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## Laser texturing of superhydrophobic surfaces on stainless steel: influence of storage conditions on the anti-wetting transition

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### Abstract

The superhydrophobic properties of natural surfaces (e.g. lotus leaves), have been a key driver behind research focused on laser texturing to provide such properties on metallic surfaces via the creation of similar topological features. Anti-wetting surfaces have associated self-cleaning and antibacterial characteristics and hence the ability to controllably create such surfaces on metal parts would be beneficial in many industrial sectors, such as food, medical, and aerospace.

In this paper we focus on creating superhydrophobic surfaces on thin sheets of SS316L using a nanosecond pulsed fibre laser, with typical parameters of 220 ns pulse duration, scanning speed of 150 mm/s and repetition rate of 25 kHz. As observed by a number of researchers, immediately after laser processing the textured surfaces display hydrophilic properties, with some of these surfaces subsequently transitioning to anti-wetting over a period of days or even weeks. The duration of this transition (and the ultimate degree of hydrophobicity) depends not only on the laser texturing parameters, but also on the sample storage conditions. In this paper we present a study of the impact of both laser scanning parameters (line separation, pulse energy) and storage conditions on the degree of hydrophobicity, by periodic measurement of the contact angle of the textured samples.

Keywords: laser texturing, hydrophobicity, superhydrophobic, antiwetting

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### 1. Introduction

In recent years, laser generation of superhydrophobic surfaces has received increased interest from various research groups. The main driving force is the possibility of industrial applications of such surfaces providing self-cleaning (Fürstner, Barthlott, Neinhuis, & Walzel, 2005) and anti-icing properties (Kulinich, Farhadi, Nose, & Du, 2011). Such surface properties can be found in nature, e.g. the lotus leaf exhibits such

qualities. The combination of the leaf microstructure and its epicuticular wax crystals enable anti-wetting and self-cleaning properties (Barthlott & Neinhuis, 1997). By using laser processing it is possible to mimic the lotus leaf surface structures. The majority of research has been focused on using ultrafast lasers to produce such structures (Vorobyev & Guo, 2013), but nanosecond lasers can also be used (V. D. Ta et al., 2016). In this paper we focus on using a nanosecond fibre laser to create superhydrophobic surfaces on stainless steel 316L.

Immediately after laser texturing, the surfaces exhibits hydrophilic properties, that with time transition to hydrophobic (contact angle between  $90^\circ$  and  $150^\circ$ ) or even to superhydrophobic (contact angle  $>150^\circ$ ). Note however that the static contact angle is a necessary, but not sufficient indication of superhydrophobicity; the formed droplet must also readily roll off the surface (V. D. Ta et al., 2016).

The transition between hydrophilic and superhydrophobic states is via a series of surface chemistry changes. During laser processing, a layer of oxides is created on the surface which are hydroxylated due to the water molecules present in the air. These hydroxylated oxides enable absorption of hydrocarbons resulting in superhydrophobic behavior (R. Diaz, Marimuthu, & Ocaña, 2017). There are many contributing factors to the anti-wetting transition such as the material used (Jagdheesh, García-Ballesteros, & Ocaña, 2016), storage conditions (Ngo & Chun, 2017), laser parameters and scanning strategy (D. V. Ta et al., 2015).

In this paper we focus on creating superhydrophobic surfaces on thin sheets of stainless steel 316L using a nanosecond pulsed fibre laser, using typical parameters of 220 ns pulse duration, scanning speed of 150 mm/s and repetition rate of 25 kHz. We present a study of the impact of both laser scanning parameters (line separation, pulse energy) and storage conditions on the degree of hydrophobicity, by periodic measurement of the contact angle of the textured samples.

## 2. Experimental procedures

A nanosecond pulsed fibre laser was used for all experiments (SPI 20 W EP-S). The laser beam was focused down to a spot of  $26\text{ }\mu\text{m}$  using a 160 mm f-theta lens. The pulse duration used was 220 ns, at a repetition rate of 25 kHz. The beam was scanned across the surface of the sample using a galvanometer scan head. Each sample was textured using two laser passes at  $0^\circ$  and  $90^\circ$ . The laser processed samples were imaged using an Alicona Infinite Focus profilometer.

The static contact angle of the droplet was measured for deionized water using a bespoke contact angle measurement setup, and droplets with a volume of  $\sim 15\text{ }\mu\text{L}$ . Images of the droplets were analyzed using an ImageJ software package (Stalder, Kulik, Sage, Barbieri, & Hoffmann, 2006).

