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Inclined-wall regular micro-pillar-arrayed surfaces covered entirely with an alumina nanowire forest and their improved superhydrophobicity

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Abstract
This paper reports a multiple-scale hierarchically structured superhydrophobic surface that is composed of inclined-wall regular micro-pillar arrays covered entirely with an alumina nanowire forest (ANF) to improve the surface wettability. The multiple-scaled structures were fabricated stably using a simple batch process based on an anisotropic chemical silicon etching process and a subsequent time-controlled anodic aluminum oxide technique. The surface wetting properties of the mono-roughened surfaces with inclined-wall micro-pillar arrays, which are normally in the Wenzel wetting regime, could be transitioned perfectly to the slippery Cassie mode and enhanced greatly in the Wenzel regime in cases of a high- and low-density of the micro-pillars, respectively, by easily amplifying the intrinsic contact angle through the entire coverage of the ANF on the micro-roughened surfaces. The wettability of the proposed multiple-scaled surfaces could also be predicted using analytic surface models and the experimental results agreed greatly with the wetting trends estimated theoretically due to the geometrical regularity of the base micro-structures.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

A water droplet placed on a lotus leaf can roll off the surface easily, collecting dust and other contaminants on the surface, even when the surface is tilted only slightly. This attractive ‘self-cleaning’ property (so-called ‘Lotus effect’) of the lotus leaf has been a major research topic for industrial applications. Therefore, to date, a number of studies have been carried out to demonstrate an artificial superhydrophobic surface with the maximal contact angle (CA, $>150°$) and minimal contact angle hysteresis (CAH, $<10°$), mimicking the lotus leaf in a range of approaches [1, 2]. The superhydrophobicity of such surfaces can be obtained from the increase in surface roughness and decrease in surface energy. In particular, it has been proven experimentally and theoretically that the double-scale roughness of a surface can improve the surface hydrophobicity considerably compared to surfaces with mono-roughness

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[3, 4]. Therefore, recent research has concentrated mainly on demonstrating micro/nano double-roughened surfaces [5–18]. Most frequently, vertical-wall micro-pillar structures decorated with nano-roughness have been used to realize the engineered superhydrophobic surface because well-designed micro-pillar structures with a moderate aspect ratio generally exhibit good hydrophobic properties in slippery mode (Cassie state). Moreover, the addition of nano-roughness on the micro-pillar structures can lead to a further increase in CA and a decrease in CAH of the surface [5–13]. However, nano-roughness is normally found only on the top surfaces of the micro-pillars in most cases of hierarchical approaches based on vertical-wall micro-pillar arrays [5–11]. Although Kwon *et al* and Cha *et al* demonstrated superhydrophobic surfaces with vertical-walled micro-pillar arrays covered entirely with nano-roughness, which was formed by an isotropic plasma treatment with specific etching ions [12, 13], nano-roughness covering the micro-pillars can be produced only on silicon-based structures. Therefore, the applicability of the surfaces is quite restricted. The limitation of the conventional approaches based on the vertical-walled pillars comes mainly from the difficulty in fabrication compatibility between the different size regimes. Alternatively, micro-scale structures with inclined sidewalls (not vertical), such as pyramid and horn structures can also be used to produce the micro/nano hierarchically structured surfaces. Although they are less effective for improving the hydrophobicity of a surface in Cassie mode compared to vertical-wall micro-structures, the inclined walls of these micro-structures are more desirable for structuring the additional different scale roughness entirely, resulting in the easy integration of nano-roughness to various micro-structures. Xiu *et al* reported a micro/nano hierarchical superhydrophobic surface with a CA larger than 150° and a CAH less than 5°, based on inclined-wall micro-structures with nano-roughness generated by the metal-assisted etching process [14]. Kim *et al* presented a dual-scale structured polymeric surface using a sandblasted porous alumina template and a PTFE-replication process. The CA of the fabricated dual-scale surface was 165°, which is superior to that of the mono-scale micro surface [15]. Although they could improve the surface hydrophobicity by increasing the intrinsic contact angle (ICA) of the surface with nano-roughness formed entirely on the micro-pillar structures, specific experimental conditions must be imposed to fabricate the dual-scale roughness and a theoretical evaluation of the surface hydrophobicity is very difficult due to the structural ambiguity of the base micro-structures.

This paper reports a multiple-scale hierarchical approach to demonstrate a superhydrophobic surface that can enhance greatly the surface wetting property by simply amplifying the ICA of the base surface. The proposed hierarchical surface consists of inclined-wall regular micro-pillar arrays covered entirely with the alumina nanowire forest (ANF) and can be fabricated efficiently using a simple combination of the anisotropic chemical silicon etching and time-controlled aluminum oxide (AAO) processes. The significances of the proposed approach can be claimed as follows: first, perfect transition of the wetting state of the inclined-wall micro-pillar-arrayed surface to the slippery Cassie mode can be achieved by amplifying remarkably the intrinsic CA through entire formation of the ANF based on a simple nonlithographic process. In addition, the ANF can also be applied on various structures with inclined sidewalls and etchable materials as a base surface with increased intrinsic CA, as well as on the silicon substrate, to enhance the surface wetting property because the ANF can be formed on the all aspects of the inclined-walled structures conformally and uniformly. Second, the proposed hierarchical surface can be realized stably and reproducibly ensuring uniform wetting behavior due to well-established microfabrication and AAO technologies. Third, theoretical estimation of the surface wettability and theory-based fine surface design are achievable due to the geometrical regularity of the micro-pillar array. These advantages of the proposed hierarchical approach can provide feasibility to increase its applicability. The effectiveness of the proposed hierarchical surface on the enhancement of the surface wetting property was evaluated experimentally and theoretically.

