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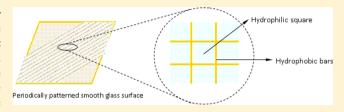


Delta-Comb Potential in Modeling Three-Phase Contact Line (TPCL) on Periodically Patterned Surfaces

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ABSTRACT: This work is a study of wetting of small water droplets on smooth glass surfaces with periodic patterns in the form of imprinted net with hydrophilic cells and hydrophobic bars. Microcover slides consisted of soda lime glass were used. The imprinted images of the net were with cell sizes in the range 40–200 μ m, which corresponds to a quite narrow scope of hydrophilic surface fractions $f_1(30-36\%)$ due to the relative increase in the size of the hydrophobic bars. The receding



contact angles θ_R of small water droplets, positioned on the patterned surfaces, were measured. The experiment showed significantly lower receding contact angles as compared to the theoretical expectations by the Cassie formula, which accounts for the contribution to the contact angle of the surface fraction of the imprinted hydrophobic/hydrophilic net. For this reason, we developed new theory accounting for the periodicity of the surface and the contribution of the three-phase contact line on the contact angle. This new theory considered delta-comb potential energy $\Delta(x,y)$ of the surface, effective line tension κ , and the lattice parameter a. The restriction of theory was discussed as well. It was pointed out that the theory is not valid for very small and very large lattice parameters.

INTRODUCTION

Contact between liquid droplets and solids is controlled by adhesive, capillary and other forces, the balance of which determines the properties of the three-phase contact line (TPCL) and the regime of wetting. 1-5 The classical models on wetting dynamics 6-10 are derived assuming morphologically and chemically homogeneous surfaces. In most cases the wetting occurs however, on naturally rough surfaces. 11 It is established that the hydrophobicity⁵ and morphology¹²⁻¹⁵ of the solid substrate influence significantly the regime of wetting. To study these effects, methods for chemical and morphological 16 pattering of solid substrates were developed in the past decade. Thus, it was established that the hydrophobic surfaces become superhydrophobic upon the increase of their roughness¹⁷ due to microbubbles trapped in between the surface asperities. It was found out as well that complete wetting is possible on superhydrophobic surfaces via the Cassie–Wenzel wetting regime transitions. 18–20 One of the common ways to study wetting is by measuring the receding contact angle of evaporating droplets. ^{21–24} Thus, was established ^{21,24,25} that two wetting modes during the droplet evaporation exist: constant contact area and constant contact angle modes. The realization of each one of them depends on the solid hydrophobicity. If the contact angle θ_c < 90°, it decreases in time thus realizing constant contact area mode. On the contrary, if the contact angle $\theta_c > 90^\circ$, it remains constant in time thus realizing constant contact angle mode. The wetting behavior determined in a such way has been tested for various surfaces, 26-28 hence, showing significant effects emerging from the molecular interactions at the contact line.²⁹ Along with the seminal

works of Cassie, Baxter, and Young, ^{12,30,31} the pioneering works of Johnson and Dettre^{32–34} about the wetting on idealized and nonidealized heterogeneous surfaces should be mentioned. They established that a number of metastable configurations caused by the surface heterogeneity and the vibrational energy of the drops control the contact angle hysteresis. However, this vibrational energy is closely related to the line tension of the droplet.7,35

 $\hat{\text{It}}$ is clearly shown in the literature $^{36-38}$ that line tension plays significant role only in the cases of small liquid droplets in contact with a solid surface. In addition, a number of metastable states corresponding to different contact angles exist due to the contribution of the line tension. The classical theories on static wetting of Cassie, Cassie–Baxter, 12 and Young 31 do not consider the effect of line tension. Hence, these theories cannot be applied to wetting of very small amounts of liquid on solid surfaces. The wetting on rough and energetically heterogeneous surfaces has been intensively studied 31,38-42 for many years. For example, it is known that the Cassie theory³⁰ predicts the contact angle of enough large droplets on a chemically heterogonous surface. In such a case, as mentioned above, the effect of line tension is negligible. However, in the case of enough small droplets positioned on such surface the line tension becomes essential and should be affected by the chemical surface heterogeneity. The droplet shapes on a chemically and morphologically inhomogeneous surface have

July 22, 2012 Received: Revised: October 12, 2012 Published: October 18, 2012 been studied in the literature (e.g., see refs 38, 39, and 42–44). It was shown that the chemically heterogeneous surface causes wetting transitions at which the shape of the wetting edge changes in a specific and usually abrupt manner. If the droplet is substantially larger than the surface patterns the contact angle usually is described by the classical Cassie—Baxter equation. However, if their length-scales are in the same order of magnitude the periodicity of the solid affects the contact angle. For this reason corrections of Cassie—Baxter equations has been made. More detailed analysis on the problem detailed indicates the existence of contact line contrast and gradients of the intermolecular interactions engendered by the surface inhomogeneity. In addition, it was recently proved experimentally and theoretically detailed and hydrophobic patterns the shape of the droplet can vary substantially.

