



# Article Wetting Patterns of Liquid-Repellent Femtosecond Laser Textured Aluminum Surfaces

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**Abstract:** Although liquid-repellent surfaces are in demand in many applications, their use is limited by the Cassie state's sustainability to environmental factors, such as the repellency of liquids with a surface tension lower than that of water. This phenomenon remains not fully understood, despite a well-developed theory. In the current work, wetting of femtosecond laser-textured aluminum surfaces for probe liquids with a surface tension of 72.8–21.2 mN/m was considered. The resulting patterns were an array of pillars at the micro level and fractal-like structures at the nano level. These structured surfaces were treated with alkoxysilanes and oleic acid. All textured samples exhibited a Cassie state with water, and contact angles greater than 150° were achieved with silane-treated surfaces. A decrease in the surface tension of the probe liquid led to a transition to the Wetzel state at 44–46 mN/m for alkoxysilanes and at 52 mN/m for oleic acid. A typical shape of the textured surface wetting curve is proposed. It was shown that the determined values of the surface tension of the Cassie-Wenzel transition were 10–15 mN/m lower than those predicted by the Cassie equations.

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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). **Keywords:** functional materials; superhydrophobic surface; laser micro-texturing; ultrashort-pulse laser machining; nanostructures; laser-induced periodic surface structures (LIPSS); water-repellent coatings; alkoxysilanes; Cassie–Baxter equation; Wenzel equation

# 1. Introduction

Nanotextured surfaces represent a promising class of functional materials due to their water-repellent properties, i.e., when the value of the static contact angle with water is more than 150° and the sliding angles are less than 10° in the Cassie state. One of the most important factors limiting the widespread use of anticorrosive [1], anti-icing [2], and self-cleaning materials is the low stability of the Cassie state under operating conditions [3]. This concerns bouncing water droplets [4,5], increased mechanical abrasion [6–8], and water vapor condensation [9]. Relatively little attention has been paid to the stability of repellant properties for liquids with a surface tension less than that of water. Although this case is associated with classical methods for determining the critical surface tension, such as the Zisman method, the full form of the corresponding curve is rarely described in the study of textured surfaces [10]. However, such curves provide information about the Cassie–Wenzel transition and can be used as a reliable indicator of liquid repellency.

The theoretical criteria for the stability of the Cassie state and the conditions for its transition to the Wenzel state [11] are described mathematically and are used to model the wetting dynamics of a surface of a given geometry [12]. This makes it reasonable to use this approach to determine the characteristic points of the wetting curve. Such a prediction, in addition to the practical use of known theoretical dependencies, is important in the development of liquid-repellent substrates with low surface tension.

In this work, aluminum substrates coated with a protective oxide layer with microand nanoscale textures were studied. The choice of material was due to its engineering significance [13] and susceptibility to femtosecond laser micro-texturing with high structure regularity and repeatability [14]. Given the intentional hydrophilicity of the oxide layer, it was further treated with alkoxysilane, its fluorinated form, and a long-chain acid to reduce the texture surface energy. Varying the texture and type of the treating agent was aimed at obtaining information about the influence of these factors on the form of the wetting curve.

Thus, there are a limited number of works describing the dependence of textured surface wetting on the surface tension of a probe liquid for a particular texture or transition type. This prevents a clearer understanding of the interaction of a textured surface with a liquid and makes it difficult to assess the stability of these surfaces. Hence, the aim of this study was to obtain characteristic wetting patterns for textured surfaces, where the Cassie and Wenzel states were expected; to determine the influence of the structure and type of treating agent on the position of characteristic points; as well as to compare experimental data with theoretically predicted values.

#### 2. Materials and Methods

The objects for investigation of the stability of textured surfaces in this work were aluminum surfaces textured by a laser at the microscale (mAl) and nanoscale (nAl). Both microand nanotexturing were performed using an air-cooled "Carbide" laser (Light Conversion) with a maximum average power of 6 W at a wavelength of 1030 nm, a pulse width of ~360 fs, a beam diameter of 1.7 mm (at  $1/e^2$  level) and a repetition rate of 60 kHz (Figure 1a). Samples with a polished surface of  $20 \times 20$  mm<sup>2</sup> and a thickness of 2 mm were made from 7500 series aluminum (inset to Figure 1a). To form fractal-like nanostructures, these samples were moved at a speed of 60 mm/s relative to the laser beam using precision X-Y linear stages (lower Aerotech PRO165LM-0500 and upper PRO165LM-0300 (Aerotech, Inc., Pittsburgh, PA, USA) in Figure 1a). In this case, the energy of the laser pulse on the sample reached ~46 µJ, and the spot diameter of the focused laser beam was ~80 µm (at  $1/e^2$  level), which corresponded to an energy density of ~0.9 J/cm<sup>2</sup> on the sample surface. For this, a spherical lens (L1) with a focal length of 100 mm was used. The laser ablation spots overlapped on the sample by ~60% with continuous scanning and a step between adjacent lines of 30 µm.