2. Design

To obtain a surface with ‘self-cleaning’ properties, the surface wetting property must be maintained in the Cassie regime to ensure a minimal CAH, which means there is a sufficient amount of air between the water droplet and surface, as well as the maximal CA. In particular, in the case of an inclined-walled micro-structured surface, as shown in figure 1, the inclination angle (β) of the top corner of the structure must be smaller than the ICA on the flat surface to satisfy the Cassie state [19]. This is a serious limitation of inclined-wall structures to obtain a slippery surface, even though they can be realized in a cost-effective manner and be more stable mechanically compared to the vertical-wall pillar structures. In general, the periodic inclined-wall silicon micro-pillars can be fabricated by etching a {1 0 0} silicon substrate anisotropically using a tetramethylammonium hydroxide (TMAH) solution. In this case, the inclination angle (α) of the bottom corner is always 54.74° due to the different etch rates depending on the crystalline planes of silicon. As a result, the inclination angle of the top corner of the pillar structure is fixed at 125.26° accordingly. This value acts as a critical CA (θ_C) between the Wenzel and Cassie states. Therefore, the inclined-wall micro-structured surfaces will always be placed in the Wenzel state because the ICA of the flat surface after the low-energy surface treatment is usually less than 120°. In the present experiments, the ICA measured on the flat surface after a low-energy surface coating using a plasma polymerized fluorocarbon (PPFC) was 115.2 ± 0.9°, which is also much smaller than the critical CA. Figure 2 shows the estimated surface wettability of the inclined-wall micro-pillar array due to ICA variations. In this case, the length of the top-square, the height and the period of the micro-pillar were fixed to 50, 20 and 100 μm, respectively.

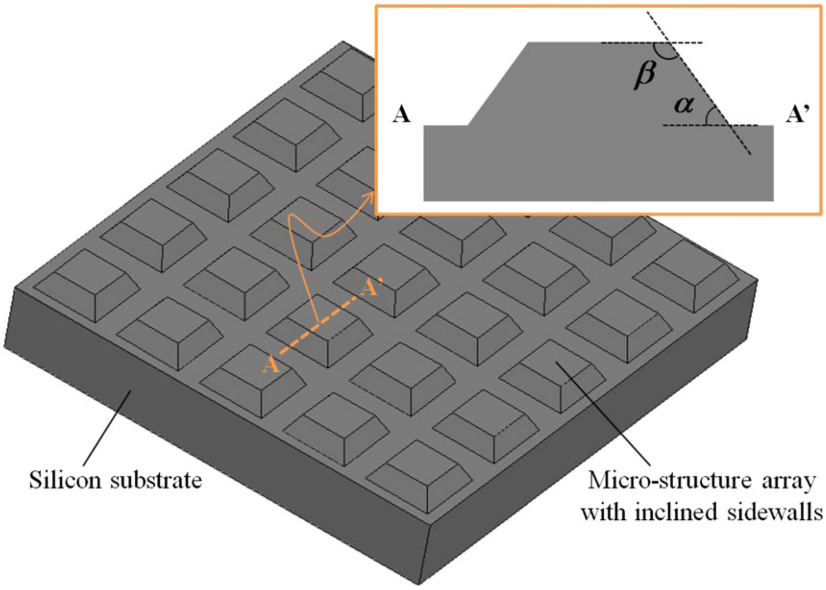


Figure 1. Schematic view of the inclined-walled micro-pillar arrayed surface.

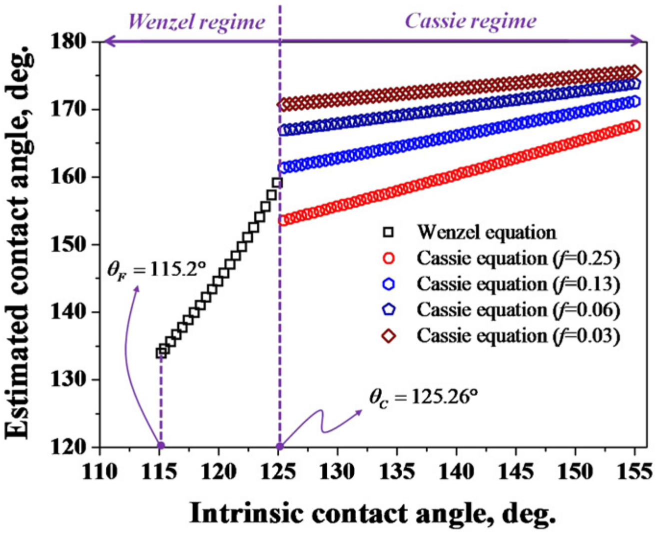


Figure 2. Estimated CAs in the Wenzel and Cassie states due to the ICA variations.

