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## Triangular laser-induced submicron textures for functionalising stainless steel surfaces

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#### ABSTRACT

Processing technologies that engineer surfaces with sub-micron topographies are of a growing interest to a range of optical, hydrophobic and microbiological applications. One of the promising technologies for creating such topographies employs ultra-short laser pulses to produce laser-induced periodic surface structures (LIPSS) that often result in non-regular, quasi-periodic nanoripples and nanopillars. In this research near infrared ultra-short pulses of 310 fs with a circular polarisation was used to texture ferritic stainless steel workpieces. A single-step process was designed to generate low spatial frequency LIPSS (LSFL) over relatively large areas. Apart from highly regular and homogeneous parallel lines with approximately 900 nm periodicity, extraordinarily uniform triangular-LSFL in hexagonal arrangements was created. The generation of such LSFL was found to be highly repeatable but very sensitive to the used laser processing settings. Therefore, the sensitivity of triangular-LSFL formation to the used laser processing settings, i.e. pulse to pulse distance, pulse fluence and focal plane offsets, were investigated in regard to the resulting morphologies and functional properties, i.e. structural colors and superhydrophobicity. Finally, the capability of this technology for producing uniform triangular-shaped LSFL on relatively large surface areas of stainless steel plates was studied.

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#### 1. Introduction

The interest and research activities in the field of laser-induced periodic surface structures (LIPSS) have been growing in recent years due to their promising applications for surface functionalisation. In particular, such surface engineering technology has been used for decorative purposes [\[1\]](#page--1-0), anti-counterfeiting [\[2\]](#page--1-0) and improved solar cell efficiency [\[3\]](#page--1-0) due to the resulting structural coloring, broadband light absorption, antireflection and blackening effects. In addition, surfaces processed in this way exhibit a superhydrophobic behaviour that finds applications in self-cleaning [\[4\]](#page--1-0) and anti-icing [\[5\].](#page--1-0) Another area is biomedical, i.e. dental and orthopaedic implants, where LIPSS textured surfaces have been used to influence biocompatibility  $[6]$ , cell proliferation  $[7]$  and also bacterial adhesion and biofilm formation  $[8,9]$ . Furthermore, LIPSS texturing of hard coatings has also been investigated for tribological applications [\[10\].](#page--1-0)

These applications can require surface topographies with dimensions in the sub-micron range. Therefore, ultra-short laser pulses are used to generate a plethora of low spatial frequency LIPSS (LSFL), such as nanoroughness, nanopillars and nanogratings,

⇑ Corresponding author. E-mail address: [J.Romano@bham.ac.uk](mailto:J.Romano@bham.ac.uk) (J.-M. Romano). with periodicities much lower than the laser spot size. There are many factors affecting their generation such as irradiated material and laser wavelength  $[11]$ , beam polarisation  $[12]$ , fluence per pulse and number of pulse [\[13\],](#page--1-0) but also the irradiation environment [\[14\]](#page--1-0) and irradiation angle [\[15\]](#page--1-0). LSFL are usually orientated perpendicular to the incident linear polarisation and have a spatial periodicity close to the laser wavelength [\[11\]](#page--1-0). In the case of circular polarisation, several cases can be observed: wavy ripple-like LSFL generated at  $45^{\circ}$  orientation [\[12,16\].](#page--1-0) With the increase of the total energy dose per unit area, referred to as accumulated fluence, early-stage LIPSS undergo a sharp change in periodicity and uniformity [\[17\].](#page--1-0) When both peak fluence and accumulated fluence increase, more complex, hierarchical LIPSS referred to as microbumps can be obtained  $[18]$ . Bumpy surfaces are shown to be a consequence of heat accumulation at high average power and repetition rates [\[19\].](#page--1-0)

Such complex LIPSS can be generated with linear polarised femtosecond laser pulses by tailoring a compound mix of LIPSS in the sub-micron range [\[20\]](#page--1-0). Furthermore multi-scale topographies can be obtained by using a pre-processed surface  $[21]$ . The fabrication of rhombic-shaped LIPSS can be achieved using promising single-step laser processes using a dynamic rotation of polarisation [\[12\]](#page--1-0) or a cylindrical vector beams generated with a radial polarisation converter [\[22\].](#page--1-0) Another approach is the two-step processing

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employing a train of pulses or multiple passes with varying relative orientation of the scans in regard to the polarisation direction that lead to square- or diamond-shaped LIPSS. Superimposition of LIPSS can occur with significantly less fluence per pulse than that used in the initial pulses [\[9,23,24\]](#page--1-0).

