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Bioinspired Hierarchical Surfaces Fabricated by Femtosecond Laser and Hydrothermal Method for Water Harvesting

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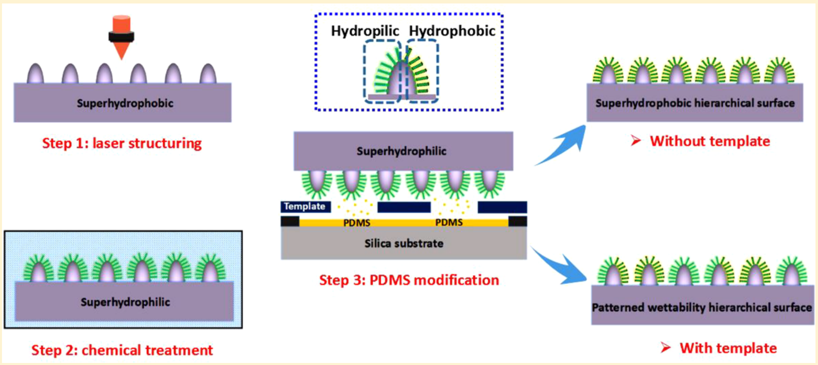
Supporting Information



ABSTRACT: The world is facing a global issue of water scarcity where two-thirds of the population does not have access to safe drinking water. Water harvesting from the ambient environment has a potential equivalent to ∼10% of the fresh water available on the earth's surface, but its efficiency requires a special control of surface morphology. We report a novel facile physicochemical hybrid method that combines femtosecond laser structuring with hydrothermal treatment to create a surface with a well-arranged hierarchical nanoneedle structures. Polydimethylsiloxane treatment of the thus-produced hierarchical structures nurtured superhydrophobic functionality with a very low water sliding angle (∼3°) and a high water adhesion ability. About 2.2 times higher water-collection efficiency was achieved using hierarchical structures over untreated flat Ti surfaces of the same area under a given experimental condition. The comparison of water-collection behavior with other samples showed that the improved efficiency is due to the structure, and wettability induced superior water attraction and removal ability. Moreover, a uniform water condensation under low humidity (28%) is achieved, which has potential applications in harvesting water from arid environments and in high-precision drop control.

■ INTRODUCTION

The incessant demand for clean water has grown tremendously because of rapid population growth, increased water pollution, and global climate change.[1,2] Recently, harvesting water from atmospheric humidity or fog has been considered to be a promising method to solve the water-shortage problem through condensing air moisture.[3−7] Related studies suggest that water harvesting from the ambient environment usually consists of three steps as follows: condensation of water vapor into tiny droplets, the growth of tiny water droplets already adhered to the surface, and the fall off of droplets. Therefore, fast vapor condensation, high-speed drop growth, and quick removal of large-sized droplets are the key elements needed for efficient water harvesting.[8,9]

More recently, water harvesting with artificial surfaces has been demonstrated by imitating creatures in nature.[6,7,10−13] For example; Heng et al. mimic the unique structural features of cactus spines as small-sized 1D structures for trapping and transportation of water droplets.[14] Another approach utilizes surfaces with patterned wettability following the lesson from Namib Desert beetles that possess effective water-collection abilities under extremely dry conditions.[10−12] Furthermore, surfaces with two-tier hierarchical micro/nanostructures are also predicted to promote high-frequency droplet growth and removal due to the existence of its unique structures.[15−19] Despite the many differences between the methods described above, Laplace pressure determined by surface morphology or chemical composition is crucial to water-harvesting efficiency because it can influence the tiny water droplet condensation and transportation process.[20,21] Therefore, morphology or chemical composition adjustment with special wettability would be beneficial for the improvement of water-collection efficiency.