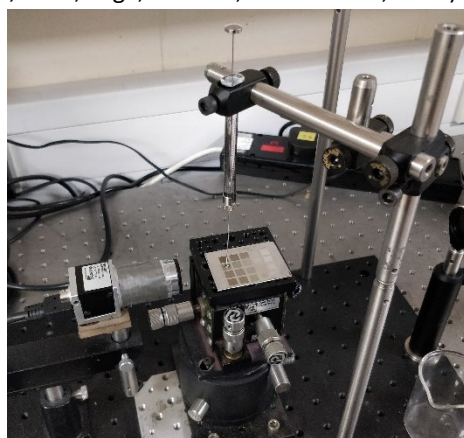


Fig. 1. Contact angle measurement setup

### 3. Results and discussion

#### Effect of average laser power and line-to-line spacing

Three different laser average powers were tested (4.0, 5.5 and 7.0 W, corresponding to pulse energies of 0.16, 0.22 and 0.28 mJ) as well as different structure periodicities (26, 52 and 78  $\mu\text{m}$ ). Following our previous results (Juan Pedro Godoy Vilar et al., 2018) which showed that post-process storing of samples in a chipboard cupboard as opposed to ambient laboratory conditions significantly accelerated the anti-wetting transition, in this case all samples were stored in a chipboard cupboard. Initial experiments were focused on finding the optimal processing power. Figure 1 shows surface profiles of created structures with a line-to-line spacing of 52  $\mu\text{m}$ . Figure 2 shows the contact angle as a function time for various power levels.

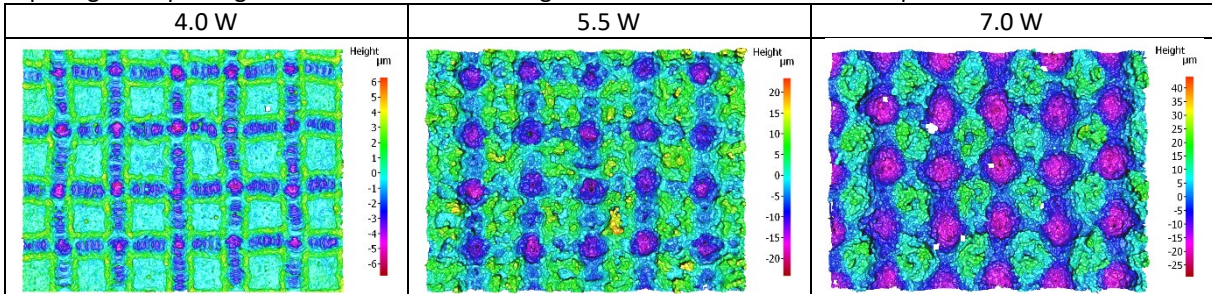


Fig. 2. Surface maps of the hydrophobic surfaces textured using 3 different laser powers. The line-to-line spacing was 52  $\mu\text{m}$  in all cases.

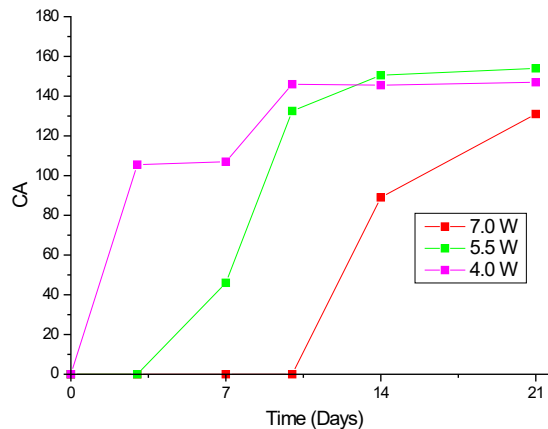


Fig. 3. Contact angle as a function of time for 3 different laser powers. The line-to-line spacing was 52  $\mu\text{m}$ . Samples were stored in a chipboard cupboard.

As indicated in Figure 2, all of the produced laser textures transitioned to superhydrophobic over a period of time, but dependent on the average power used, the duration of this transition varied. A higher average power leads to an increased oxide volume on the surface of the processed samples. This increased volume requires more time to reach the hydroxylated state, hence slowing down the absorption of hydrocarbons.

Another parameter that was investigated was the line-to-line spacing. Figure 3 shows surface profiles for three different line-to-line spacings. Figure 4 shows the contact angle as a function of time for these line-to-line spacing. During these experiments, an average laser power of 5.5 W was used.

