrate surface, without the application of an additional coating or hierarchical structures. The strategy can of course also be applied to coated surfaces. The idea of letting the water flow efficiently in between the structures on the surface can be compared to the principle of drag-reduction for shark skin, but we are working here on a different scale and with a different structure. We show that our simple pancake approach leads to a radical self-cleaning effect. The pancake approach is particularly suitable for applications in optical technology because the optical scattering caused by wall roughness and non-vertical sidewall profiles is not an issue that needs to be addressed. Furthermore the pancake approach is robust because the pancakes are less prone to breaking than high aspect ratio pillar of the same diameter would be. Finally pancake structures on the micron scale can be mass produced comparatively cheaply in a huge range of materials (i.e. with nanoimprint and etching). This is particularly relevant for sapphire where it is very difficult to make high aspect ratio pillar structures with controllable sidewall profile.

Figure 1 shows one of our structured sapphire surfaces. The preparation method is described in the Materials and Methods section. We kept the lateral pancake dimension (d) fixed at 5 µm. This gives the desired self-cleaning properties with minimum impact on optical properties. The height (h) was varied between 200 nm and 1 µm and the pitch (p) between 8 and 17 µm.
Figure 1. The “pancakes”. Scanning electron microscopy images of a structured sapphire surface with $d/p/h$ as 5 $\mu$m/8 $\mu$m/600 nm. The left and right images are taken at a 0° and 45° sample tilt, respectively. Images are taken before removal of the etching mask layer.

We compared the optical transmission of structured and unstructured samples in the range 300-1000 nm. Only a slight reduction in transmission was observed for $h$ variation between 200 and 600 nm as shown in Figure 2. However, in case of non-vertical and rough walls, a considerable loss can be expected for high pillars. As shown in Figure 2 the transmission reduces by up to 60% already for a sapphire window with a “thick pancake” structure with $h=1$ $\mu$m. Since sapphire is an extremely difficult material to etch due to its remarkable hardness and chemical resistance, it is particularly difficult to make vertical wall profiles in sapphire (see the Supporting Information).

The sapphire windows were mounted in a custom-made test vessel and subjected to self-cleaning tests using an oil-based contamination environment that is similar to that in subsea pipelines. The contamination mixture consisted of water with i) oil droplets: 1000 ppm of crude oil from the Norwegian sector (Troll B) the droplet diameter varied between 5-1000 $\mu$m and ii) particles: 1000 ppm of sand and 1000 ppm calcium carbonate scales. The flow speed was 1.2 m/sec parallel to the square stacking direction.
Figure 2: Transmission spectra measured for the as-received, chemically cleaned sapphire and for the structured sapphire surfaces with fixed $d = 5 \, \mu m$ and various combinations of $p$ and $h$ ($p/h$). Note the dramatic decrease in transmission for $h = 1 \, \mu m$.

The most remarkable results from the self-cleaning tests are displayed in Figure 3, where transmission images of three sapphire windows can be seen after 46 hours exposure to contamination. The top row, panels (a), (b) and (c) show structured samples (with 15 $\mu m$/ 200 nm, 10 $\mu m$/ 600 nm and 17 $\mu m$/600 nm respectively as $p/h$) while panel (d) shows a chemically cleaned but unstructured sample (see the Experimental section). The unstructured sample is strongly contaminated compared to the structured samples. Panels (a1) to (d1) in Figure 3 shows how the adhesion decreases strongly as a result of structuring; the flow was stopped after 46 hours and then immediately started again to generate a transient flow disturbance close to the surface. As a result, the oil contamination reduces significantly for the two structured windows with lower pitch (a1 and b1). It disappears partly from the window with highest pitch (c1) and remains unchanged on the unstructured window (d1).
Figure 3. Build-up of contamination on sapphire windows exposed to the contamination mixture for 46 hours. Top row: Images taken after 46 hours exposure to the contamination mixture for sapphire windows with surface structures having $d/p/h$: a) $5 \mu m/15 \mu m/200 \text{ nm}$ b), $5 \mu m/10 \mu m/600 \text{ nm}$ and c) $5 \mu m/17 \mu m/600 \text{ nm}$, d) As-received, chemically cleaned sapphire window
shown as reference. Bottom panels (a1-d1): corresponding images taken after generating transient flow disturbance in the test vessel containing the contamination mixture. The windows are 12.7±0.1 mm in diameter. (e) A statistical analysis of the build-up of contamination over time on the corresponding sapphire surfaces. The last data points highlighted in blue show reduction in contamination after generating transient flow disturbance in the test vessel.