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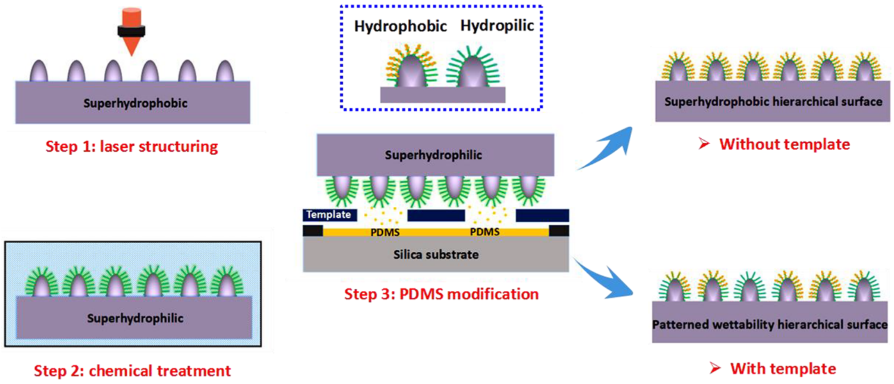

Figure 1. Schematic illustrations of the fabrication process and wettability change of hierarchical structures at different steps.

Among the artificial surfaces used for water harvesting, those with specially designed two-tier hierarchical micro/nanostructures stand out. The fabrication of such type of hierarchical structures is typically composed of a prestructuring treatment to fabricate microstructures, followed by the growth of nanostructures on the surface of microstructures.[16−19] Electron-beam lithography (EBL) is the most commonly used prestructuring method in this stage; it can provide precise control over the design and fabrication of microsized structures, but considering the slow, costly, complex, and, in particular, limited fabrication associated with EBL, this prestructuring method does not allow large-scale fabrication for practical water-harvesting applications. A large-scale fabrication of hierarchical micro/nanostructures is of paramount importance for the practicality and realization of high-efficiency water-collection technology. As a versatile structure fabrication method, femtosecond laser structuring has attracted a great amount of interest for its unique advantages of flexibility, precise structuring, and designability to efficiently obtain a large variety of microstructured surfaces on a large scale.[23−28] It is much faster and cheaper than other approaches for the creation of microstructured patterns and has been widely used to control surface wettability.[26−30] Although these features can enable the application of femtosecond laser structuring in this field,[29−32] higher water-collection efficiency could be achieved through the fabrication of large-scale well-arranged hierarchical structures, as proved in related researches.

Here 1D nanoneedle-based hierarchical structures were fabricated with a hybrid physicochemical approach of femtosecond laser structuring in combination with a scalable hydrothermal treatment. After the fabrication of hierarchical structures and the following treatment, we further develop a series of surfaces with special wettability, and their water-harvesting abilities are compared. About 2.2 times higher water-collection efficiency was recorded using hierarchical structures over untreated flat Ti surfaces of the same surface area under a given experimental condition. Moreover, a uniform water condensation under low humidity (28%) is achieved, which has potential applications in harvesting water from arid environments and in high-precision drop control.

■ EXPERIMENTAL SECTION

Fabrication of TiO_2 Hierarchical Micro/Nanostructures with Special Wettability. First, Ti sheets (purity 99.99%) were structured by a femtosecond laser system (TRUMPF, TruMicro 5000) with a central wavelength of 1030 nm, a duration of 800 fs, and a repetition rate of 400 kHz. A scanning galvanometer suitable for the laser beam was used to reach a high scanning speed of 1000 mm/s to create uniform microstructure square arrays on Ti sheets efficiently. Then, Ti sheets with microstructures were hydrothermally treated in 3 mol/L NaOH solution at 220 °C for 24 h using an electric oven. Next, the surface component was transformed to TiO_2 due to its low toxicity and strong stability under harsh environments. This process was conducted by immersing the hydrothermally treated titanium sheets in 1 mol/L HCl solution for 10 min, washing with distilled water, and annealing at 450 °C for 1 h in air using a muffle furnace. TiO_2 hierarchical structures were then achieved by the above treatments.

For the creation of surfaces with special wettability, a thin layer of polydimethylsiloxane (PDMS) liquid ($(C_2H_6OSi)_n$, SYLGARD 184) was spin-coated on a flat silica surface; then, the substrate was put into a preheated muffle furnace and maintained at 300 °C for 20 min to evaporate PDMS onto the TiO_2 surface. Superhydrophobic surfaces with hierarchical micro/nanostructures were fabricated due to the modification of low-surface-energy PDMS.

Characterization of the Fabricated Samples. The morphology of the sample was examined using a scanning electron microscope (SEM, Phenom-World, Phenom Pro). X-ray diffraction (XRD, Bruker D8 Advance) patterns were collected in the range of 20−80°.