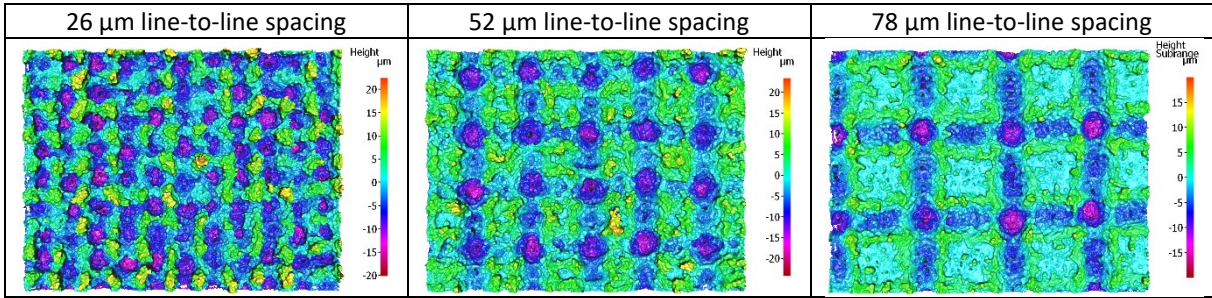


Fig. 4. Surface profiles of the hydrophobic surfaces textured using 3 different line-to-line spacing. Laser average power used was 5.5W.

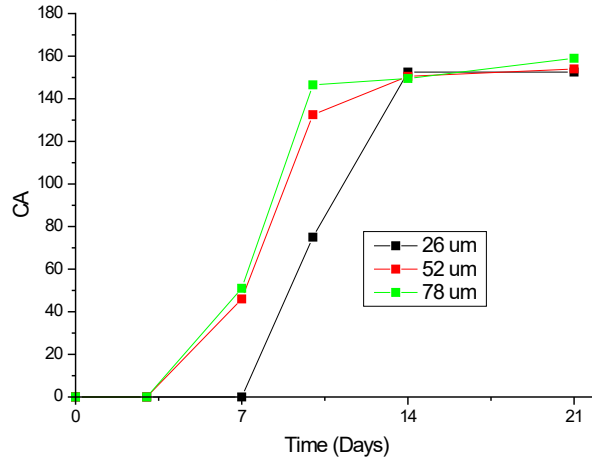


Fig. 5. Contact angle as a function of time for line-to-line separation. Average laser power of 5.5 W was used. Samples were stored in a chipboard cupboard.

For all of three line-to-line spacings tested, the superhydrophobic state was reached after 14 days, but the smaller spacing took slightly longer. The likely reason is the larger total energy input into the surface leading to a larger volume of oxides.

### Storage conditions

The transition to superhydrophobic surface properties is also strongly dependant on the conditions in which samples are stored. It was shown previously that by storing samples in a chipboard cupboard as opposed to the ambient laboratory conditions can significantly shorten its duration (by ~3 times) (Juan Pedro Godoy Vilar et al., 2018). As a result, we decided to test various post-processing storing conditions including the chipboard cupboard, ambient laboratory conditions, an empty plastic enclosure and a plastic enclosure with an open container of paint (~25 ml Hammerite Anti-Corrosion Smooth Black Paint).

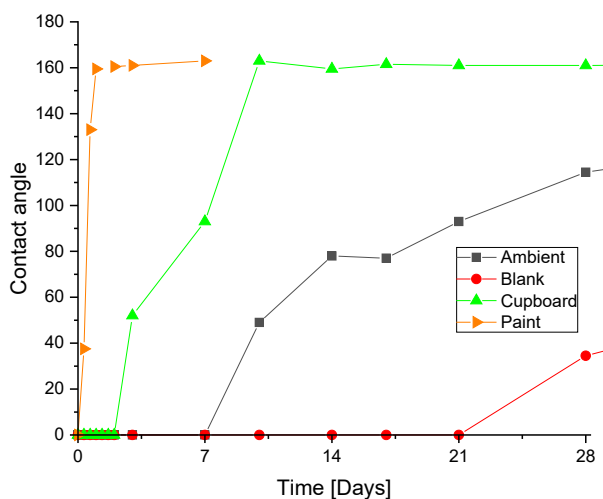


Fig. 6. Contact angle as a function of time for various storage conditions. An average power of 5.5 W and a line-to-line spacing of 52  $\mu\text{m}$  was used.

The slowest antiwetting transition was recorded for samples placed in the plastic container. This isolated atmosphere had a limited amount of water molecules and an organic compound. The second slowest transition was recorded for the samples placed in ambient laboratory conditions with samples still not hydrophobic after 4 weeks. Samples placed in the chipboard cupboard were hydrophobic within two weeks. The quickest anti-wetting transition (48 hours) was recorded for the samples placed in the plastic box with a small amount of paint present. We postulate that the alkanes (saturated hydrocarbons) that paint consists of have significantly increased the number of organic compounds available to be absorbed by the hydroxylated oxides.

#### 4. Conclusions

Superhydrophobic structures can be generated on stainless steel 316L surfaces using a nanosecond fibre laser. The laser post-process time for these surfaces to transition to this superhydrophobic state is strongly affected by the scanning strategy, average power, and sample storage conditions. In particular, storing the samples in a closed box that also contains a small open container of paint it was possible to reduce the transition time to <48 hours.

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