



# Control wetting state transition by micro-rod geometry



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## ABSTRACT

Understanding the effect of micro-structure geometry on wetting state transition is important to design and control surface wettability. Micro-rod model was proposed and the relationship between micro-rod geometry and wetting state was investigated in the paper taking into account only the surface roughness and neglecting the chemistry interaction. Micro-rods with different geometric parameters were fabricated using micro-fabrication technology. Their contact angles were measured and compared with theoretical ones. The experimental results indicated that increasing the height and decreasing the space of micro-rod may result in Cassie wetting state, while decreasing the height and increasing the space may result in Wenzel wetting state. A suspended wetting state model due to scallops was proposed. The wetting state transition was interpreted by intruding height, de-pinning and sag mechanism. It may offer a facile way to control the surface wetting state transition by changing the geometry of micro-rod.

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## 1. Introduction

Control of surface wettability is desirable in a wide range of domains as industry, biology, and medicine [1,2]. For instance, the possibility of making hydrophobic and hydrophilic surfaces on the substrate is of particular interest for bio-analytical applications and micro-fluidics.

Hydrophobic surface should have a large contact angle and a low contact angle hysteresis, and vice versa. It is known that the surface hydrophobicity or hydrophilicity is highly related to wetting state. A drop reside on top of roughness grooves, i.e., in Cassie state [3], tend to be hydrophobic, while a drop impale the roughness grooves, i.e., in Wenzel state [4], tend to be hydrophilic. Thus, it is of interest to understand when a drop will transfer from Cassie state to Wenzel state and thus lead to hydrophobicity–hydrophilicity transition.

A lot of theoretical and experimental works have been done to study the wetting state transition mechanism. Several wetting state transition criteria, such as the contact line density [5], the energy barrier [6] and the spacing factor [7], have been formulated to predict the transition from a Cassie state to a Wenzel state. Various means such like plasma etching [8–13], soft lithography [14,15], laser processing [16,17], electroplating [18] and template replica [19] plus chemically modification, have been used to change the surface roughness and chemical composition and thus control the wetting state.

Despite so many works, the mechanism of wetting state transition is not totally understood. Especially, a correlation between surface micro-structure geometrical parameters and wetting state transition and a thorough mechanism responsible for this effect of micro-structure have been lacked. Further experimental observation and theoretical analysis on the above aspects are still important for fundamental research and real application.

The main aim of this paper is to understand the relationship between micro-structure geometry and wetting state transition. Micro-square rod model was proposed for controlling the surface wettability. Micro-fabrication technology based on lithography was used since it is easy to control the geometric parameters of micro-structure. Different geometric parameters were investigated taking into account only the surface roughness and neglecting the chemistry interaction. The experimental results indicated that the wetting state can be adjusted by only changing the micro-rod geometry. Wetting state transition can be explained by intruding height, de-pinning and sag mechanism in some extent. Particularly, a suspended wetting state model due to scallops was proposed. The results may help to find a facile way to control the surface wetting state and thus can tailor the hydrophobicity/hydrophilicity by only control the micro-structure geometry.

## 2. Experiments

P-type silicon (100) wafers were used as substrates. Before the experiment, the silicon wafers were thoroughly cleaned. The rod pattern was transferred from the mask to a photoresist by lithography (Karl SUSS Inc., MA6). The patterned silicon surface with

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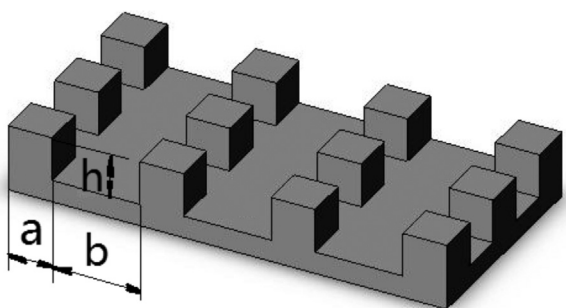


Fig. 1. Geometry of periodical micro-rod.

micro-rod was generated by deep reactive ion etching (STS Inc., ICP ASE).

The morphology of the silicon surface was observed with scanning electron microscope (JEOL, JSM-6309A) at 20kV. The contact angle and contact angle hysteresis of the as-prepared silicon surfaces with micro-rod were measured based on the sessile droplet method using an optical contact angle measurement device (OCA15EC, DataPhysics Company Ltd.). Deionized water droplets with volumes of 2  $\mu\text{L}$  were employed for the contact angle (CA) measurements. The contact angle hysteresis (CAH) was obtained by subtracting receding angle ( $\theta_R$ ) from advancing angle ( $\theta_A$ ). Deionized water droplets with volumes of 4  $\mu\text{L}$  and increment of 1  $\mu\text{L}$  were employed for advancing and receding angle measurements. The mean value and standard deviation was calculated from at least 5 individual measurements. The experiments were performed at 20 °C and 30% relative humidity.

### 3. Results and discussions

#### 3.1. Geometry and measurement contact angles of micro-rod

A periodical micro-rod model is proposed for control the surface wettability. Geometry of periodical micro-rod is shown in Fig. 1, where  $a$  denotes rod width,  $b$  denotes rod space,  $h$  denotes rod height.

Micro-rods with different geometric parameters were designed and fabricated. Considering the feasibility of design and

Table 1

Measurement contact angle and contact angle hysteresis of micro-rod ( $a=10\ \mu\text{m}$ ,  $h=40$  and  $20\ \mu\text{m}$ ).