The measurement of surface contact and sliding angles was performed on a video-based contact-angle measurement system (Powereach, JC2000D3) in the ambient environment. Measurements were conducted at five different positions on the same sample using a 4 μL water droplet, and the average value was used. The measurements for the surface with the largest water contact angle were taken with a 6 μL water droplet because it is too difficult to put a smaller water droplet on that sample.

Water-Collection Performance Measurement. A commercial ultrasonic humidifier with a flow rate of 0.0867 cm^3/s was used for supplying tiny water droplets and flow to the sample surface. The distance between the output of the fog source and the surface was ∼10 cm with a 45° inclined angle. A bottle was placed below the sample to collect water droplets falling from the sample surface. The water-collection process was recorded using an optical microscope and charge-coupled device (CCD). The detailed experiment setup is shown in Figure S1, similar to previous methods.[31,33]

■ RESULTS AND DISCUSSION

The detailed schematic and explanation of the fabrication process and wettability change of the samples with hierarchical structures is shown in Figure 1. After femtosecond laser structuring, the Ti surface presents uniform microscale square arrays. This process, or later stalling procedures in the air, can easily introduce some organic groups from the air to the surface of laser-treated structures. The increased surface roughness in conjunction with relatively lower surface energy

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organic groups can lead to the superhydrophobic properties of Ti surfaces after femtosecond laser structuring. (SEM images and the contact angle of the sample are shown in Figure S2.)[34−36] Following chemical treatment, the Ti surface exhibits good superhydrophilicity due to the presence of hierarchical TiO_2 created during this process. (XRD results of the sample after related chemical treatment and energy-dispersive X-ray spectrometry (EDS) results after PDMS modification are shown in Figure S3.) Finally, modification using PDMS by heat treatment can reduce the sample surface energy again.[37] Heat treatment, with or without a template (a stainless-steel sheet with 160 μm diameter circular hole arrays, the distance between adjacent hole is also ∼160 μm), can receive superhydrophobic hierarchical micro/nanostructures on the whole surface with a low sliding angle (referred to as Hierarchical-SHPO) and patterned wettability hierarchical micro/nanostructures (referred to as Hierarchical pattern), respectively, as the template can protect some part of TiO_2 from becoming covered with PDMS.

After femtosecond laser structuring and following chemical treatments, the morphology of the Ti surface shows hierarchical nanoneedles, about a few hundred nanometers on 13 μm microstructures, as shown in Figure 2. This kind of hierarchical structure with well-arranged nanostructures has proven to be useful for water collection.[14,15,19] We provide a novel method for achieving this kind of structure more easily simply based on the oriented growth of nanostructures on curved surfaces formed by femtosecond laser structuring. We should also note here that both the laser-induced structures and the hydrothermal treatment conditions are important for the formation of this kind of structure. For example, a lower concentration will lead to small-sized agglomerate nanowires, and a shorter reaction time will lead to twisted nanostructures or many particles. Even under the same hydrothermal treatment conditions, appropriate microarrays with curved outlines are more suitable for the growth of better structures. (SEM images in Figure S4 are used to show the importance of the fabrication condition.) Because many kinds of nanostructured metal oxides can be fabricated by chemical treatment, this proposed hybrid fabrication method may be easily extended to other metal oxides with similar structures and applications.[19,37−39]

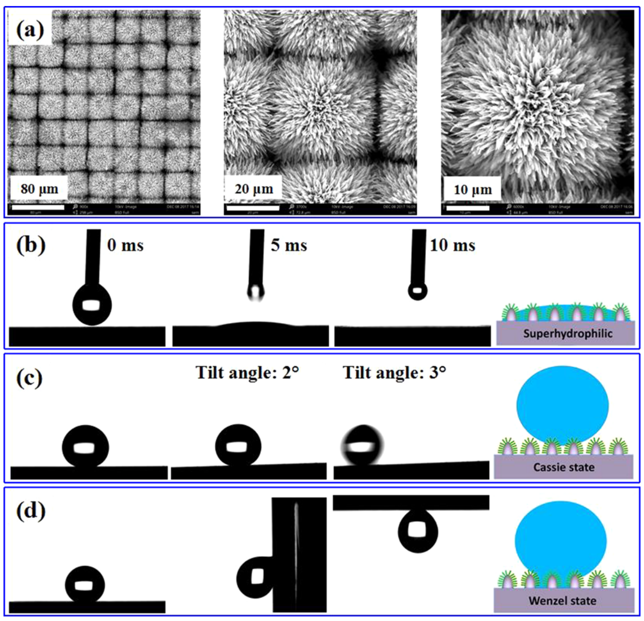

Figure 2. (a) SEM images of hierarchical structure Ti surface and (b) corresponding wettability for initial superhydrophilic surface. (c) Hierarchical-SHPO modified without template and (d) Hierarchical pattern modified with a template.