When the ICA of the surface is smaller than the critical CA, the surface wettability can be estimated using the following Wenzel equation:

$$\cos\theta^W = r\cos\theta_F \qquad (1)$$

where θ^W and r are the CA (in the Wenzel state) and roughness factor of the micro-roughened surface, respectively, and θ_F is the intrinsic CA of the flat surface.

On the other hand, when the intrinsic CA of the surface is larger than the critical CA, the CAs of the surface can be calculated using the Cassie equation:

$$\cos\theta^C = f(1+\cos\theta_R) - 1 \qquad (2)$$

where θ^C, f and θ_R are the CA (in the Cassie state), solid fraction and amplified intrinsic CA of the micro-roughened surface, respectively.

The transition between the Wenzel and Cassie states can occur with an ICA larger than 125.26°. In addition, a further increment of the ICA can enhance the superhydrophobicity of a micro-roughened surface, as shown in figure 2. Therefore, the efficient amplification of the ICA is essential for improving the wetting property of a surface with an inclined-wall micro-pillar array. A multiple-scale hierarchical surface was proposed in this paper to transition the Wenzel to Cassie mode efficiently by amplifying the ICA. The proposed hierarchical surface basically consists of an inclined-wall micro-pillar array, and the ANF is covered entirely on the micro-structured surface, as shown in figure 3. Because the surface covered with the ANF contains sufficient air pockets to support a water droplet, the increase in CA in the Cassie regime can be achieved more easily by minimizing the liquid/solid contacting area compared to a flat surface, which was already proven experimentally in our previous work [20]. Therefore, the ICA of the micro-structured surface can be amplified efficiently, resulting in an easy and complete wetting transition to slippery mode. Moreover, the surface wettability can be also expected and characterized theoretically because the entire formation of a nanowire forest is achieved on a micro-roughened surface without geometrical changes in the base micro-structures.

3. Fabrication

3.1. Fabrication of inclined-wall micro-pillars

The inclined-wall silicon micro-pillar arrays were basically formed on a {1 0 0} silicon substrate by the anisotropic chemical etching based on the TMAH. Although the TMAH etchant is used widely to produce V-grooves and cavities with inclined sidewalls and sharp corners on the {1 0 0} silicon substrate, it is impossible actually to make the inclined-wall pillar-like structures owing to a rapid undercutting rate at sharp convex corners. On the other hand, it was reported that the

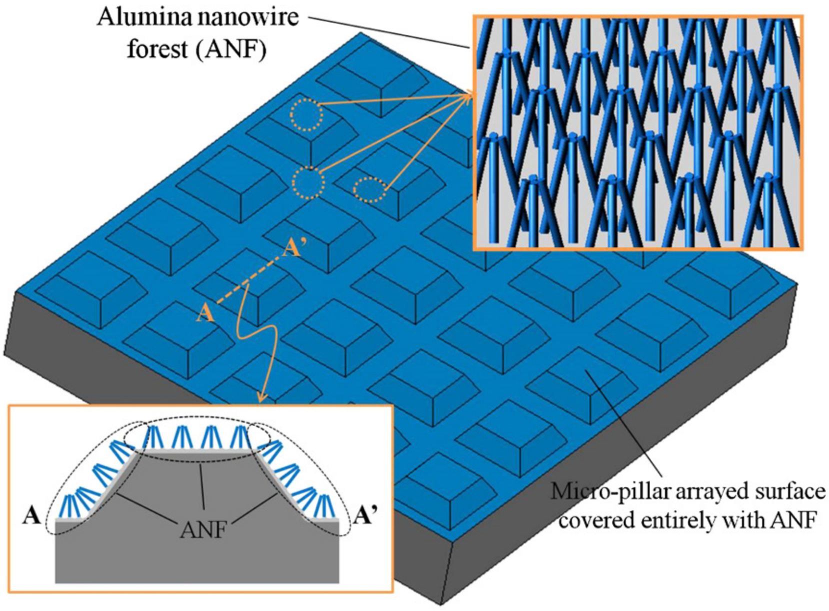
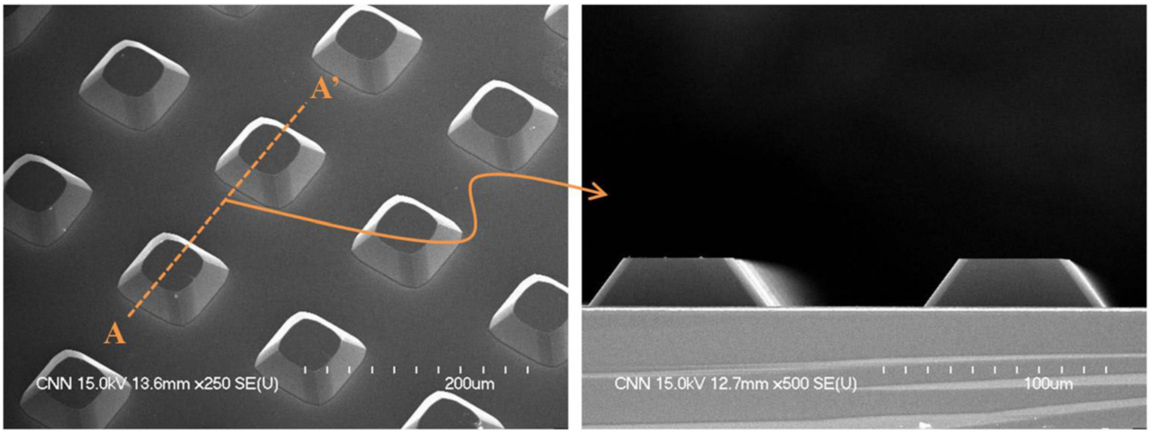


Figure 3. Perspective view of the proposed multiple-scale hierarchical surface.



