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Laser texturing to control the wettability of materials

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- Invited Paper -

Abstract

Many applications of different materials are related to the properties of their surface. Wettability is a key property affecting applications in all fields: adhesives, lubricants, detergents, all types of coatings, implant integration, heat transmission, corrosion, etc. Laser texturing has been demonstrated to be an excellent technique to modify surface wettability of many different materials: polymers, metals, ceramics, or even natural stones. The relative simplicity and robustness of the results, together with the widespread availability of affordable industrial laser sources made laser texturing a very promising tool for modifying the surface of parts in manufacturing plants. In this paper we introduce the basics of the technique and show some examples of applications. On one hand, treating the surface of different polymers for biomedical applications. And on the other hand, the production of surfaces with extreme wettability properties is shown: superhydrophilic, superhydrophobic and omniphobic surfaces were obtained by laser texturing.

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1. Introduction

Wettability is the ability of one fluid to spread on, or to adhere to a given surface. This property is relevant in many physical, chemical or biological processes (such as in tribology, pipe flow, adhesion of dust to surfaces, cell or bacteria-surface interactions, etc.) [1]. Hence, the control of this property on the surface of different materials is relevant in science and industry and can find many applications, such as the production of surfaces with self-cleaning properties, reduced viscous drag, improved lubrication properties, or better osseointegration [2].

Wettability is mainly controlled by the surface topography and chemistry of materials [1,3]. Surface topography refers to both the profile shape and the surface roughness (including the waviness and the asperity or the finish).

In order to tailor the topography or chemistry of surfaces, many techniques have been explored (for example, application of paints and coatings [4], chemical etching [5], electrochemical machining [6,7], lithographic techniques [8–10], sandblasting [11], etc. However, these techniques have some limitations, some of them only can modify either the topography or the chemistry, others are limited to small areas, in others the modification is not local or not valid to treat large surfaces, etc. Recently, laser texturing has become one of the most popular techniques to modify the surface topography or chemistry to modify the surface wettability [12,13]. This technique is able to create a wide variety of surface topographies, both at the micro and nanoscales in many materials, with large repeatability, with excellent control of the surface features shape and size, and with a minimum

affectation of the bulk material (then, making this technique attractive to treat materials sensitive to mechanical damage). On the other hand, this technique is also able to modify the surface chemistry of some materials (e.g. polymers) as well as the topography. Another advantages of this technique are the non-contact nature of the process (relevant to avoid the contamination of surfaces), the high processing speed, the easy automation, the possibility to treat large areas, and the environment friendliness.

In this paper, we will review the fundamentals of this technique and the potential application of laser texturing for the production of surfaces with improved biomedical properties (mainly from the point of view of wettability) or with extreme wetting characteristics.

2. Wettability fundamentals

Wettability is the ability of a specific liquid to maintain its contact with a particular solid surface. Therefore, it depends on the nature and chemistry of both liquid and surface. This is a balance between the intermolecular adhesive (liquid to surface) and cohesive (liquid to liquid) interactions [14].

This property is commonly expressed by the contact angle (CA), i.e., the angle formed by the interface liquid-vapor (typically water is the test fluid) with a solid surface (as depicted in Figure 1) [15,16]. When a solid surface has a high affinity to the liquid the water easily spreads on the surface and the CA is low (e.g. when the test fluid is water, these surfaces are called hydrophilic, but in the case of oil, they are named oleophilic). In the opposite case, the liquid does not spread and forms, at equilibrium, a spherical cap resting on the surface of the material; then the CA is large, as shown in Figure 2 (when the test fluid is water, these surfaces are called hydrophobic, while they are named oleophobic for oil).

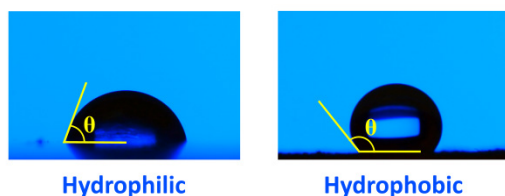


Fig. 1. Contact angle for hydrophilic ($\theta < 90^\circ$), and hydrophobic materials ($\theta > 90^\circ$).