**Figure 1.** (a) Laser processing setup: M1, M2 and M3—Deflecting mirrors, L1—Focusing lens, L2 and L3—Plano-concave and plano-convex lenses of the expanding telescope. (b) Comparison of contact angles of reference probe liquids and water–alcohol mixtures on a POTS-treated fAl surface.

To engrave microtextures, the laser beam was expanded by a factor of 4 to a diameter of ~6.8 mm (at  $1/e^2$  level) in front of a lens (L1) with a focal length of 1 inch for a sharper focus of ~5 µm (at  $1/e^2$  level). To expand the laser beam, a telescope formed by a planoconcave lens (L2) and a plano-convex lens (L3) was used. In this case, the energy of the laser pulse on the sample varied from ~21 µJ to ~35 µJ, which corresponded to an energy density from ~107 J/cm<sup>2</sup> to ~178 J/cm<sup>2</sup>, respectively. The samples also moved at a speed of 60 mm/s at a laser pulse repetition rate of 60 kHz. The laser processing parameters described above were optimized in a separate investigation based on maximum process throughput under the constraints imposed by maximum power and energy of the laser source, as well as minimal contamination of the sample.

As reference surfaces, plates of degreased aluminum and aluminum after anodization (fAl) for 20 min at a current density of 2 A/dm<sup>2</sup> in a 2.4 M H<sub>2</sub>SO<sub>4</sub> solution at 10 °C were used. The surface energy of laser-processed textures was reduced by the following modifiers:

- 1H,1H,2H,2H-Perfluorooctyltriethoxysilane (POTS);
- n-Octyltriethoxysilane (OCTEO);
- Oleic acid (OA).

This choice was justified by the fact that the first is among the most common water repellents (hence, it is recognized as the most effective) [15,16], while the second is a potential replacement for the previous one, considering the fluorine-free composition, which is in favor of the environmental friendliness of future applications [17]. The last industrial modifier [18–20] in the list was chosen as a reference. The samples were repeatedly rinsed with isopropyl alcohol and dried in an oven at 80 °C for 10 min. Then, the samples were immersed in 1 wt.% isopropyl POTS/OCTEO solution for 30 min at room temperature and dried in an oven at 120°C for 30 min [21]. The OA sample was immersed in 1 wt.% isopropyl oleic acid solution for 30 min at room temperature and dried in an oven at 80 °C for 10 min [22].

The topography of the obtained samples was studied, and photographs were taken using a scanning electron microscope "MIRA3 LMU" (Tescan, s.r.o., Brno, Czech Republic). The sessile drop technique was used to determine the initial values of contact angle. For this, a goniometer with an optical microscope and a "Delta Optical HCDE-50" digital camera (Delta Optical, Shanghai, China) with the "ScopeTek View" software (version 1.0.0.1) were used.

Probe liquids used are listed in Table 1. The surface tension values are taken from [23,24]. Water–ethanol mixtures were used to increase the number of experimental points. Their surface tension was calculated from the dependencies [25] and compared with reference liquids (Figure 1b).

Liquid (CAS)	Surface Tension, mN/m (25 $^{\circ}$ C)		
Water (7732-18-5)	72.1		
Glycerol (56-81-5)	64.0		
Ethylene glycol (107-21-1)	48.0		
Dimethyl sulfoxide (67-68-5)	44.0		
Formamide (75-12-7)	58.0		
Cyclohexane (110-82-7)	25.2		

Table 1. Surface tension of probe liquids.

#### 3. Results

The surface of the mAl sample consisted of homogeneous pillars  $30 \pm 2 \mu m$  in size, spaced  $16 \pm 1 \mu m$  apart (Figure 2b). Channels about  $2 \mu m$  thick from the beam pass were clearly visible in the gaps. The top oxide layer of the pillars broke off at the edges during the engraving process (Figure 2a). Based on the analysis of SEM images, it was found that the fraction of the area of the residual oxide layer from the area of the base of the pillars was



 $26 \pm 2\%$ , i.e.,  $12 \pm 1\%$  of the total projection area. The surface of the pillars also contained texture due to shrinkage and material fragmentation during processing.