(*a*) (*b*)

Figure 4. SEM images of the fabricated inclined-wall micro-pillars. (*a*) Arrayed view and (*b*) side view.

rapid etch rate at the sharp convex corners can be alleviated and any shapes of the structures can be demonstrated by adding some amount of surfactant to the TMAH etchant [21]. In the present study, 25 wt% TMAH mixed with a 0.1 vol.% surfactant (Triton X-100, $C_{14}H_{22}O(C_2H_4O)_n$) was prepared to fabricate a surface with densely arrayed inclined-wall pillar structures on the silicon substrate. The fabrication process begins with an oxidized 4 inch silicon substrate with a {1 0 0} crystalline orientation. Initially, a 0.3 μm thick silicon dioxide etching mask was patterned using standard photolithography and subsequent reactive ion etching (RIE) process. The silicon substrate was then etched anisotropically with a silicon dioxide etching mask in the prepared chemical mixtures at a temperature of 80 °C. Although the etch-rate in the vertical direction (approximately, 0.33 μm min^{-1}) becomes slower than that in TMAH without the surfactant, the square patterns including four sharp convex corners were almost maintained without serious geometrical changes. Therefore, the inclined-wall micro-structured surfaces were fabricated stably, as shown in figure 4.

3.2. Fabrication of multiple-scale hierarchical surface

The fabrication procedure of the proposed multiple-scale hierarchical surface is shown in figure 5. Initially, a 2 μm thick aluminum (Al) layer was deposited by a sputtering process on the prepared inclined-wall micro-structured surface (figures 5(*a*) and (*b*)). The ANF was formed simply on the micro-structured surface using the time-controlled AAO technology. For this, the sputter-deposited Al layer was anodized in a 0.3 M $C_2H_2O_4$ aqueous acidic solution under a constant voltage and temperature of 45 V and 10 °C, respectively, for 10 min. After the anodization process, a porous alumina layer including periodic pores with an initial diameter of approximately 20 nm was formed (figure 5(*c*)). The pore diameter was then widened by etching the grown alumina in 5 wt% H_3PO_4 acidic etchant at 30 °C. In this case,

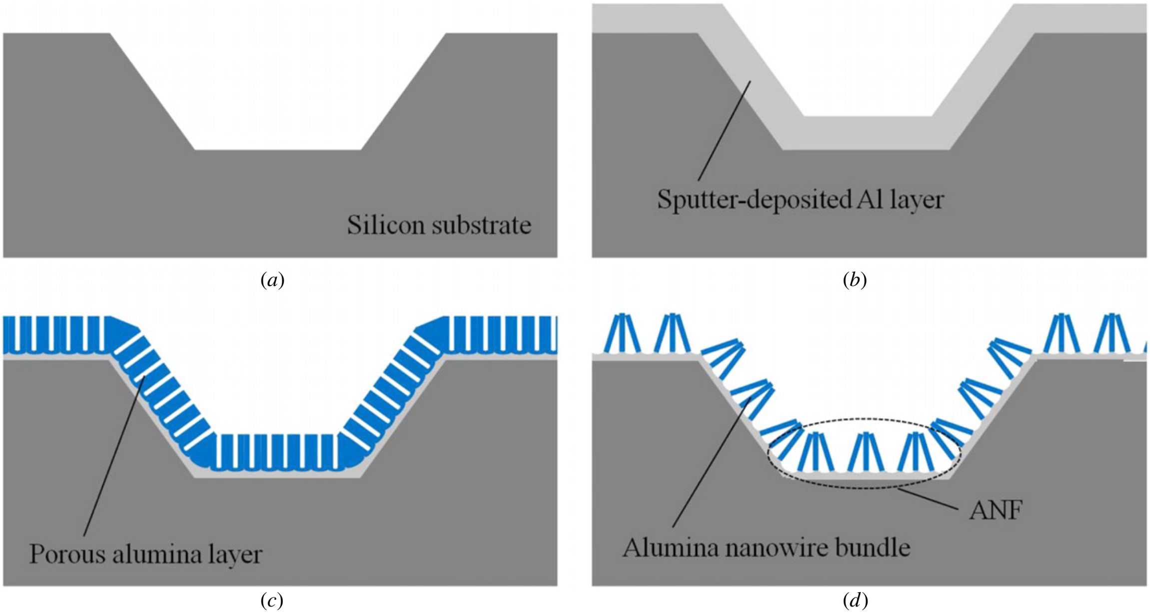


Figure 5. Fabrication process. (*a*) Anisotropic chemical etching of silicon, (*b*) aluminum deposition, (*c*) aluminum anodization and (*d*) pore-widening and subsequent formation of alumina nanowire bundles.