The CA is relevant for static situations. Another relevant wetting properties used when we have dynamic phenomena are the contact angle hysteresis (defined as the difference between the advancing and the receding contact angles) and the roll-off angle (angle at which a water drop rolls off a tilted flat surface) [17–19]. These parameters are especially relevant to determine the roll-off of droplets along superhydrophobic surfaces (and hence the lateral adhesion of the droplet to the surface).

Surface topography have a large impact on the wetting of surfaces. Two main approaches have been developed to treat the influence of the roughness of any surface on its wetting characteristics. Wenzel obtained a relation between roughness and wettability:

$$\cos(\theta^*) = r \cos(\theta) \quad (1)$$

where θ^* is the apparent contact angle (that for a textured surface), r is a measurement of the roughness (ratio of true area of the solid surface to the apparent area), and θ is the Young's contact angle (i.e. that for an ideal and untreated surface) [20]. This equation shows the amplification of the intrinsic wettability of a material with the surface roughness. Then, the increment of the roughness in a hydrophobic surface make it even more hydrophobic, and vice versa, it is chemically hydrophilic.

Wenzel's model assumes that the liquid penetrates into the roughness grooves (see Figure 2); however, in some cases, the liquid is not able to penetrate into the surface features, and this equation no longer applies. Cassie and Baxter addressed this problem, and assumed that the liquid does not penetrate into the surface features, as depicted in Figure 2 [21]:

$$\cos \theta^* = f_1 \cos \theta - f_2 \quad \text{with } f_1 + f_2 \geq 1 \quad (2)$$

where f_1 is the total area of the surface under the droplet per unit projected area under the droplet, θ_1 the contact angle on a smooth PTFE surface, and f_2 the total area of air under the droplet per unit projected area under the droplet (sometimes, this equation is commonly expressed as $\cos \theta^* = f \cos \theta - (1 - f)$; however, this is only valid if coplanar liquid-vapor and solid-liquid interfaces are present, which is not always the case [22]). In a simplified way, the increase of the roughness increases the amount of air pockets trapped into the micro- and nanopopography; then, the fraction f_2 increases, and also the apparent contact angle θ^* .

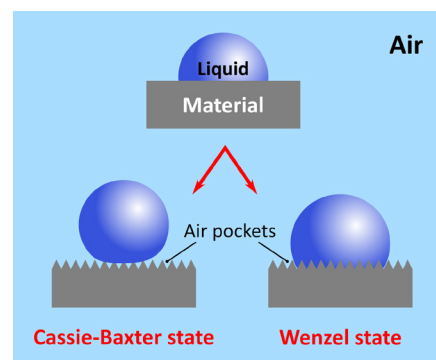


Fig. 2. Scheme of the influence of the roughness on the wettability under the framework of the Wenzel and Cassie-Baxter models.

The main result of this model is that the presence of air pockets tends to increase the hydrophobicity of the material, independently of its intrinsic wettability.

In general, we observe that Cassie-Baxter's model is more appropriated for very rough surfaces, while Wenzel's model for surfaces with low roughness [23].

Surface chemistry also plays a relevant role on the wettability of surfaces. The modification of the surface chemistry to induce, for example, the presence of polar or charged functional groups (for example, by anchoring in the surface OH⁻ groups) is a widely used to increase the wettability of polymers [24]; on the contrary, the surface

fluorination of surfaces groups substantially decreases the wettability [25,26].

Then, this simple analysis indicates us the way to tailor the wettability of different surfaces, only by changing the surface roughness.

3. Process fundamentals

Laser texturing (also called laser surface texturing, laser structuring, or laser patterning), is based on the direct treatment of surfaces with a laser beam [27]. This process relies in the localized removal (or ablation) of material by the direct action of a focused laser beam.

