



# Wetting transition of sessile and condensate droplets on copper-based superhydrophobic surfaces



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## ABSTRACT

Superhydrophobic state on natural materials and synthesized surfaces has been exploited in a broad range of technologies including thermal management, water harvesting, anti-icing, and flow control. However, under certain circumstances wetting transition from Cassie's mode to Wenzel's mode becomes inevitable. Such wetting transition degrades the performance of superhydrophobic surfaces and limits their applicability. Here, we report distinct wetting stabilities of two copper-based superhydrophobic surfaces which are with nano-asperities (diameter  $\sim 70$  nm) of different packing density. Both the static (sessile droplet) and dynamic (dropwise condensation) wetting stabilities of the two surfaces are characterized. We show both theoretically and experimentally that sessile droplets on the surfaces of densely packed nano-asperities (pitch  $\sim 120$  nm) can remain in stable Cassie's mode, while the wetting transition from Cassie's mode to Wenzel's mode occurs spontaneously on the surfaces of coarsely packed nano-asperities (pitch  $\sim 300$  nm). The apparent contact angle on the surfaces of coarsely packed nano-asperities reduces from over  $150^\circ$  to around  $110^\circ$ , and the sliding angle increases from less than  $5^\circ$  to over  $60^\circ$  within 200 s, whereas the changes of both angles on the surfaces of densely packed nano-asperities are not noticeable. We also find that in dropwise condensation, condensed droplets on the surfaces of densely packed nano-asperities maintain a stable Cassie's mode, while condensate droplets on the surfaces of coarsely packed nano-asperities are in Wenzel's mode. Exploiting the coupling effects of surface topography and wetting behaviors can open up existing vistas on surface engineering, leading to durable and sustainable surface design for diverse applications such as dropwise condensation and boiling heat transfer.

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## 1. Introduction

The purposeful design of generating micro/nano scale asperities and modulating interfacial energy to achieve surfaces with specific wetting properties is of great importance due to the significance from both fundamental and application viewpoints [1–8]. Roughness-induced superhydrophobicity, usually referred to as “the lotus leaf effect”, is one of the well-known surface designs from this aspect. The study of superhydrophobicity has gained intensive attention recently, due to the outstanding performances of superhydrophobic surfaces in diverse applications, such as condensation/frosting heat transfer [9–14], self-cleaning [15–17], antifouling coating [18,19], and drag reduction and flow control in smart microfluidic and lab-on-chip devices [20–23]. Despite most of superhydrophobic surfaces are characterized by using

apparent contact angle larger than  $150^\circ$  and small sliding angle, their wetting mode and stability can vary largely. The degradation of hydrophobicity also becomes inevitable as a result of liquid imbibition and boundary pinning effect [24–27], particularly in the cases of condensation [28–30] and droplet impact [31]. The underlying physics associated with wetting stability remains incomplete [32–35], which makes it challenging to achieve optimal design and process control of the performance of superhydrophobic surfaces.

When placing a water droplet onto a rough superhydrophobic surface, wetting occurs in accordance with a composite state (Cassie's mode) with many micrometer/nanometer sized air pockets trapped underneath the droplet, yielding a partially wetted surface. The contact interface is thus comprised of air/water and water/solid interfacial components. An irreversible transition from the metastable Cassie's mode to the stable Wenzel's mode can be triggered by external stimuli such as Laplace pressure, kinetic impact, thermal perturbation and phase changes. The ability of a

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rough superhydrophobic solid surface to resist this wetting transition depends on geometry and packing density of the asperities. Identifying a universal criterion for wetting in a stable Cassie's mode is thus the key to developing novel functionalized surfaces.

In this work, we study the wetting stability of two types of superhydrophobic copper surfaces packed with nano-asperities ( $\sim 70$  nm in diameter), to explore the effect of the packing density of asperities on the surface wetting behavior. We fabricated these two types of the copper surfaces using the electrochemical deposition method via simply changing the deposition rate such that the smaller deposition rate produced a relatively large packing density ( $\sim 120$  nm in pitch), and the larger deposition rate produced a relatively small packing density ( $\sim 300$  nm in pitch). We characterized the wetting stability of these surfaces in two cases: (a) the evolution of static contact angle and sliding angle for a sessile water droplet; (b) the dynamic wetting behavior during dropwise condensation. We found that the wetting on the surface with densely packed nano-asperities shows a stable Cassie's mode, and the wetting transits to Wenzel's mode spontaneously on the surface with coarsely packed nano-asperities. The different dynamic wetting behaviors also lead to different regimes of inter-drop coalescence during the dropwise condensation. The findings of the present study can shed light on the design of durable and sustainable superhydrophobic surfaces which have wide applications such as condensation and boiling heat transfer.