A statistical analysis of the build-up of the contamination over time is shown in Figure 3 (e). The experiment was repeated 6 times over a period of 3 months and showed the same results in all cases. The overall trend shows an increase in the area covered by particles with increasing time. The local fluctuations in the data are arising from the complex process of fouling of the windows, which include contamination attachment and detachment over time in the presence of flow and deposit of new contamination on previously deposited contamination. The results from Figure 3 indicate minimum build-up of the contamination for surface structures with a pitch of around 10 μm. To investigate further the role of surface structures versus height, experiments were also done on a structured sapphire surface with \( p/h \) 10 μm/200 nm. After 46 hours of exposure to the contamination mixture, 27.0±1.0% area was covered with contamination. This reduced to 12.0±1.5% after the transient flow disturbance previously described. As can be seen from figure 3e the performance of the 10 μm /600 nm surface is much better than this. Hence for our particular test settings, we obtain the overall best anti-fouling and self-cleaning performance for 10 μm/600 nm \( (p/h) \) sample. This is presumably because for the smaller heights and/or larger pitches, bigger oil drops are able to come into contact with the substrate in-between the pancake structures through sagging.

After these contamination measurements, a structured window was installed in an offshore installation, where it is used as part of an optical sensor that measures the contamination level of
water pumped back into the reservoir. In this installation it would typically be necessary to clean
the window manually after 7 days, a very costly procedure since it involves stopping the
production. The structured window has now been running continuously for more than one year
without the need for manual cleaning.

The contamination measurements in the lab and in a real offshore installation show that the
structuring leads to a strong self-cleaning effect. To investigate this further we carried out a
series of static and tilted underwater contact angle measurements on oil droplets.
The results in the Supporting Information show that all structured sapphire surfaces are
oleophobic with static contact angles of more than 130° (see Figure S2). For the pancake
diameter and pitch range studied here, the underwater oil wetting behaviour is expected to be in
the Cassie-Baxter state for height > 200 nm as shown in Figure S2 and Equation 2 in the
Supporting Information. The as-received sapphire surface is hydrophilic in air with a contact
angle of ~ 85° and underwater oleophilic with a contact angle of 74°±2°. Chemical cleaning
removes any carbon-based contamination leaving behind a clean oxide surface that is
superhydrophilic in air with a water contact angle < 20°. As expected the clean surface is also
measured to be underwater oleophobic with a contact angle of 120°±2°. After exposure to air for
a few days the surface gets contaminated again and returns to the original state.²⁵,²⁶ The
structured surfaces also get contaminated just as the as-received surface when stored in air. Even
so these surfaces, when submerged in water, exhibited the same oleophobicity with contact
angles of 130° or more, even after being stored for several weeks in air. This is due to the surface
structuring that leaves the surface in the Cassie-Baxter state for oil droplets underwater
regardless of carbon-based contamination.
Tilted-drop measurements (Figure 4 and movie S1 and S2) show that an oil droplet with a volume of 15 µl slides off the structured surface at 20° inclination, while it remains adhered to the as-received sapphire surface at an inclination as high as 85°. Droplets of smaller volume do not adhere to the structured surfaces even after being pushed onto the surface for at least eight hours (see movie S3). This is a further demonstration of the very low adhesion on the structured surfaces. Smaller droplets adhered readily to the chemically cleaned, as-received surface (see movie S4).

Figure 4. Static underwater contact angle measurements for oil droplet. (a) A 15 µl oil droplet underwater on as-received sapphire after chemical cleaning treatment. (b) A 15 µl oil droplet underwater on a structured sapphire surface with 5 µm/15 µm/200 nm as d/p/h. Left panels show the droplets at 0° sample inclination. The right panels illustrate the movement of the underwater oil droplets on inclined samples. The oil droplet slips off the structured surface at a 20° inclination while a similar oil droplet remains static on the cleaned as-received surface at an inclination as high as 85°. Red marks are given as guides to the eye.

From the fact that the structured surfaces are in the Cassie-Baxter state and prevent adhesion in the presence of flow, we conclude that the presence of a dynamic water layer in-between the
pancakes, makes the contamination slip away more readily. This is confirmed by a numerical
modelling. Figure 5 shows a simulation of the water flow distribution between the surface
structures at a height of 100 nm above the bottom surface of the flow tube (see the Experimental
section for details on the numerical modeling). The water flow velocity between the pancakes
(200 and 600 nm for $d/p$ 5 μm/15 μm) is significantly higher than between the high aspect ratio
pillars despite the high flow velocities close to the pillar tops in all structured surfaces (Figure
S3 in the Supporting Information). This indicates that surfaces with high aspect ratio structures
may show good self-cleaning behavior initially when submerged underwater due to the reduced
adhesion and drag. However, the decrease in flow velocity in between the 10 μm high pillars
when moving down close to the bottom ($h < 5\mu m$) means that oil droplets and particles with
diameters smaller than the interpillar distance can get trapped and aggregate in between the high
aspect ratio structures over time. Furthermore higher pillars with rough wall profile provide
larger surface area for the nucleation and agglomeration of contamination particles over time. In
case of low aspect ratio structures, particles are less likely to aggregate due to the higher water
flow close to the bottom and the smaller wall surface area.