The removal or the change in the surface chemistry mainly depends on the characteristics of the laser radiation used in the treatment (wavelength, laser irradiance, pulse length, etc.), but also on the processing atmosphere (gas or liquid), and the nature and properties of the material. The modification of the surface chemistry with lasers, in terms of the formation or attachment of chemical groups, is mainly performed on polymeric surfaces; however, metals and ceramics can also be subjected to a chemical modification during or after the laser treatment due to the large free energy in the newly formed surface.

Modification of the surface topography during laser texturing is performed by the creation of regular or irregular patterns of grooves, bumps, dimples, ripples (laser-induced periodic surface structures or LIPSS), etc. as depicted in Figure 3 [27,28]. These structures are produced by the localized removal or ablation. The specific phenomena involved in the formation of these structures, and the influence of the processing parameters is complex as it depends on the specific approach (e.g., photo-thermal or photo-chemical ablation of the material, laser swelling or bumping, laser grooving, etc.), the laser characteristics but also the nature of the material. These are beyond the scope of this work, and we refer the interested reader to more specialized works (e.g., [29–33]).

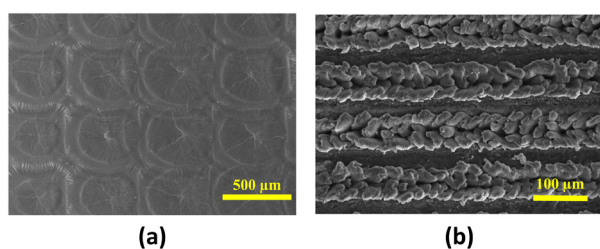


Fig. 3. Typical features produced during laser texturing (a) dimples (produced in titanium with a Nd:YAG laser) and (b) grooves (produced in stainless steel with a frequency-doubled Nd:YVO₄ laser).

Laser texturing of surfaces can be performed in the UV–IR spectral range. The nature of the material to be treated mainly determines the most suitable laser radiation, although different laser sources can be applied to each material; however, the surface quality of the texture is greatly improved by using short-wavelength laser radiation. On the other hand, continuous-wave (CW) [34–36] or pulsed laser radiation can be used in this treatment [37]. In general, operation in CW mode produces surface textures with low quality, and large thermal effects around the laser-treated area; moreover, a large

amount of debris is easily formed. Hence, this processing mode is not commonly applied. On the contrary, pulsed laser radiation (ranging from microsecond to femtosecond pulse duration) produces finer patterns, especially if ultrashort (picosecond or femtosecond) laser pulses are applied. In this case, the thermal effects are completely or almost suppressed, and the debris formation or the thermal damage of the material close to the radiated area is absent or negligible [38].

Typically, laser texturing is performed in atmospheric conditions, and the control of the processing atmosphere is not required. However, it should be pointed out that different works showed that the influence of the processing atmosphere (gas or even in liquid) can be relevant, as they can influence on the surface chemistry. For example, Guan et al. showed that the processing atmosphere can promote or slow down the wettability transition of laser textured metallic surfaces [39]. Pflöging et al. observed a marked increase in the wettability of PMMA using O₂ (instead of He) as processing gas due to the oxidations of the surfaces [40]. Wang et al. showed that laser texturing of Ti6Al4V can be dramatically reduced if the process is performed in ethanol as a consequence of the final roughness and the carbon content of the surface [41]. However, few other works have paid attention to this parameter, and its influence on the final wettability of the treated surfaces.

4. Laser techniques and sources for surface texturing

Two main approaches are followed to tailor the texture of surfaces with laser: (1) using a stationary laser beam, or (2) with a moving laser beam. The first approach involves the exposure of the surface to an unfocused laser beam. In this case, quasi-periodic microstructures (e.g. ripples, cones or pillars) or LIPSS are formed in the irradiated area (after one or several laser shots) on almost any material (metals, semiconductors, and dielectrics) [42]. These are commonly produced using excimer or ultrashort laser and for a wide range of processing parameters (e.g. using CW processing mode, or for a large range of pulse lengths, from millisecond up to femtosecond pulses).