Accordingly, the water contact angle of the as-prepared Ti surface with hierarchical structures at different steps or treated by different methods is also presented in Figure 2. The Ti surface after hydrothermal treatment exhibits superhydrophilicity as the water drop spreads quickly within 10 ms and has a water contact angle of nearly 0° (Figure 2b). After modified with lower-surface-energy PDMS on the hierarchical structures without a template, the whole surface is covered by PDMS and presents superhydrophobic properties. Here a superhydrophobic surface with low adhesion is achieved for the surface water contact angle larger than 150° with a sliding angle of only ∼3° (Figure 2c). However, in contrast with PDMS-coated surfaces on the whole surface with a small sliding angle, water droplets were firmly pinned on the surface of the Hierarchical pattern sample and did not roll off their surfaces even when the patterned surfaces were tilted vertically or turned upside down (Figure 2d). According to previous research, whether a water droplet adheres to or rolls off the superhydrophobic surface is ascribed to the distinct contact modes.[40−42] In the Cassie state, a water droplet is suspended by the gas layers trapped at the structures, and thus the adhesion of the surface is extremely low and the water droplet easily rolls off even with the slightest tilt of the surface.[41] In contrast, in the Wenzel state, a water droplet fully penetrates into the grooves of a textured surface; then, the surface possesses high adhesion that could pin the water droplet to the surface without any movement.[42] Here, through modified PDMS with a template, patterned wettability is achieved in the Wenzel state with water droplets penetrating into the hydrophilic positions and remaining adhered despite inversion or tilting. On the contrary, experiments in the Supporting Information (Figure S5, Table S1, Figure S6) show that the surface fabricated by this method enables good durability in harsh environments (after contacting with a droplet of 1 M NaOH or HCl for 2 min or impinging of 1.2 kg of water flow from a height of 2 cm).

To investigate the water-harvesting behavior of different surfaces, an optical microscope was used to record the water-collection processes in situ. Figure 3 shows the representative optical images during the water-harvesting process on the four samples, including the untreated flat Ti surface (Untreated Ti), flat-surface nanostructure TiO_2 with patterned wettability (Nanoneedle pattern), Hierarchical-SHPO, and Hierarchical pattern, in our experiments. Morphology and wettability of other samples are shown in Figure S7. As we can see from the optical images taken during water harvesting, there are many differences among the four samples in our experiments. On the Untreated Ti surface, tiny droplets condensed very quickly, and the droplets became larger as the condensation continued (Figure 3a). Furthermore, after coalescing with neighboring drops, water films were formed over a prolonged period, which prevents further condensation and slows the fall off the already existing drops. For the Nanoneedle pattern sample, smaller sized drops with circular shape were observed due to its hydrophobic wettability (Figure 3b).

In contrast, on the hierarchical surfaces after coating with PDMS, tiny water droplets nucleated on the surfaces at a lower

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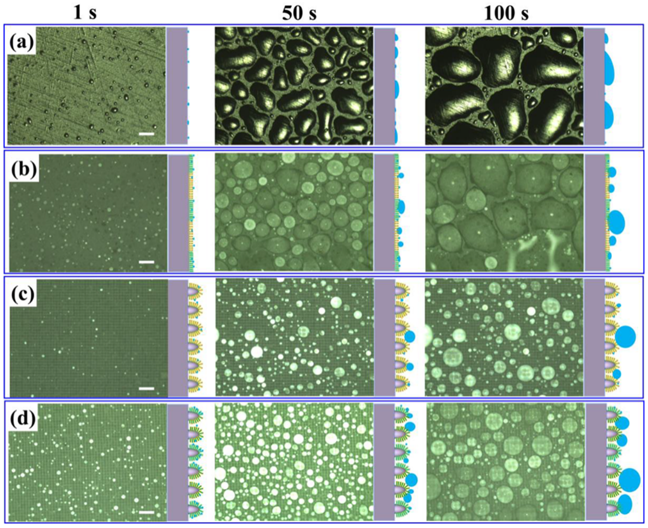