**Figure 2.** Laser-textured surface structures on 7500 series aluminum plates. (**a**,**b**) Rectangular micropattern (mAl);.(**c**,**d**) Nanotexture (nAl).

The obtained nanopattern in the strict sense could not be characterized as a laserinduced periodic surface structure (LIPSS), since it did not contain periodically repeating texture elements. Although at the microlevel (Figure 2c), it was quite homogeneous and contained branching grooves ~2  $\mu$ m in size, upon closer examination (Figure 2d), the groove walls and the surrounding surface were formed by merged texture elements with characteristic sizes of about 80–120 nm.

Water repellents provided an increase in the contact angles (Figure 3), confirming that the water-repellent properties of the textures were formed due to both the surface geometry and its chemical composition.



Figure 3. Surface water contact angles of treated textures.

The nanotextured surface provided superhydrophobic water repellency with all treatments. POTS provided superior hydrophobicity on all surfaces, while OA and OCTEO exhibited nearly indistinguishable water contact angles for nAl. For the mAl sample, OCTEO proved to be more efficient. An interesting observation was the reduced contact angle of the OA sample with a flat surface compared to the untreated one due to the lack of orientation of its molecules on the surface. OA samples were not used in further experiments due to the low resistance of the treated layer.

The dependence of the static contact angle on the surface tension of the probe liquid (Figure 4) had a characteristic form: a gradual decrease to ~48 mN/m and a subsequent sharp decay. The next sloping section of the curve in the range from 35 to 20 mN/m could be extrapolated to the value of the critical surface tension of the modifier. Also noteworthy was the intersection of the curves for the textured and flat surfaces.



**Figure 4.** Contact angles of a probe liquid with variable surface tension on aluminum surfaces chemically treated with (**a**) OCTEO; (**b**) POTS. (black dot line is tangent to curve).

# 4. Discussion

#### 4.1. Determining the Distribution of the Liquid Contact Surface

The use of the classical Cassie–Baxter Equation (1) makes it possible to estimate the fraction of the material surface that interacts with the liquid.

$$\cos\theta_{\rm app} = f_1 \cos\theta_1 + f_2 \cos\theta_2 \tag{1}$$

where  $cos\theta_{app}$  is the contact angle of a two-phase heterogenous surface,  $f_1$  and  $cos\theta_1$  are the surface fraction and the contact angle of the first phase, and  $f_2$  and  $cos\theta_2$  are the surface fraction and the contact angle of the second phase (air).

Let us assume that the second phase is air, and make a deliberately controversial assumption that  $f_2 = 1 - f_1$ , which, as shown by Milne [26], can be conditionally recognized as correct if the structure of the sample with rectangular pillars (Figure 2a,b) is close to that of the theoretical model. In this case, the proportion of the phase in contact with the liquid will be calculated according to Equation (2):

$$f_1 = \frac{\cos\theta_{\rm app} + 1}{\cos\theta_1 + 1} \tag{2}$$

In practice, the proportion of the contacting surface depends on both the type of structure and the modifier (Table 2). The smallest value of the contact area was achieved for the nanotexture, and the most effective modifier was POTS.

Modifier —	Water Contact Angle, Deg			Wetted Surface Percentage	
	fAl	mAl	nAl	mAl	nAl
OCTEO POTS	94 99	144 160	152 164	0.21 0.14	0.07 0.07

Table 2. Wetting parameters of the studied surfaces with water.

The dependence of the fraction of the surface in contact with the probe liquid, depending on its surface tension, is shown in Figure 5. It is obvious that the surface of the columns (Figure 2a) contained the remains of an anodizing layer. These fragments, in fact, were topographical elements in contact with the liquid. The share of their area in the total surface area of the sample was 12.6%, which corresponded to the value of  $f_1$  at the level of 0.126, if the surface of the pillars is assumed to be flat, and this value corresponded, within the error, to the rectilinear segment of the stable Cassie state for mAl in Figure 5.



**Figure 5.** Wet surface fraction  $f_1$  from Equation (2), calculated using the contact angles of a series of liquids with different surface tension. (red dot line is the percentage of solid-liquid contact).