As mentioned above the contact angle between the as-received, chemically cleaned surface and
the structured surfaces only differs by about 10° (see Figure 4 and the Supporting Information).
They are all oleophobic underwater, but only the structured surfaces display self-cleaning in the
presence of flow.
Figure 5. Flow velocity distribution. Numerical model showing the flow velocity distribution at a height of 100 nm above the bottom surface of the flow tube for unstructured surface (top left), 200 nm high pancakes (top right), 600 nm high pancakes (bottom left), 10 μm high pillars (bottom right). All structured surfaces are fixed at 5 μm/15 μm as d/p. Water flows from bottom to top in each image with a flow velocity of 1.2 m/s similar to what was used in the real contamination experiments. The velocity in between the surface structures is higher for the pancakes i.e. 19.5 times and 4.5 times higher at the white and black marks respectively for the 200 nm pancakes compared to the 10 μm pillars.

We do not see trapping of contamination in the structures over time such as it has been suggested for high aspect ratio structures with static water layers.27 This is as expected because the water can flow freely in between the pancakes. However, as it is seen through the difference in the experiments for the 200 and 600 nm pancakes; contamination does increase if the pillar height ("pancake thickness") is too low, presumably because large droplets can more easily sack...
and stick between the pancakes. Adhesion is still very low, as demonstrated by the almost complete removal of contamination in Figure 3 (a1).

In summary, we have demonstrated a new strategy for creating self-cleaning surfaces. We show that a low aspect ratio surface structure ("pancakes") facilitates a dynamic water layer that is continually replenished in the presence of flow. This can be an advantage compared to surfaces with high aspect ratio structures that are self-cleaning through trapping of a (semi-)static water layer which gives drag and adhesion reduction on top of the pillars, but slow down the flow speed strongly in between the pillars as shown in Figure 5 and Figure S3. The self-cleaning property of such high aspect ratio surfaces may therefore be prone to long term degradation due to aggregation of contamination in between the pillars. The pancakes design has the additional advantage of being more robust (the pillars break more easily) and optically transparent. The optimum pancake structuring for a given application will be determined by a combination of the contamination type and size distribution, the interfacial energies and the water flow properties (speed, type and directionality). This will be the topic of further investigations.

Experimental section

Surface structuring of the sapphire samples. Sapphire crystals were purchased from Freudiger with diameter between 12.67−12.73 mm (with bevel edges of 45°, 0.2 mm), thickness between 1.55−1.60 mm and crystal miscut specified as less than 30° relative to the Z-axis. The crystal surfaces were chemically polished with a Scratch/Dig number of 40/20. Standard photolithography was employed to pattern the sapphire surfaces, involving the following steps: (a) spin coating of photoresist (AZNLof2000 from Microchemicals) on the surface of the substrate; (b) UV exposure through a photomask to pattern the photoresist layer; (c) removal of the photomask and development of the photoresist to produce a patterned photo-resist layer; (d)
deposition of an etching mask (Cr layer); (e) removal (lift-off) of the remaining photoresist; (f) inductively coupled plasma etching (Oxford Plasmalab System 100) of the sapphire substrate using the etching mask as an etching template\textsuperscript{24}; and (g) removal of the remaining etching mask layer using Cr etchant 1020 purchased from Transene Company, Inc. A stylus profilometer (Veeco DEKTAK 150) was utilized to confirm the heights of the surface microstructures.

**Contact angle measurements.** A video-based optical contact angle measurement system, OCA20 LHT, from Dataphysics with SCA software (version 4.3.19) was used to measure contact angles of oils and water on sapphire windows. The system is equipped with an electronic tilting base unit TBU 90E that allows software controlled inclination of the instrument up to an angle of 90° with accuracy of ±0.1°. For measurements of the water contact angle in air, a water droplet of about 2 µl was directly placed on the sapphire surface. For oil contact angle measurements in water, an oil droplet (hexadecane) having a volume varying between 3-15 µl was gently deposited from the bottom of the system onto the sapphire window surface, which was submerged in water.

**Chemical cleaning.** Surface cleaning treatment involves the following steps: (a) soaking of samples in a 3:1 solution of H\textsubscript{2}SO\textsubscript{4} and H\textsubscript{2}O\textsubscript{2} for 20 min at 80 °C (b) soaking of samples in a 1:1:5 solution of NH\textsubscript{3}, H\textsubscript{2}O\textsubscript{2}, and water for 20 min at 80 °C (c) soaking of samples in a 1:1:5 solution of HCl, H\textsubscript{2}O\textsubscript{2}, and water for 20 min at 80 °C. All three steps were followed by rinsing with ultrapure ion free water with a resistivity greater than 18 MΩ-cm and drying with nitrogen stream.