Figure 3. Schematic illustration and optical images showing the water-collection process for different samples under vapor flow environment. (a) Untreated Ti, (b) Nanoneedle pattern, (c) Hierarchical-SHPO, and (d) Hierarchical pattern. The scale bar is 200 μm.

speed. When the droplet reached a critical size, it fell off very quickly from the surface with a tiny droplet size. On the contrary, we can also compare the water-harvesting behavior of superhydrophobic hierarchical structures by examining their different sliding angles with optical images. The Hierarchical-SHPO with low sliding angle has fewer water drops on its surface mainly due to poor water-trapping ability and larger drops easily falling away, which are both caused by high superhydrophobic performance, whereas tiny drops can grow very quickly on the surface of the Hierarchical pattern sample due to the existence of some hydrophilic sites. Additionally, the superhydrophobic sites of the Hierarchical pattern sample ensure the growth and falling away of larger drops, which is highly beneficial for the improvement of water harvesting.

It is also worth noting that nanoneedle structures are important for the improvement of tiny droplet water trapping, as we can see from the water-harvesting phenomenon of the femtosecond-laser-structured microsized superhydrophobic surface without chemical treatment in Figure S8. A phenomenon similar to that of the Hierarchical-SHPO surface is observed, except that the Nanoneedle surface has poor tiny-drop-trapping ability due to its lack of hierarchical structures.

The average mass of the first falling droplet and the average time for the first droplet to fall from the surface measured five times are shown in Figure 4a, marked in blue. The first falling droplet is the first water droplet that falls into the container. Because the mass and onset time of the first single falling droplet represent the water-capturing capacity and water removal ability, more efficient water-harvesting ability can be achieved by realizing shorter onset times and larger droplet sizes. Here we can easily select out the hierarchical-SHPO and untreated Ti samples for the smaller relative weight of the droplet. It takes a longer time for the droplet on the untreated Ti surface to fall as compared with the hierarchical-SHPO mainly due to the hydrophilic property of the untreated Ti sample. For the patterned wettability surfaces (both flat-surface nanostructure TiO_2 and hierarchical structure), the mass and time are similar because they both possess a superhydrophobic state with a low sliding angle, but the higher contact angle of hierarchical structures can result in a short time for the droplet to fall, which is desirable for achieving the greatest possible water-harvesting ability.[43] Furthermore, the average drop mass and time for the droplet to fall from the surface (excluding the first droplet) were also measured five times, as shown in Figure 4a, marked in red. The variation tendencies of the four samples are similar for the first droplet, and the average measurements, except the average measurements (in red), were observed to have an overall shorter droplet fall time.

Furthermore, water-harvesting performance was compared by measuring the total weight of collected water over 1 h. The mass of collected water for 1 cm^2 area was measured every 10 min, as shown in Figure 4b. It was demonstrated that the water-harvesting efficiency is highest for Hierarchical pattern sample, which exhibits better performance than not only Untreated Ti but also Hierarchical-SHPO sample with the same structure. Compared with the Untreated Ti sample, which has a water-harvesting efficiency of only ~247 $mg{\cdot}cm^{-2}{\cdot}h^{-1}$, the efficiency of the hierarchical structure surface is ~2.2 times that of the Untreated Ti. As far as we know, the water-collection efficiency improvement in our work is currently the highest level based on laser structuring.[29−32]

The dropwise condensation behavior of the substrate is also important for water harvesting in places with extreme weather conditions, thermal management, and drop control, except for in extremely high humidity or foggy weather.[21,22] Another experiment was carried out in an ambient environment for in situ observation of the water droplet condensation performance. The ambient temperature is 25 °C, and the relative humidity is controlled at 28%. The samples were pasted onto a