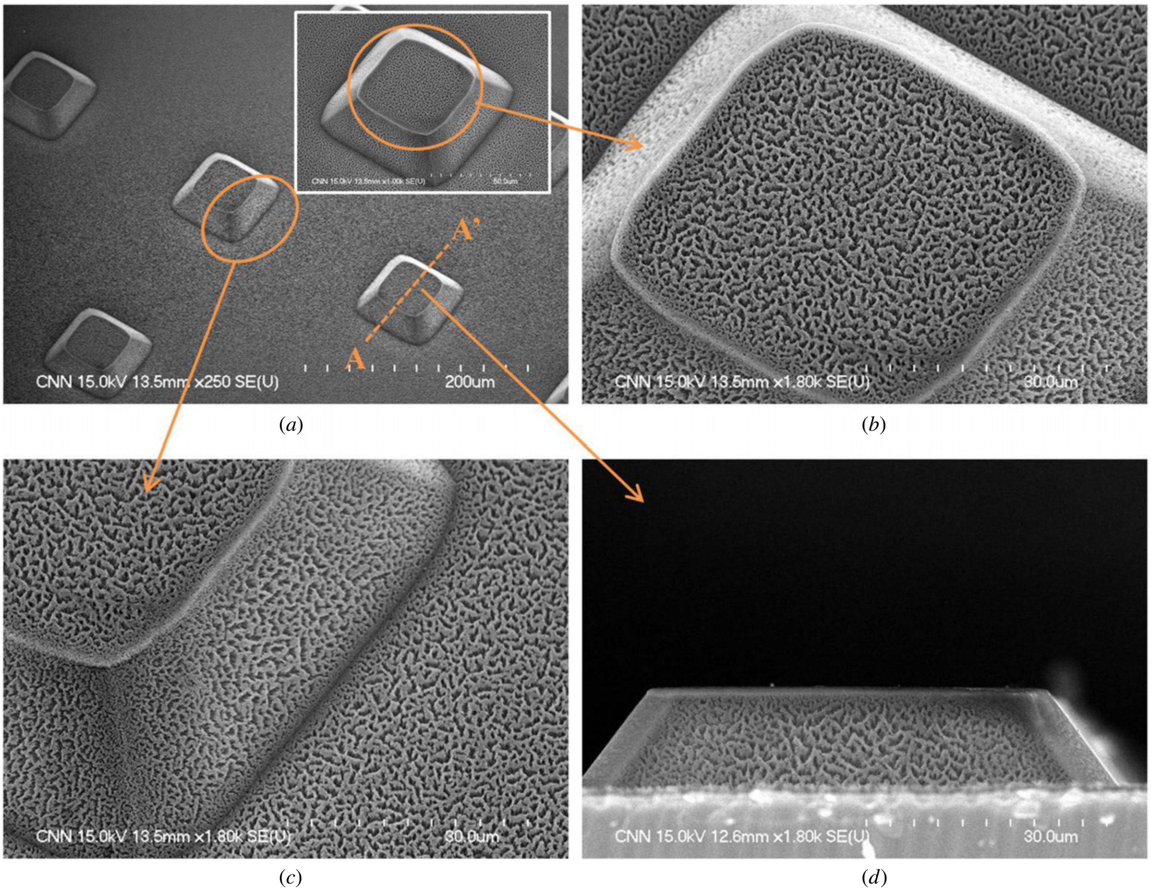


Figure 6. SEM images of the fabricated multiple-order hierarchical surface. (*a*) Arrayed view (inset: enlarged view of one cell), (*b*) enlarged view of the top surface, (*c*) enlarged view of the sidewall and bottom surface, and (*d*) enlarged side view of an inclined-wall micro-pillar.

the inter-pore walls and barrier layer become thinner with increasing etching time. In general, the alumina nanowires were generated at an etching time of 45–50 min, and the nanowire bundles were formed simultaneously after the rinsing and drying steps (figure 5(*d*)). Figure 6 shows scanning electron microscopy (SEM) images of the fabricated multiple-level hierarchical surface. As shown in figure 6, the ANF was formed entirely and uniformly on the inclined-wall micro-

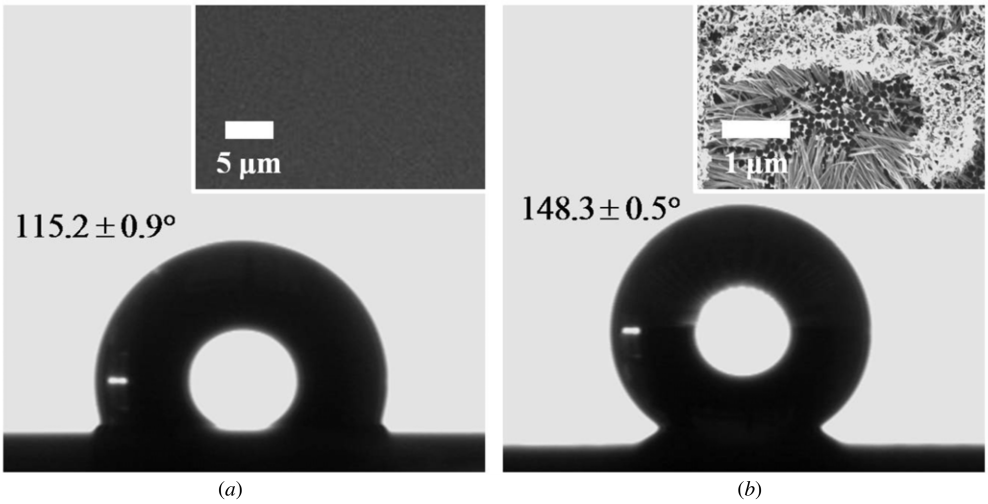


Figure 7. Images of the water droplets placed on each base surface. (*a*) On the flat surface, and (*b*) on the surface covered entirely with the ANF (inset figures: SEM images of the flat and ANF surfaces).

pillar arrays, and nanowire bundles and nano-roughness were also defined well. This suggests that the formation of the ANF based on the AAO technology must be one of the most efficient and stable ways to improve the surface wettability due to its fabrication simplicity and uniform wetting behavior, which have been already examined in our previous work [20], and geometric conformality on the roughened surface.