In our preceding paper⁴⁹ we investigated the behavior of aqueous wetting films on periodically patterned smooth surfaces. It was shown that a net of periodically ordered small droplets are formed upon the rupture of the wetting films. These droplets are positioned on spots from the surface containing both hydrophobic and hydrophilic segments. However, their wetting behavior could not be determined. We performed with this work a profound study on the wetting of small droplets on smooth periodically patterned surfaces.

EXPERIMENTAL METHODS

Four types of microscopic grids with different sized hydrophilic squares and hydrophobic bars were patterned on glass surfaces. The parameters of nets used for patterning are shown in Table 1.

Table 1. Sizes of Grids Imprinted on the Glass

No.	square side $(\mu \mathrm{m})$	distance between squares (μm)	lattice constant (μm)
1	40	32	72
2	71	50	121
3	100	74	174
4	200	136	336

Cleaning Procedure. Micro cover glass slides of soda lime glass with sizes 18×18 mm were cleaned with bichromic cleaning solution. The cleaning procedure consisted of soaking the glass plates for about 24 h in the cleaning solution followed by rinsing with DI water. After this the plates were dried in a dry oven. Every water droplet positioned on such a glass plate spread completely, which corresponds to zero contact angle.

Hydrophobization. Thus dried, the glass plates were left for a couple of days in a desiccator, in contact with vapors of hexamethyldisilazan (Fluka). The hydrophobization of the glass surface is due to the chemical interaction of the hexamethyldi-

silazan $(CH_3)_3$ -Si-NH-Si- $(CH_3)_3$ with Si-OH groups of the surface producing Si-O-Si- $(CH_3)_3$ (see Figure 1)⁵⁰

Every droplet positioned on such hydrophobized glass plate had contact angle larger than 90°.

Surface Patterning. It is known⁵¹ that UV light is a powerful tool for oxidation of organic matter. Hence, it should promote the oxidation of the organic groups anchored on the glass surface. A possible mechanism^{51,52} of the UV light effect on the hydrophobized surface is presented in Figure 2.

The UV light ($\lambda \sim 185-270$ nm) atomizes oxygen from the air thus causing production of ozone. The latter is known to be one of the most powerful oxidizers, which converts the silica and carbon species in silicon and carbon dioxides. When the hydrophobized surface is illuminated with UV light for about 30 min, it becomes completely hydrophilic. Our experiment showed that water droplets positioned on such a surface spread completely, thus, forming liquid monolayers (contact angle 0°).

A grid with hydrophilic squares and hydrophobic bars was patterned on the glass surface. The surface patterning was performed in the following way: after the hydrophobization procedure, a grid with a given size of cells was positioned on the glass surface and the latter was illuminated with UV light (pen ray of spectral lamp, L.O.T. Oriel GmbH, Germany) for about 30 min. Thus, the illumined areas from the glass surface (the grid squares) became hydrophilic, while the non-illuminated areas (covered by the bars of the grid) remain hydrophobic (Figure 3).

A reasonable question here remains the quality of the surface patterning. Thus, we investigated the sharpness of the printed images of the different nets on the glass surfaces. The patterned surfaces were exposed to blowing with water vapors, thus, causing condensation of the latter. The glass surfaces with the newly formed water microdroplets were visualized by means of a metallurgical inverted microscope, captured by CCD video camera and recorded on computer. A typical example of thus observed images is presented in Figure 4. The latter shows that the condensation of water vapors on the patterned surface occurs everywhere, but the hydrophilic spots are preferred (see Figure 4A). The typical size of the microdroplets is within the range of 3-5 μ m. Their coalescence within the hydrophilic spots caused the formation of significantly larger droplets, whose diameter coincided with sizes of the square (see Figure 4B) of the imprinted net. The distances between the droplets were in a close agreement with the sizes of the hydrophobic bars (see Table 1). This is an indication for good sharpness of the imprinted net on the glass surface.