## 2. Experimental section

### 2.1. Preparation of sample surfaces

As copper is a typical structural material used in heat exchangers, we chose thin copper plates as substrates, and fabricated two types of copper surfaces in this study. We used commercially available copper plates of 99.9% purity, and with a thickness of  $D = 1$  mm and a square cut of  $2 \times 2$  cm<sup>2</sup>. The sample substrates were first polished using P800, P2000 and P6000 sand papers (with grit sizes of 21.8  $\mu$ m, 10.3  $\mu$ m and 4.0  $\mu$ m, respectively) subsequently and followed by ultrasonication for 30 min to remove any residues from polishing. After that, the sample substrates were immersed in 1 M hydrochloride acid solution and then placed in another ultrasonic bath for 20 min to remove the oxidized film. Then, each substrate was cleaned using a standard protocol comprising of rinsed with acetone, ethanol, isopropyl alcohol, and deionized water subsequently and finally dried with nitrogen gas flow.

Nano-asperities were created on the aforementioned, pre-treated copper substrates using an electrochemical deposition method. The electrochemical deposition experiments were performed in a customized electrochemical reaction chamber, consisting of two copper electrodes (Anode as the source, and Cathode as the target substrate) and a chemical reagent, driven by a DC power supply. The chemical reagent used as the electrolyte solution was CuCl<sub>2</sub> with a fixed concentration of 100 mM. Two types of surfaces were prepared using different deposition rates. Surf\_1 was generated with an electrical current density of 0.02 A/cm<sup>2</sup> and a coating time of 100 s; while Surf\_2 was generated with an electrical current density of 0.1 A/cm<sup>2</sup> and a coating time of 20 s. Setting  $m = \frac{ItM}{zeN_A}$  as the deposited mass per unit area (where  $I$  is the current density,  $t$  is the deposition time,  $M$  is the molecule weight of Cu,  $e$  is the elementary charge and  $N_A$  is the Avogadro constant), the deposition density of the two types of surfaces was the same with its magnitude as  $670 \pm 7.4$   $\mu$ g/cm<sup>2</sup>.

To promote hydrophobicity, a self-assembled monolayer of fluorinated silane (trichloro (1H, 1H, 2H, 2H-perfluorooctyl) (Sigma-Aldrich) was deposited onto the copper substrates with nano-asperities via chemical vapor deposition ( $P < 0.01$  MPa). This

self-assembled monolayer layer has a typical static contact angle of around  $108.2 \pm 0.5^\circ$  when applied to a smooth surface (as seen in the supporting information Fig. S1). Prior to each experiment, the substrates were rinsed with DI water again for 2 min to remove dust and other contaminants.

### 2.2. Surface characterizations

Electrochemically deposited copper has been used in numerous applications that require a wide range of mechanical and physical properties. In most situations, however, atomically smooth glassy coating with shining appearance is preferred [36]. In the present study, we deployed an unusual large deposition rate to obtain coarse nucleation sites and a large growth rate of copper clusters, providing the essential roughness for achieving superhydrophobicity [37,38].

Fig. 1(a)–(c) show photographs of a bare copper substrate and two prepared substrates; the latter were obtained from the electrochemical deposition and the silanization. The prepared copper substrate from a small deposition rate (Surf\_1) appears a bright red color. The substrate from a large deposition rate (Surf\_2), however, shows completely different appearance of a dark black color. As these substrates are all made of the same material (pure copper), this difference in appearance is attributed to the absorption wavelength shift due to different surface nanostructures [39,40].

Fig. 1(d) and (e) show the topography of nano-asperities for the two fabricated substrates using a field emission scanning electronic microscope (FESEM, JEOL7600). For Surf\_1 fabricated with a small deposition rate, spherical/semi-spherical copper crystals with an aspect ratio close to unity one are densely packed on the substrate. For Surf\_2 fabricated with a large deposition rate, however, copper crystals form pointy clusters with a large aspect ratio and are coarsely packed on the substrate.

The topography of nano-asperities is resulted from the growth of copper nanocrystals during the deposition process. Recent studies reported that the growth of copper crystals varies for different combinations of electrical current density, electrolyte concentration and pH value [41]. As shown in the present study, changing the deposition rate is an effective approach to alter the copper crystalline structure and nano scale roughness. At a small deposition rate, copper crystals with scattered energetically preferential spheres/semi-spheres are seen. With enough time for the alignment of these crystals, they accumulate layer by layer and eventually form a planar interface with homogeneous distributions of copper crystals. At a large deposition rate, however, facet copper crystals form tall and slender clusters with heterogeneous distributions (as shown schematically in Fig. S2 of the supporting information).

### 2.3. Experimental setup used for dropwise condensation

We characterized the wetting stability of the two surfaces under two different situations, (1) for a sessile water droplet, and (2) during dropwise condensation. Here, we define the wetting stability as the capability of a micro/nano structured surface to resist the transition from dewetted Cassie's mode to wetted Wenzel's mode. Fig. 2 shows schematically the experimental setup consisting of a thermal control unit, an isolated condensation chamber, and a direct high-speed visualization part, used for the dropwise condensation characterization on these two surfaces. The substrate surface temperature is controlled via a dual stage thermal control unit (1st stage uses a heat sink with a coolant circulator, and 2nd stage uses a Peltier with Labview data acquisition module). The chamber is filled with a mixture of water vapor and nitrogen gas with a preset humidity. A high-speed microphotography is used to capture the condensation process. The sample surfaces are aligned on a cooling stage with constant temperature of

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