4. Measurements and analysis

4.1. Amplification of intrinsic contact angle of base surface

The static wetting properties of the fabricated surfaces were characterized by measuring the apparent CAs using a sessile drop method with deionized water (DI), 10 μL in volume. Before the measurements, all the fabricated surfaces were treated by depositing approximately an 80 nm thick PPFC film based on a continuous C_4F_8 glow discharge with a gas flow rate of 100 sccm and an applied power of 800 W to reduce the surface energy. All the CA measurements were performed on more than five different regions of the surface. The apparent CA of the surface covered entirely with ANF was measured to be 148.3 ± 0.9°. The DI water droplets placed on the flat and nanowire forest surfaces are shown in figure 7. This indicates that the intrinsic CA can be amplified by ∼29% compared to that of the flat surface, and the amplified intrinsic CA is much higher than the critical CA of 125.26°. This also suggests that the surfaces with inclined-wall micro-pillar arrays, which are initially in the Wenzel state, can be theoretically transitioned to the Cassie state by forming the ANF on the flat surface entirely.

4.2. Experimental and theoretical evaluation of static surface wettability

The apparent CAs of the mono-roughened micro- and the multiple-scale hierarchical surfaces were measured and compared with the CAs estimated theoretically. In particular, the measurements were performed on each surface with different periods of 50, 100, 150, 200 and 250 μm in micro-pillars to determine the wetting state precisely by observing the changes in the static CAs due to the period variations. Figure 8 shows the static CAs measured experimentally and estimated theoretically on each surface due to the period variations.

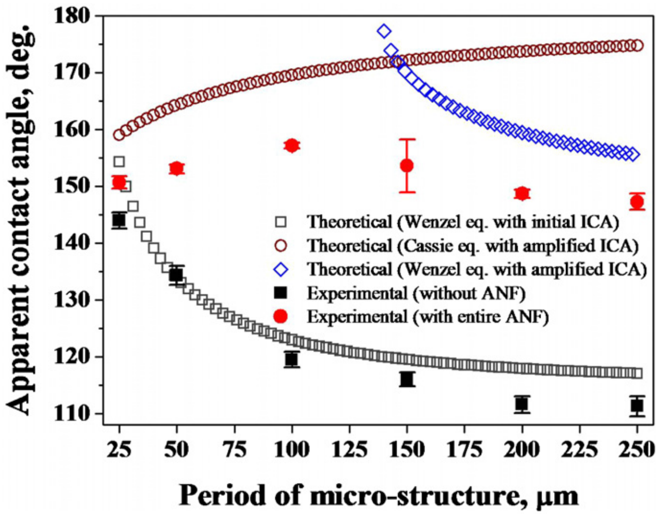


Figure 8. Theoretical and experimental apparent CAs on micro-roughened and multiple-scale hierarchical surfaces due to the period variations.

The apparent CAs measured on the micro-structured surface decreased gradually with increasing periods, which is in great agreement with the theoretically estimated values. In this case, the static wetting properties of the mono-roughened micro surface were estimated using the Wenzel equation (1) because the ICA of the flat surface was much lower than the critical CA, as described before. In particular, the gradual decrease in CA results from a decrease in the roughness factor in the Wenzel mode due to a decrease in the contact area between the water droplet and surface with the increasing

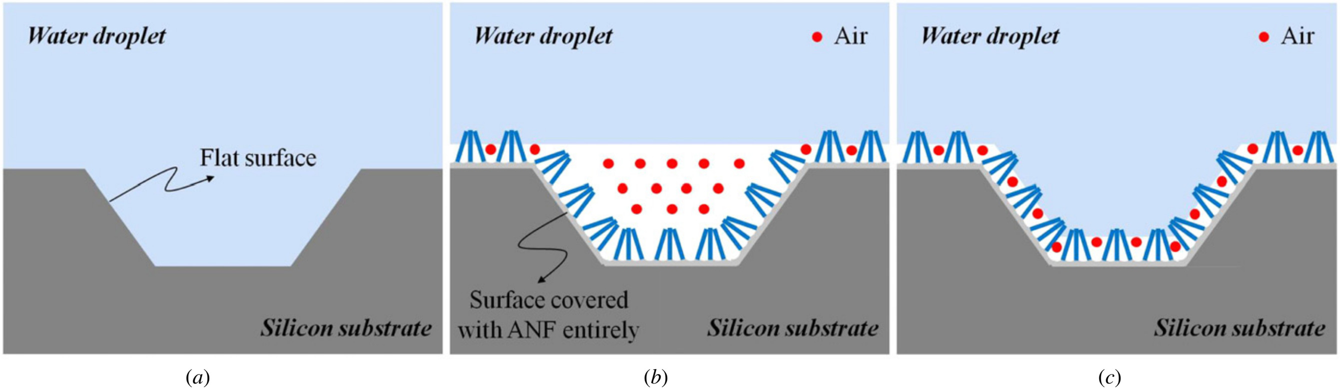


Figure 9. Possible wetting scenarios of the surfaces. (*a*) Full wetting on the micro-roughened surface (Wenzel regime), (*b*) no wetting on the hierarchical surface (Cassie regime), and (*c*) wetting on the micro-structure, no wetting on the ANF (Wenzel regime with amplified ICA). (Dot patterns denote air occupying the surface.)