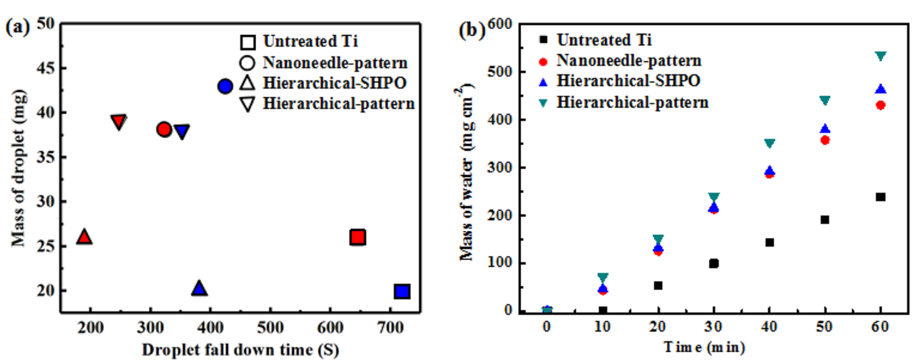

Figure 4. (a) Average mass and time of the droplet falling from each sample and (b) water-collection efficiency of different samples over 1 h.

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thermoelectric cooling stage (the temperature can be reduced to 0 °C within 2 min) using thermal grease to provide better contact. Figure 5 shows the photographs of the in situ formation of condensate water droplets on Untreated Ti, Nanoneedle pattern, and Hierarchical pattern samples after 16 min of cooling at 0 °C. We can clearly see that the water condensation is strongly dependent on the structural features. For the Untreated Ti, more tiny water droplets form due to its hydrophilic property, but the drop number density decreases very quickly as the drop diameter increases. Most importantly, in contrast with Untreated Ti and Nanoneedle patterns that have randomly distributed droplets, the size and position of droplets on the Hierarchical pattern surface are well organized. The ordered drop formation and controllability of Hierarchical patterns offer good strategies for rapid dropwise condensation and high-precision drop control.

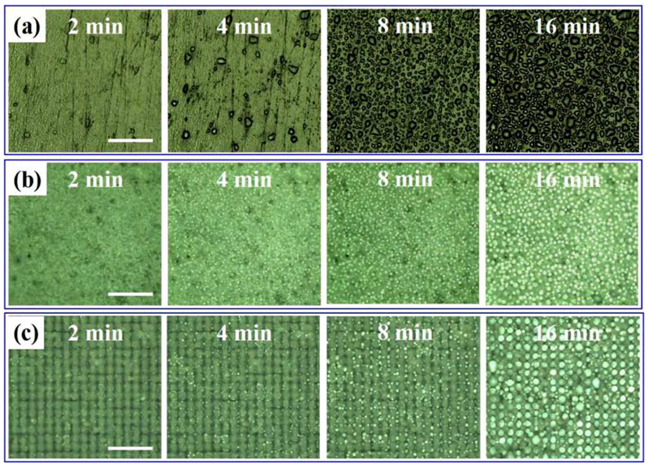

Figure 5. Optical images of (a) untreated Ti surface, (b) hydrothermally treated Ti flat surface, and (c) femtosecond-laser- and hydrothermally treated hierarchical surface under the condensation condition (surface temperature is 0 °C, surrounding temperature is 25 °C, humidity is 28%) by cooling method.

■ CONCLUSIONS

We successfully fabricated well-arranged hierarchical nanoneedle structures on a Ti surface through a simple laser–hydrothermal method. After modifying the surface using PDMS with or without a template, superhydrophobic hierarchical structures with very low adhesion or patterned wettability hierarchical structures possessing very high adhesion can be achieved, respectively. During the water-collection process under a vapor flow environment, the highest performance is achieved for patterned wettability hierarchical micro/nanostructures, which is 2.2 times that of the untreated Ti surface. Furthermore, the hierarchical surface also exhibits more uniformly distributed droplets under low humidity (28%), which shows great promise for further applications like rapid dropwise condensation and high-precision drop control. To that end, this proposed hybrid fabrication method may also be readily extended to the fabrication of a wide range of metal oxides with similar structures, which indicates great potential and flexibility for related applications.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.langmuir.8b04295.

Experiment setup for the measurement and observation during water harvesting; characterization of the sample after femtosecond laser structuring, chemical treatment, and PDMS modification; method to achieve tightly distributed nanoneedles; durability of the sample; characterization of flat-surface nanostructure and initial flat Ti; water-collection observation of laser structured surface (PDF)

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Notes

The authors declare no competing financial interest.

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