In certain cases, stationary beams are used in combination with a mask to produce a desired pattern. In this case, a short laser pulse (required to obtain high peak powers) is used to produce the pattern. Pulsed lasers (such as excimer, femtosecond, or TEA CO₂ lasers) are used in this case; however, this approach is not commonly applied to tailor the wettability of materials as it is less flexible, and time consuming due to the requirement of a mask. Another approach involving the utilization of a stationary laser beam involves the interference or two or several laser beams to produce periodic variations of the laser intensity over the processed region. This approach, called laser interference patterning can produce hierarchical structures. However, the application of this technique is also restricted as a more complex processing setup is required.

The second method to texture the surface of materials with laser, involves the utilization of a moving laser beam. In this method, the relative motion between the laser beam and the surface can be obtained using a Cartesian system (to move the laser beam or the surface) or two galvanometer mirrors (to deflect the laser beam over the surface). The last approach is

the most commonly method in laser texturing due to the larger flexibility, and processing speeds. In both cases, CW or pulsed lasers are used.

Laser sources most widely used for texturing applications are Nd:YAG (or Q-switched Nd:YAG sources) [37], Nd:YVO₄ (even frequency doubled or tripled) [43], femtosecond [44], or pulsed fiber [45] laser sources. However, CO₂ [46] or excimer [47] lasers have also been used. In general, laser texturing involves the utilization of sources with low laser power ($P < 200$ W) as only a thin layer of the surface must be treated.

5. Applications of laser texturing for the modification of the wetting properties of materials

5.1. Laser surface texturing of polymers for biomedical applications

Surface properties are the key aspect when considering material use for biomedical applications. The way the cells interact with an implant material depend of its chemical composition, surface energy, surface topography, surface roughness, and surface wettability [48]. Apart from metals and bioceramics, polymers are the main group of materials used for biomedical applications, including polypropylene (PP), polyetheretherketone (PEEK), ultra-high-molecular-weight polyethylene (UHMWPE), or acrylic bone cements (PMMA). Lasers are excellent tools for modifying surface wettability of polymers [13,30].

Figure 4 shows the change on surface wettability of PEEK by Nd:YVO₄ laser texturing using different wavelengths. Surface wettability can be tailored from hydrophilic (at 355nm) to hydrophobic (when treated with 532 nm) [43]. It was established a direct relationship between the roughness, wettability, and the formation of polar groups with the increased biocompatibility of laser-treated PEEK surfaces [49].

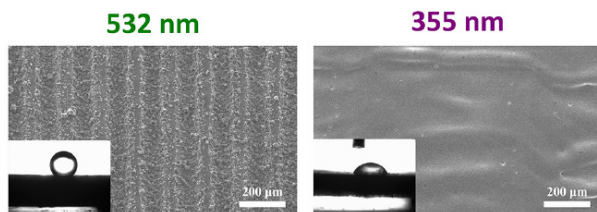


Fig. 4. PEEK Laser surface texturing allows obtaining hydrophobic surfaces (at 532nm) or hydrophilic (at 355 nm). Adapted from [43].

While PEEK is quite sensitive to the wavelength of the laser used for surface texturing, other polymers such as UHMWPE, are almost transparent to visible and near infrared radiation. Therefore, applying a black carbon coating is a good way to increase the response of the polymer to the laser radiation [50]. As can be seen in Figure 5, surface wettability can be also modified by the laser treatment, even if those changes are no so big as those found with PEEK.

Surface wettability of other polymers such as PP can also be modified by laser texturing. Using a KrF excimer laser (248 nm) is a good tool to promote the formation of functional groups on the surface of PP [51]. Their presence explains the increase in contact angle after the laser process. Oriented

topographies show enhanced cell viability on PP than on non-treated surfaces [52].

Polycarbonate (PC) is an amorphous thermoplastic polymer exhibiting high transparency to visible light, that is being widely used as biomaterial from cardiac devices to renal applications. Laser surface texturing can improve fibroblast attachment and proliferation by treating the surface with 1064nm radiation from a Nd:YAG source [53].