$h$ ( $\mu\text{m}$ )	$a$ ( $\mu\text{m}$ )	$b$ ( $\mu\text{m}$ )	$b/a$	CA ( $^\circ$ )	CAH ( $^\circ$ )
40	10	10	1	125.6 $\pm$ 3.1	6.8 $\pm$ 1.5
40	10	20	2	150.7 $\pm$ 1.5	2.7 $\pm$ 1.0
40	10	30	3	151.8 $\pm$ 2.5	2.9 $\pm$ 1.1
40	10	40	4	155.7 $\pm$ 3.0	1.0 $\pm$ 1.0
40	10	50	5	151.5 $\pm$ 2.0	3.2 $\pm$ 1.5
20	10	10	1	44 $\pm$ 7.6	15.9 $\pm$ 4.2
20	10	20	2	56.6 $\pm$ 6.3	12.1 $\pm$ 3.2
20	10	30	3	52.8 $\pm$ 4.3	13.1 $\pm$ 2.5
20	10	40	4	54.2 $\pm$ 5.4	12.8 $\pm$ 2.6
20	10	50	5	42.2 $\pm$ 6.1	13.2 $\pm$ 2.7

micro-fabrication process, the rod height, width and space is set as a series of values  $h=20, 40\ \mu\text{m}$ ,  $a=10, 20, 30, 40\ \mu\text{m}$ ,  $b=10, 20, 30, 40, 50, 60, 80, 90, 120, 160\ \mu\text{m}$ .

The SEM images of a series of micro-rod ( $h=40\ \mu\text{m}$ ,  $a=10\ \mu\text{m}$ , (a)  $b=10\ \mu\text{m}$ , (b)  $b=20\ \mu\text{m}$ , (c)  $b=30\ \mu\text{m}$ , (d)  $b=40\ \mu\text{m}$ , (e)  $b=50\ \mu\text{m}$ ) are shown in Fig. 2. The measurement contact angle (CA) and contact angle hysteresis (CAH) of silicon surface with different micro-rod geometric parameters are listed in Table 1. Here the mean value plus standard deviation of CA and CAH is given. Lots of experiments were implemented and the corresponding data can be seen in Supplementary material.

#### 3.2. The effect of micro-rod geometry on wetting state transition

It is known that there are typically two prominent wetting states. The drop either sits on the peaks of the rough surface, i.e. Cassie state, or it wets the grooves, i.e. Wenzel state, as Fig. 3 shows. Theoretical contact angles based on Cassie–Baxter model [3] and Wenzel model [4] are described as Eqs. (1) and (2). Where  $\theta_C$  is the theoretical contact angle of Cassie state,  $\theta_W$  is the theoretical contact angle of Wenzel state,  $\theta_e$  is the instinct contact angle,  $a$ ,  $b$ ,  $h$  is the rod width, space and height. Here, The average value of 10 measured contact angles of smooth silicon surface, 59.9°, was used as the instinct contact angle  $\theta_e$ , as Fig. 4 shows.

$$\cos \theta_C = f(\cos \theta_e + 1) - 1 = \frac{1}{(1 + b/a)^2} (\cos \theta_e + 1) - 1 \quad (1)$$

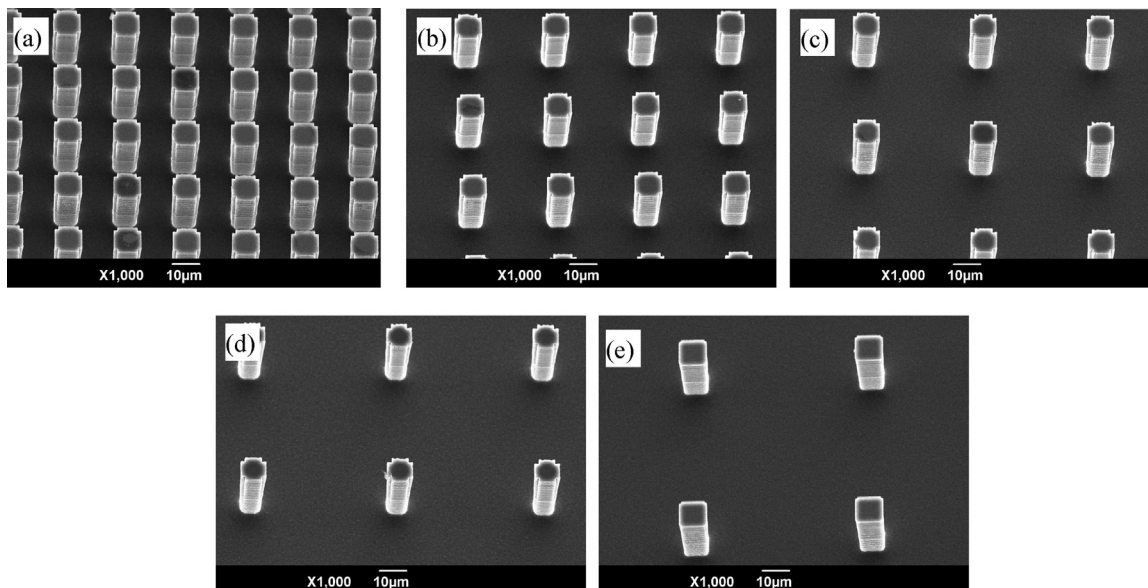


Fig. 2. SEM images of micro-rod with different geometric parameters.

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