periods. This means that the water droplet fully wets the entire surfaces and is independent of the period variations. Therefore, the surface wettability of the fabricated micro surface is always in the Wenzel regime, as illustrated in figure 9(*a*).

The static wetting properties of the hierarchical surface differed apparently from those of the micro-roughened surface as shown in figure 8 as though the two surfaces are all based on the inclined-wall micro-pillar arrays. The wetting property of the hierarchical surface was estimated basically with the Cassie wetting theory equation (2) because the amplified ICA was much higher than the critical CA. The CAs measured on the micro-roughened surface with an amplified ICA increased until the period reached 100 μm, and matched similarly with the trends estimated from the Cassie theory. The best CA, which was measured on the hierarchical surface with a period of 100 μm, was 157.2 $\pm$ 0.5°. This results from the decrease in the solid fraction of the surface with the increasing period in the Cassie mode because the amount of trapped air becomes much larger when the period is increased due to the effect of the entirely formed ANF as illustrated in figure 9(*b*).

On the other hand, the CAs of the surfaces with periods larger than 150 μm showed decreasing trends with increasing period, as shown in figure 8. To analyze this situation quantitatively, the wetting property of the hierarchal surface was also evaluated using the Wenzel model assuming that the micro-scaled surface was fully wetted, but the water droplet is supported on the ANF covering the micro-structures, as illustrated in figure 9(*c*). The wettability of the surface with periods larger than 150 μm agreed with the results estimated using the Wenzel wetting theory as shown in figure 8. This means that the water droplet can reach the bottom surface more easily in the large period because the ANF was formed conformally along the inclined walls of the base micro-structures.

Interestingly, the largest deviation of the CAs was found on the hierarchical surface with a period of 150 μm, which would be due to coexistence of the Wenzel and Cassie states in this case. That is, the two wetting states shown in figures 9(*b*) and (*c*) appeared on the surface simultaneously. Therefore, it can be probably concluded that the period of 150 μm is a geometrical boundary of the wetting transition in our surface design.

The surface wetting behavior characterized experimentally and theoretically can be clearly proven by observing the interface between the water droplet and surface. Figure 10 shows the captured images of the water droplets placed on each surface to observe the liquid/solid interface. As shown in figure 10, it was observed that the mono-scale micro-structured surfaces with the flat base surface were fully wetted without any air-trapping regions independent of the period variations. Hence, the surfaces are always in the Wenzel regime. In the hierarchical surfaces covered entirely with the ANF with periods less than 150 μm, the air-trapped sites between the micro-pillars were observed clearly as shown in figure 10. This suggests that the entire formation of the ANF on the micro-structured surface is quite effective for the transition to the Cassie state in the case of the surfaces with a high-density of micro-pillars. Although there were no sights of the air-trapped spaces between the micro-pillars in the case of the hierarchical surfaces with period larger than 150 μm, the surface wettability of the micro-roughened surfaces was enhanced greatly more than a 32% increase in the CA in the Wenzel state by amplifying the ICA of the base surface.

4.3. Measurements and characterizations of dynamic surface wettability

The slippery characteristics of the fabricated surfaces were evaluated more quantitatively by measuring the CAHs, which are defined as the differences between the advancing and receding CAs, to confirm experimentally the effectiveness of the multiple-scale integration with the ANF structures on the dynamic surface wettability. For this, the advancing and receding CAs were measured first by increasing and decreasing the volume of the water droplet, respectively, and the corresponding CAHs were obtained by calculating the differences between them. The CAH measurements were performed on at least five different regions of four types of surfaces with different surface geometries (e.g., flat, ANF, micro-roughened and hierarchical surfaces). The lowest CAH