For the nAl samples, the  $f_1$  value corresponded to 0.07 according to Figure 5. However, if it was assumed that in Equation (2)  $f_1 \neq 1 - f_2$ , then one should expect an underestimation of the experimentally obtained value of  $f_1$ , since the geometry and roughness of the wetted surface with a double structure are not taken into account. Such structures could potentially be more resistant to wetting caused by vapor condensation, as shown by Shi et al. [27]. Since the parameter  $f_1$  for nAl samples cannot be compared with any simple geometric model, its significance lies only in illustrating the higher water repellency of the sample than in the case of microtextures.

On the other hand, the use of this Cassie model modified in accordance with the surface geometry can be effective in the study of non-stochastic structures obtained by laser processing [28], anisotropic chemical etching of the silicon surface [29], etc.

#### 4.2. General Shape of the Zisman Plot for Textured Hydrophobized Surfaces

The well-known theoretical apparatus, starting with the classical equations of Zisman, Cassie, and Wenzel and ending with modern works on the thermodynamic conditions for the stability of the Cassie state, makes it possible in the general case to determine the characteristic form of the wetting curve with a change in the surface tension of the probe liquid. However, the existing discrepancies in the interpretation of theoretical calculations and the abundance of critical works require practical verification.

The characteristic appearance of wetting curves for textured surfaces includes several critical points. Indeed, in the range of surface tensions of 50–72 mN/m, on the microand nanotextured surfaces, the liquid remains in the Cassie state, and below these values, the transition to the Wenzel state occurs. Taking also into account Zisman's postulate [30] that the critical surface energy is equal to the surface tension of the probe liquid that completely wets the surface, then  $\theta = 0^{\circ}$ . This assumption allows one to determine the coordinates of the point where the curve intersects the x-axis. For convenience, the wetting curves of the nanotextured surface were plotted in standard Zisman coordinates (Figure 6). The presence of this transition makes it possible to establish extreme states that will determine the characteristic shape of a whole family of such curves. These extreme points include: A—a point corresponding to water as a probe liquid; B and C are the beginning and end points of the transition from the Cassie state to the Wenzel state; and D is the point of complete wetting of the water repellant layer (equivalent to the value of critical surface tension in the Zisman plot technique).



**Figure 6.** Wetting curves of the nanotextured aluminum surface in Zisman coordinates, characterized by specific points: A—water wetting; B—the repellence threshold; C—complete extrapolated wetting; D—complete wetting. (the blue dot line is tangent to the B-C segment, and the black dot line is tangent to the A-B and C-D curve segments).

#### 4.3. Point A Position

To predict the position of point A—the wetting angle of textured surfaces with water, theoretical approaches were developed after the classical works of Cassie and Wenzel, the evolution of which is described in [31,32]. However, their accuracy and applicability are limited by the difficulties in obtaining the exact geometric parameters of the structures.

#### 4.4. Point B Position

A clear algorithm for determining the position of point B has not yet been formulated, although it is of great practical importance for establishing the stability of the Cassie state for liquids with reduced surface tension. To develop it, it is important to consider the conditions of thermodynamic stability of the Wenzel and Cassie states [11,12] for model surfaces with different geometrical parameters of the structure. In accordance with the differential energy balance [33], the general form of the stability parameter can be written as:

$$\cos\theta_{\rm c} = \frac{-f_2}{r - f_1} \tag{3}$$

where  $\theta_c$  is the critical intrinsic contact angle of the surface material. It should be noted that if the contact angle  $\theta$  of the probe liquid on a non-textured surface is  $\theta < \theta_c$ , the Wenzel state is preferable; if  $\theta > \theta_c$ , the Cassie–Baxter state is preferable. The value of  $f_1$ , in fact, is not equal to  $1 - f_2$ , as reasonably stated in [26], and may even exceed 1.0 with a well-developed contact surface. Equation (3) is quite difficult to practically apply to statistical structures; however, with a certain degree of error, it can be used for the obtained microstructure (Figure 2a,b). Let us consider (Table 3) the state in which the probe liquid covers the entire surface area of the pillars, but the space between them remains non-wettable.

**Table 3.** Calculation of  $\theta_c$  for an idealized microtexture.

% Wet Surface Area of the Pillar Tip	$f_2$	$f_1$	r	$\theta_{\rm c}$ (Equation (1))	$\theta_{c}$ Experimental
100	0.52	0.48	1.77	113	93–98
26	0.87	0.13	1.39	133	

Thus, in the case of the considered microtexture for the Cassie state, according to Equation (2), the intrinsic contact angle of a flat surface should be much higher than its experimentally determined values. This fact can be explained by the inaccuracy of applying Equation (2) to real structures. However, it should be noted that the minimum value that can be obtained for  $\theta_c$  is 90°. As can be seen from the experimental results (Figure 4a,b), the wetting angles on the corresponding flat surfaces are much less than 90°. Similar results were obtained in [21], when contact angles of less than 90° were achieved after POTS treatment of a flat aluminum surface with a decrease in the surface tension of probe liquids. Then, the textured surface retains a stable Cassie state, which cannot be predicted using the  $\theta_c$  criterion, as in the case of other amphiphobic [34] and omniphobic [35] surfaces.