The image of the grid imprinted on the glass surface is quite sharp. Thus, one can calculate f_1 and f_2 geometrically by means of pictures of the grids. The only error can come from some small defects on certain places on the very grids. Our esteem is that the error is less than 1%.

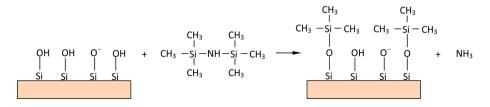


Figure 1. Reaction of the hexamethyldisilazan molecules with the glass surface Si-OH groups.

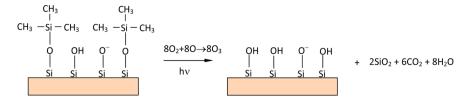


Figure 2. Mechanism of action of the UV light on the hydrophobized surface.

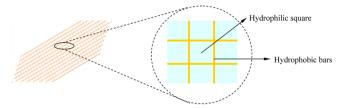


Figure 3. Sketch of the glass slide with imprinted net with hydrophilic cells and hydrophobic bars (not to scale).

■ CONTACT ANGLE MEASUREMENTS

The contact angle of 3 μ L (about 2 mm width) water droplets on the patterned glass slides was measured with an automatic goniometer/tensiometer (model 250, Ramé-Hart Instruments Co., U.S.A.). The droplets were positioned on patterned areas of the slides and left free to evaporate at a constant room temperature of about T=22 °C. The goniometer was controlled by computer with software for automatic determination of the receding contact angle.

Each measurement was repeated at least twice and showed good reproducibility of the experimental results. The standard experimental deviation was about 2°, which is significantly less than the experimental trends observed. The average contact angle between the left and right sides of the upfront view of each droplet versus time was measured. In all of the cases the difference between the two angles did not exceed 1.5°. Cosines of each set of contact angles was presented versus time in line

with eq 7 (see the Theory section). The geometrical characteristics of the droplets (height and width) were measured as well but are not a subject of the present study. Their processing gives information about the overall shape of the droplets during their evaporation. An interesting point is the final collapse of the droplets when reaching certain critical volume. The present study does give information about the shape of the three-phase contact line (TPCL) and its shrinking during the evaporation. Such study will be useful and it is indeed to be conducted in future.

Theory. The classical theoretical model of Cassie³⁰ was tested for our particular case of wetting of 3 μ L water droplets on the chemically patterned solid surfaces. According to this model the contact angle is formed due to the combined action of hydrophilic and hydrophobic fractions of the solid surface:

$$\cos \theta = f_1 \cos \theta_1 + f_2 \cos \theta_2 \tag{1}$$

where f_1 and f_2 are surface fractions of solid areas of types 1 and 2 and θ_1 and θ_2 are their specific contact angles. For our particular case, 1 and 2 correspond to hydrophilic ($\theta_1 = 0^\circ$) and hydrophobic ($\theta_2 = 93.4^\circ$) solid areas. Our calculations showed significant discrepancy between the Cassie formula and the experiment. For this reason we derived a new theory accounting for the periodicity of the surface patterns.

The problem of wetting of structured surfaces possesses both equilibrium and kinetic aspects. For instance, the equilibrium, advancing and receding contact angles could be strongly

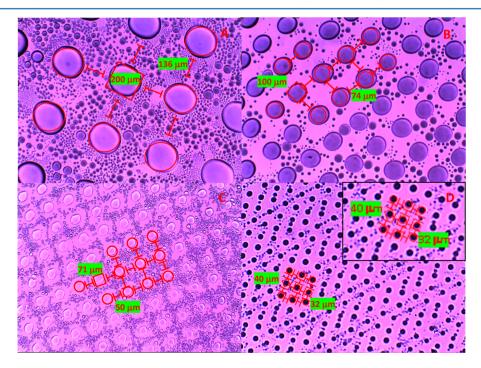


Figure 4. Images of condensed water droplets of 200 (A), 100 (B), 71 (C), and 40 μ m (D, see the inlet) patterned surfaces.