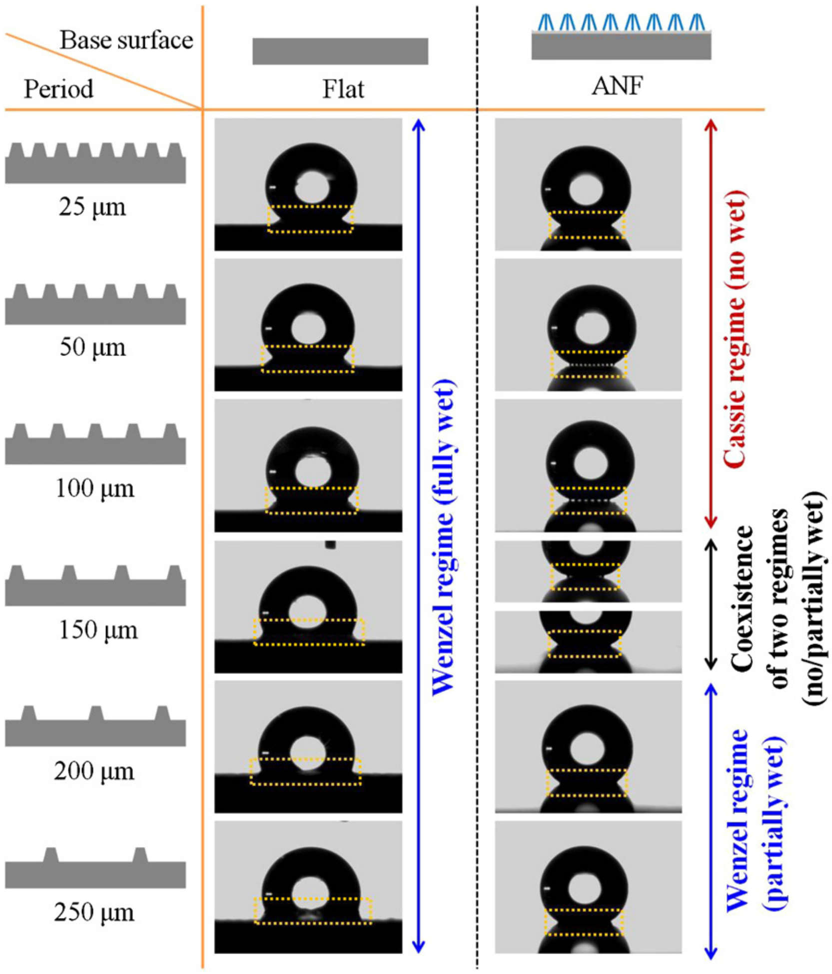


Figure 10. Images of the water droplets placed on each surface to observe the interface between the water droplet and surface. (Note the dotted box in all the figures.)

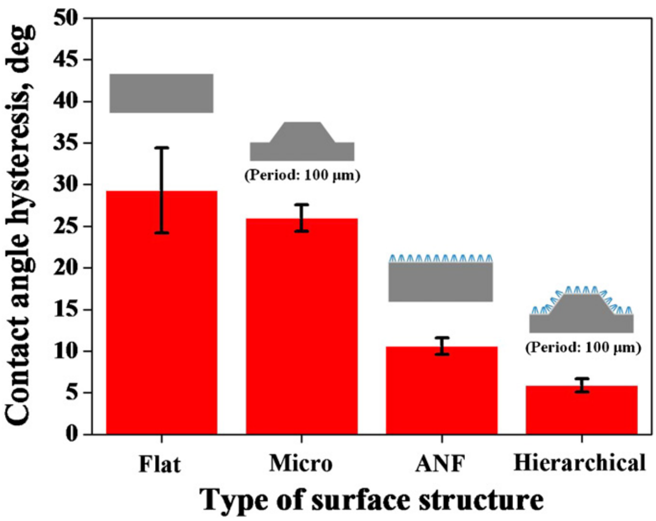


Figure 11. CAHs obtained experimentally from the four types of the surfaces with different surface geometries (flat, ANF, micro-roughened and hierarchical surfaces).

was found on the hierarchical surface, as shown in figure 11. In particular, the CAHs of the micro-roughened and hierarchical surfaces with the same period of 100 μm were measured and compared. The CAHs obtained experimentally from the micro-roughened and hierarchical surfaces were $26 \pm 1.6^\circ$ and $5.9 \pm 1.5^\circ$, respectively. The remarkable decrease in the CAH on the hierarchical surface means that the air-trappable sites are considerably increased due to the formation of the ANF entirely compared to the micro-roughened surface with the same period, resulting in perfect transition from the Wenzel to the Cassie state.

5. Conclusions

A multiple-scale hierarchically structured superhydrophobic surface based on inclined-wall regular micro-pillar arrays covered entirely with ANF was demonstrated. The proposed hierarchical surfaces were fabricated using a simple batch process based on the anisotropic silicon wet etching and subsequent time-controlled AAO processes. The effectiveness of the proposed hierarchical approach on improvement of the surface wettability was evaluated by measuring the apparent CAs and CAHs, and comparing with those of the mono-roughened counterpart. The ICA of the micro-scale mono-roughened surface was amplified ~29% by forming an

ANF entirely on the silicon base surface through a time-controlled nonlithographic AAO process. It was confirmed experimentally that the wetting property of the micro-scale mono-roughened surface could be enhanced remarkably by amplifying the ICA. In particular, the wetting property of the micro-roughened surface with a high density of the micro-pillars showed a perfect transition to the slippery Cassie mode. The mean static CA and CAH of the hierarchical surface with a period of 100 μm were 1.3 times higher and 4.4 times lower, respectively, than those of the micro-roughened surface with the same period. In addition, the average CAs of the hierarchical surfaces with a low density of the micro-pillars was increased by more than 32% compared to those of the micro-roughened surfaces in the Wenzel regime. The wetting property of the proposed hierarchical surfaces could also be predicted theoretically due to the structural regularity of the base micro-structures, and the experimental results agreed well with the wetting trends estimated using the analytic models. The experimental and theoretical observations clearly show that the proposed simple hierarchical approach is quite effective in improving the wetting property of the surface with the inclined-wall pillar-like micro-structures.

Acknowledgments

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