#### 4.5. Positions of Points C and D

The possibility of using the extrapolation of the descending part of the curve by analogy with the Zisman method [30] postulates the equality of the critical surface energy and the highest surface tension of a fully wetting liquid. It should be noted that the direct use of this method is limited, firstly, by the presence of the surface texture and, secondly, by the polar component of the used probe liquids [36,37].

The position of point D for non-textured surfaces (Figure 7) can be found at the intersection with the horizontal axis at points close to the values of the critical surface energy of polymers corresponding to modifiers: polydimethylsiloxane—19–21 mN/m [38] and polytetrafluoroethylene—20.2 mN/m [39].



**Figure 7.** Zisman plots after surface treatment with (**a**) OCTEO and (**b**) POTS; B—the Cassie-Wenzel threshold; C—extrapolated complete wetting point for textured surfaces (the blue dot line is the tangent to the B-C curve segment and the black dot line is the tangent to the A-B and C-D curve segments).

Extrapolation of the descending part of curves B–C (Figure 7) for flat surfaces at the intersection with the horizontal axis gives the position of point C: in the case of OCTEO, it is 39.0 mN/m for both textures, and in the case of POTS, it is 39.4 mN/m for nanotexture and 33.2 mN/m for microtexture. The decrease in contact angles compared to flat surfaces in this area can be explained by the Wenzel equation [40], which takes into account the surface roughness (4):

$$\cos\theta_{\rm app} = r \times \cos\theta_0 \tag{4}$$

where  $cos\theta_{app}$  is the wetting angle of the textured surface,  $cos\theta_0$  is the wetting angle of the corresponding flat surface, and *r* is the roughness parameter, which is the ratio of the total surface area to its projection.

Under complete wetting conditions (5),  $\theta_{app} = 0$ ,  $cos\theta_{app} = 1$ , that is:

r

$$=\frac{1}{\cos\theta_0}\tag{5}$$

Since for liquids with a surface tension of 39.0 mN/m ( $cos\theta_{app} = 1$ ), the contact angle of a flat surface treated with OCTEO was  $42 \pm 2^{\circ}$ , it was found that the roughness parameter r in this case was  $1.35 \pm 0.042$ . For POTS, it was  $39 \pm 2^{\circ}$ , which means  $r = 1.29 \pm 0.036$ .

Thus, we can conclude that the prediction by the Wenzel equation for the systems under study is rather inaccurate, and the roughness parameter obtained with its help is in most cases underestimated, especially for nanotextures with an expectedly more developed surface. This can be explained by the unaccounted for effect of capillary wetting, the rate of which for porous surfaces is described by the Washburn equation and is large enough to introduce a measurement error (an example of the temporal dependence of the capillary wetting rate can be found in [41]).

### 5. Conclusions

It has been established that for chemically hydrophobized micro- and nanotextured aluminum surfaces, the dependence of the static water contact angle on the surface tension of probe liquids forms an S-shaped curve with certain inflection points. Point A corresponds to water as the probe liquid; B and C are the beginning and end Cassie–Wenzel state transition, and point D is of complete wetting. Of practical interest is the position of point B, corresponding to the beginning of the Cassie–Wenzel transition, which determines the resistance of the surface to wetting by liquids with reduced surface tension. Although the position of point B changes negligibly with texture scale, it is modifier-dependent,

corresponding to 44 mN/m for siloxane and 49 mN/m for oleic acid repellents. The experimentally obtained values for the surface tension of the probe liquid at this point are underestimated compared to those predicted from the energy balance and the Cassie–Baxter equations. Furthermore, the configuration of the C–D area of the experimental wetting curve cannot be described by the Wenzel equation. However, the Cassie–Baxter equation accurately determines the solid and gas fractions on the contact surface under the droplet. In the considered microtexture, 12% of the solid–liquid interface, corresponding to the projection of the surface area of the tip, ensures the stability of the Cassie state at surface tensions of the probe liquid above 44 mN/m.

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