affected by the Cassie or Wenzel states. The present paper describes wetting by water of a hydrophilic glass surface patterned by a net of hydrophobic stripes with a lattice parameter a. As was shown, we prepared surfaces with different lattice parameters. Let us consider a drop of water deposited on this structured solid surface, with a drop radius much larger than the characteristic size of the hydrophilic island. The overall effective Young—Laplace equation for the contact angle θ reads

$$\cos \theta = (\sigma_{\rm SG} - \sigma_{\rm SL}) / \sigma_{\rm LG} \tag{2}$$

where σ_{SG} , σ_{SL} , and σ_{LG} are the solid/gas, solid/liquid, and liquid/gas surface tensions, respectively. The pure glass surface is completely wettable and in this case the three surface tensions above define an equilibrium contact angle θ_1 , which is almost 0° . On the structured surface, an additional force per unit length $\overline{\Delta}$ acting on the three-phase contact line of the drop should be added to obtain

$$\sigma_{\rm SG} - \sigma_{\rm SL} = \sigma_{\rm LG} \cos \theta_1 + \overline{\Delta} \tag{3}$$

The parameter $\overline{\Delta}$ determines the effective equilibrium contact angle and could take into account either Cassie or Wenzel states.

To model the effect of the stripes square lattice one can employ a delta-comb potential, which corresponds to the following effective potential energy per unite surface

$$\Delta(x, y) = -(\pi \kappa/2) \sum_{j} [\delta(x - ja) + \delta(y - ja)]$$
(4)

where $\delta(\cdot)$ is the Dirac delta-function, a is the lattice parameter, and κ is a specific constant accounting for the interfacial energy differences on hydrophobic and hydrophilic parts. Because the drop possesses radial symmetry, one can calculate the angular average of this energy as follows

$$\overline{\Delta}(r) = \frac{1}{2\pi} \int_0^{2\pi} \Delta(r \cos \varphi, r \sin \varphi) d\varphi$$

$$\approx (\kappa/a) [\arcsin(a/r) - \pi/2]$$

$$\approx \kappa/r - \pi\kappa/2a \tag{5}$$

The last simplified expression here takes into account the fact that the drop is much larger than the pattern characteristic size, that is, $r \gg a$. Introducing now eq 5 in eq 3 and substituting the result in eq 2 yields

$$\cos \theta = \cos \theta_1 - \pi \kappa / 2\sigma_{\text{LG}} a + \kappa / \sigma_{\text{LG}} r \tag{6}$$

Equation 6 shows that the parameter κ plays the role of an effective line tension. The order of the second term is about 10^{-1} , while this one of third term is about 10^{-9} . Hence, the third term can be neglected. In addition, since the glass surface is completely wettable it follows that $\cos\theta_1\approx 1$. Hence, eq 6 simplifies additionally to

$$\cos \theta = 1 - \pi \kappa / 2\sigma_{LG} a \tag{7}$$

RESULTS AND DISCUSSION

In the case of a homogeneous hydrophobic surface, the three phase contact line (TPCL) moves keeping regime of constant contact angle, which is known in the literature. However, in the case of evaporating small droplets on hydrophobized and patterned surfaces (see Figure 5), the situation is different. Clear "stick—slip" motion of TPCL during the evaporation can be observed. After the disposition of the droplet, the TPCL is

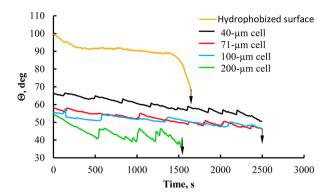


Figure 5. Contact angle vs time of evaporating droplets on hydrophobized and patterned surfaces. Arrows signify sudden collapses of droplets due to their complete evaporation.

pinned. The evaporation decreases the height of the droplet and the contact angle until a certain critical value, at which the triple line depins and jumps some distance ahead thus pinning again. The latter increases the contact angle above its critical value and makes the droplet taller. Afterward, this scenario is repeated periodically until complete evaporation of the droplet. The critical value of the contact angle at which the triple contact line depins is actually the contact angle of receding. One can see in Figure 5 as well that the time distance between the cycles becomes shorter during the course of the evaporation. The latter is indication of increasing impact of the line tension on the very act of depining. Similar behavior has been observed in the case of small evaporating droplets on rough Teflon surfaces. ^{53–55} It was found ⁵³ that the stick–slip regime of sliding of the three-phase contact line (TPCL) obeys the theoretical model of Shanahan-Sefiane. However, this model cannot explain why the cyclic distance between pinning and depinning of the TPCL becomes shorter upon time. A possible reason could be the fact that they do not account for the effects originating from the line tension.