**Contamination tests.** For long-term surface contamination and self-cleaning tests of the sapphire surfaces, a test setup was designed in house. The images are taken through the sapphire window so that the contaminated surface is viewed from the back. The experimental set-up is
described in detail in reference 9. The image analysis was done using the freeware ImageJ software developed at the National Institutes of Health, Bethesda, Maryland. For statistical analysis, data from 3 different experiments were analysed (a total of 280 images were used in the analysis).

**Numerical modeling.** The numerical modeling was conducted with COMSOL Multiphysics 5.3. The model consists of a quadratic pattern of 5×5 pillars with diameter of 5 μm and pitch of 15 μm. The height is varied between 0, 200 nm, 600 nm and 10 μm. These pillars are placed at the bottom of a rectangular flow tube with width/height/length as 90 μm/40 μm/180 μm. Both the inflow and outflow velocity are set to 1.2 m/s, similar to what has been used in the experiments. A no-slip boundary condition is applied between flowing water and the bottom surface and pillars. The flow regime is assumed to be laminar.

ASSOCIATED CONTENT

**Supporting Information.** Figure S1 showing the optical images of the structured surfaces. Figure S2 showing the static underwater contact angles for an oil droplet and Figure S3 showing the calculated flow velocities as a function of pillar height for structured sapphire surfaces. Movie S1 and S2 showing the tilted-drop measurements on the structured sapphire and the cleaned as-received sapphire surface respectively, and movie S3 and S4 showing the deposition of an oil droplet on the structured sapphire and the cleaned as-received sapphire surface respectively.

AUTHOR INFORMATION

**Corresponding Author**

*Email: Bodil.Holst@uib.no*
Author Contributions

N.A. made the samples and performed the contact angle experiments, N.A. and B.H. planned the experiments and wrote the manuscript with contributions from all the authors, B.S. and P. J. T designed and constructed the setup for testing contamination and P.J.T. and N.A. did the contamination experiments. S.M. performed the offshore tests. All authors discussed the results.

Notes

A patent application, owned by the University of Bergen, has been submitted on the basis of this work.

ACKNOWLEDGMENT

We thank Petra Rudolf, Gerrit Zijlstra, Jacob Klein, Lars Egil Helseth and William Allison for useful discussions. This project was supported by the Norwegian Research Council Project No. 217233/E30. We thank the Bergen Research Foundation for supporting the foundation of the UiB Nanostructures laboratory.

REFERENCES


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as-received  Pancake design

Underwater self-cleaning window
The "pancakes". Scanning electron microscopy images of a structured sapphire surface with $d/p/h$ as 5 μm/8 μm/600 nm. The left and right images are taken at a 0° and 45° sample tilt, respectively. Images are taken before removal of the etching mask layer.

30x11mm (300 x 300 DPI)
Transmission spectra measured for the as-received, chemically cleaned sapphire and for the structured sapphire surfaces with fixed $d = 5 \, \mu m$ and various combinations of $p$ and $h$ ($p/h$). Note the dramatic decrease in transmission for $h = 1 \, \mu m$.

83x63mm (300 x 300 DPI)
Build-up of contamination on sapphire windows exposed to the contamination mixture for 46 hours. Top row: Images taken after 46 hours exposure to the contamination mixture for sapphire windows with surface structures having \(d/p/h\): a) 5 µm/15 µm/200 nm b), 5 µm/10 µm/600 nm and c) 5 µm/17 µm/600 nm, d) As-received, chemically cleaned sapphire window shown as reference. Bottom panels (a1-d1): corresponding images taken after generating transient flow disturbance in the test vessel containing the contamination mixture. The windows are 12.7±0.1 mm in diameter. (e) A statistical analysis of the build-up of contamination over time on the corresponding sapphire surfaces. The last data points highlighted in blue show reduction in contamination after generating transient flow disturbance in the test vessel.
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65x50mm (300 x 300 DPI)
Flow velocity distribution. Numerical model showing the flow velocity distribution at a height of 100 nm above the bottom surface of the flow tube for unstructured surface (top left), 200 nm high pancakes (top right), 600 nm high pancakes (bottom left), 10 μm high pillars (bottom right). All structured surfaces are fixed at 5 μm/15 μm as d/p. Water flows from bottom to top in each image with a flow velocity of 1.2 m/s similar to what was used in the real contamination experiments. The velocity in between the surface structures is higher for the pancakes i.e. 19.5 times and 4.5 times higher at the white and black marks respectively for the 200 nm pancakes compared to the 10 μm pillars.

124x81mm (300 x 300 DPI)
**Underwater self-cleaning window**

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34x19mm (300 x 300 DPI)