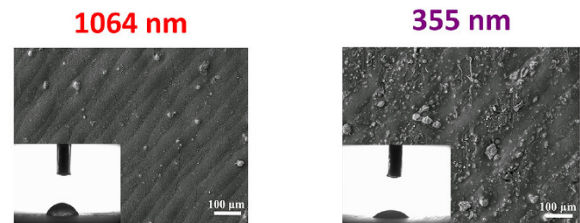


Fig. 5. UHMWPE laser surface texturing under different wavelength allows increasing wettability in a 25% which is beneficial for cell attachment. With permission [50].

5.2. Production of surfaces with extreme wettability

Laser texturing can promote extreme changes of surface wettability under specific processing conditions. Changes induced on the topography at micro- and nanoscale, as well as surface chemistry are responsible for dramatic changes of surface wettability: a hydrophilic surface can be converted on superhydrophilic or superhydrophobic just by changing processing conditions.

Figure 6 shows the water contact angle of AISI 304 stainless steel before the laser treatment (Figure 6a), after laser treatment promoting superhydrophilic behavior (Figure 6b), and after laser texturing producing a superhydrophobic surface (Figure 6c). The most interesting fact is that both treatments, leading to these extremely different wettability behaviors, were obtained using the same type of laser (a Nd:YVO₄ laser working at 532 nm), but using different processing atmospheres. This points out the importance of both surface topography and surface chemistry on the wettability of materials [54].

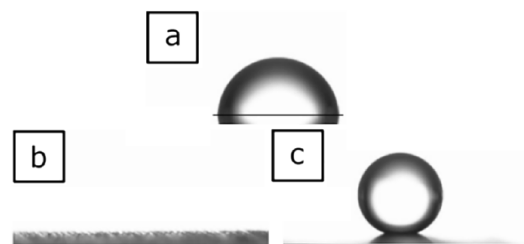


Fig. 6. Comparison of the wettability of (a) base material (AISI 304 SS) with $\theta = 88 \pm 2^\circ$, (b) superhydrophilic surface with $\theta = 0 \pm 2^\circ$ and (c) superhydrophobic surface with $\theta = 152 \pm 4^\circ$. Surface (b) was treated in air atmosphere, while surface (c) was obtained in argon atmosphere. With permission [54].

The different behavior of these surfaces to water contact can be illustrated by observing the falling of a drop of water under a high speed camera. Figure 7 shows 5 frames of a video recording in which it can be noticed the different behavior. While on the untreated material, the drop spreads to form a typical lentil like shape (Figure 7a), on the superhydrophilic surface the water spreads completely on the surface to almost

disappearing (Figure 7b). And on the superhydrophobic surface, the water drop bounces several times before resting on the surface (Figure 7c).

Most of the research works in the field of surface wettability modification are devoted to water-surface interaction. But studying the behavior of the wettability of a material with regards to oil or other organic liquids is also very interesting taking into account the widespread presence of these hydrocarbons in industrial and domestic environments. Figure 8 shows the modification of the wettability of a Polytetrafluoroethylene (PTFE) surface after treatment with a CO₂ laser source emitting at 10600nm [55].

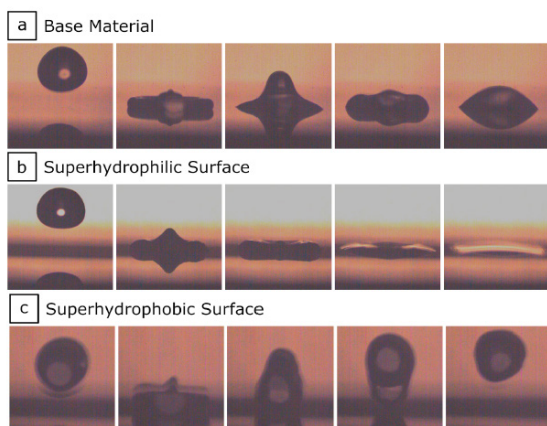


Fig. 7. Comparison of the impact of a water drop on the three surfaces shown in figure 5: base material (a), superhydrophilic surface (b) and superhydrophobic surface (c). With permission [54].