The behavior of the droplets on all kinds of patterned surfaces is identical. Only the critical values of the mean contact angle are different. With such a "stick—slip" behavior of the mean contact angle upon time its critical value, at which a jump is realized, is assumed as a receding contact angle. We must confess that such behavior is not purely receding contact mode as in the case of the hydrophobized surface.

Such abrupt behavior of the contact line crossing a periodically patterned surface was already reported in the literature. ^{38,42-44} It is due to the so-called contrast of the contact line crossing both hydrophobic and hydrophilic spots. The theoretical model derived in this work is in line with the classical theory expressed in minimizing the excess free energy of droplet, which is in contact with solid substrate. However, in contrast to the classical works, ^{38,42-44} where the contribution of the periodicity of the surface on the contact line is accounted for implicitly, this work supposes delta comb periodical potential of the surface, that is, the theory in this work is much more simple and easy to exploit.

The comparison between the experimental data, the Cassie theory prediction via eq 1 and our theory via eq 7 are shown in Figure 6. The discrepancy between Cassie theory and the experiment is evident. As mentioned above, the reason is the ommiting of the effect originating from the effective line tension. The discrepancy between our theory and experimental data is less than 10% for the first three grids (40, 71, and 100

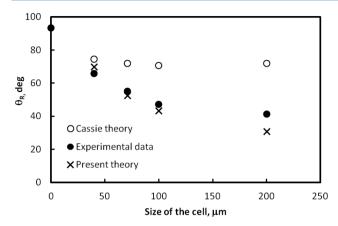


Figure 6. Comparison between the experimental data and predictions of the Cassie and present theories.

 μ m) and it is about 50% for grid size 200 μ m. Evidently this discrepancy increases with increasing the size of the cell. In Figure 7 our experimentally measured contact angles are

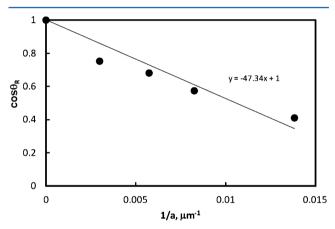


Figure 7. Comparison between the experimental data and eq 7.

presented in accordance with eq 7 as a function of the reciprocal value of the lattice parameter. It is seen that the theoretical dependence describes well the experimental observations. From the slope of the theoretical fit one can calculate the effective line tension $\kappa = (2\sigma_{LG}/\pi) \times 47.34 \ \mu m =$ 2.2 μ N, which appears to be positive. Obviously, the linear dependence (eq 7) becomes nonlinear at smaller lattice parameters, because it predicts for $a = 45 \mu m$ the contact angle of θ_2 = 93.4° for the pure hydrophobic surface. This is not surprising, because at small lattice parameters the depth of the stripes becomes important, which is not accounted for in eq 4. Hence, the present theory is applicable for relatively large lattice parameters. The obtained value of the line tension (2.2 μ N) is a typical value of line tension, which obviously does not change in the range of the lattice parameters (sum of the sizes of cells and bars) used in the present experiment.

As mentioned above the typical size of the droplets were about 2 mm in diameter. Table 2 presents the approximate number of surface patterns (square + wall) in contact with the droplet in each particular case. One can see (Figure 6) that the deviation between Cassie—Baxter theory and the experimental data increases upon reducing the number of surface patterns, which are in contact with the droplet. The reason for this discrepancy is well-known. When the droplet is significantly

Table 2. Sizes of Grids Imprinted on the Glass

No.	square side (μm)	number of surface patterns in contact with the droplet
1	40	2424
2	71	858
3	100	415
4	200	111

larger than the size of the pattern, then a large number of patterns on the wet spot are located. Under such conditions the contact angles (receding and advanced) should be affected by the average surface energies of the wet spot and the dry surface. However, when the droplet is located on a spot with larger surface patterns, the periodicity of the water/solid and air/solid surface tensions starts playing an essential role causing abrupt receding of the contact line and different equilibrium states of the droplet. One can see a larger discrepancy between the experimental data and the Cassie—Baxter theory predictions under such conditions. In particular cases, all these features are evident. In addition, the very evaporation is slow and does not affect the value of the receding contact angle.

CONCLUSIONS

The experiment conducted in the present work showed significant discrepancy between classical Cassie theory and the experimental data. For this reason a new theory accounting for the periodicity of the smooth surface and the effect originating from the line tension is developed. The value of the effective line tension obtained is reasonable.

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Notes

The authors declare no competing financial interest.

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