PTFE is a synthetic fluoropolymer well known to have excellent hydrophobic properties. But the beauty of the laser texturing is that we can even increase its properties to repel the water making the surface superhydrophobic, as shown in Figure 8. Moreover, the laser texturing treatment converts the surface from oleophilic into oleophobic. The same behavior was found with mixtures of water and ethanol (at 40 and 20% respectively). In these last cases, the increment in contact angle is quite large, reaching values of 60%.

Typical chemical treatments used to increase hydrophobicity and oleophobicity of surfaces are lost due to the attack by acids or strong base substances [56]. In order to demonstrate the persistence, the wettability modification achieved by laser texturing, PTFE treated samples were submitted to attack by strong acid and base substances.

Figure 9a shows that the laser textured PTFE surface remains very stable, in presence of a strong acid substance: aqueous HNO₃ (pH = 1), as well as in the presence of a strong base substance: aqueous NaOH (pH = 14). Liquid drops show typical spherical shape as well as pure neutral water (pH = 7) when lying on the laser textured PTFE surface [55].

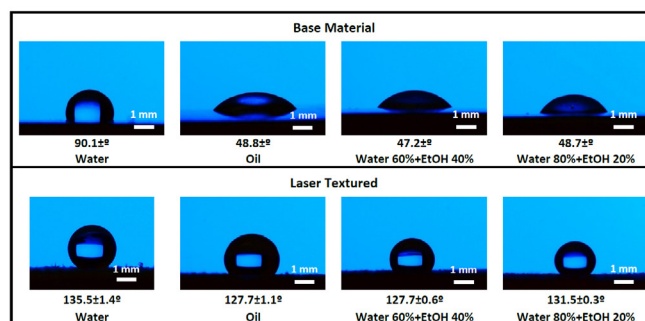
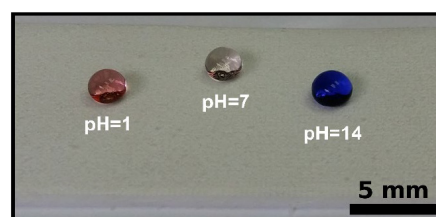
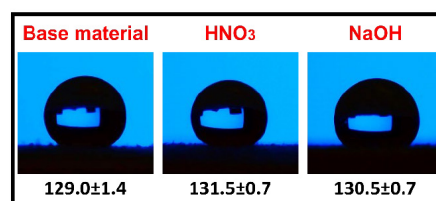


Fig. 8. Maximum contact angles for the base material and the laser textured PTFE surface, as a function of the test fluid: water, oil, water 60%+EtOH 40% and water 80% + EtOH 20%. With permission [55].



(a)



(b)

Fig. 9. a) Images of different water droplets (pH=1, 7, and 14) on the laser textured surface of PTFE. b) Contact angles of a water droplet on a PTFE laser textured surface after being immersed into a HNO₃ (pH=1) and NaOH (pH=14) solution for 2h. With permission [55].

Moreover, after immersion of the laser textured PTFE material in strong acid (HNO₃) and strong base (NaOH), the superhydrophobic behavior was not destroyed (as seen in Figure 9b). This corroborates the advantages of laser texturing versus chemical treatment with regard to surface wettability modification [55].

Conclusions

Laser texturing is a quite versatile technique for modification of wettability of the surface of different materials.

Laser texturing allows converting surfaces into superhydrophobic or superhydrophilic just by tuning the processing parameters adequately. Comparing with other techniques, this is a robust method, that allows treating materials as different as polymers or metals.

Laser texturing allows converting the surface of PTFE into an omniphobic material, repelling water, oil, or different water-ethanol mixtures, and this behavior remains stable even after base or acid attack.

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