The texturing of larger surfaces with such self-organised quasiperiodic nanofeatures is essential to broaden the use of this technology for surface functionalisation in a number of promising application areas. Such large-area texturing has been investigated by employing different processing strategies, e.g. the use of moving discrete spot laser irradiation to merge LIPSS [\[25,26\]](#page--1-0) or through pulse overlapping [\[18,27–30\].](#page--1-0) High repetition rates combined with high scanning speed, in the order of MHz and m/s respectively, has also been shown to enable area processing with sufficient pulse fluence and pulse overlap to generate uniform LIPSS in one pass; the uniformity being then obtained by optimising the distance between scanned lines [\[29\]](#page--1-0). However, potential local nonuniformities of LIPSS are known to occur as a result of preceding polishing step [\[28,31\],](#page--1-0) grain boundaries [\[32\]](#page--1-0) and surface defects [\[28\].](#page--1-0) Uniformity of LIPSS generation can potentially be improved using pre-processed gratings [\[21,33\]](#page--1-0).

In this paper, the LIPSS generation on stainless steel plates in ambient air is presented. A single-step femtosecond laser process (one pass) is investigated using high scanning speed and pulse frequency, up to 1 m/s and 500 kHz, respectively, to enable highthroughput processing. The generation of uniform large-area LIPSS with Gaussian intensity laser beam is studied. In addition to wavy and relatively disorganised LSFL reported in literature [\[12,16,22\],](#page--1-0) this research presents a single-step texturing with highly regular linear-LSFL and triangular- like LSFL in hexagonal arrangements employing a conventional beam delivery set-up with circular polarisation. The functional properties of processed surfaces in terms of light scattering and wettability are also investigated together with process robustness, in particular the sensitivity of the texturing process to variations in the focal position.

#### 2. Material and methods

Commercially available X6Cr17 ferritic stainless steel plates with 0.7 mm thickness and average roughness of  $R_a = 35$  nm are used in this research.

The texturing was performed using a femtosecond laser source (Satsuma from Amplitude Systemes) with the following technical characteristics, a central wavelength,  $\lambda$ , of 1032 nm, 310 fs pulse duration, a maximum pulse repetition rate of 500 kHz and 5 W average power. The beam line incorporates a beam expander and a quarter waveplate to convert the s-type linear polarisation of the laser source into a circular one. A galvo scan head (RhoThor RTA) equipped with a 100-mm focal length telecentric lens is used to deflect the laser beam over the surface. The spot diameter  $2\omega_0$  at  $1/e^2$  is 30 µm.

Initially, the field textured were limited to 3  $\times$  3 mm<sup>2</sup> in order to study the LSFL generation. Then, larger areas up to  $40 \times 40$  mm<sup>2</sup> were processed without stitching in order to investigate the surface wettability and optical properties. The theoretical depth of focus is 1.1 mm, in particular twice the Rayleigh length  $z_R = \pi \omega_0^2 / (\lambda M^2)$ , where  $M^2$  is better than 1.2 for the used laser source.

The samples were positioned normal to the incident beam and fixed onto a 5-axis motorised stage that allows three linear and two angular movements. All texturing trials were performed in atmospheric conditions.

The textured areas were processed line by line, using a bidirectional raster scanning strategy (see Fig. 1). The distance between scan lines is defined as hatch distance, h, ranging from  $1 \mu m$  to  $10 \mu$ m. The scanning is executed with a variable velocity, v, from 100 mm/s to 1500 mm/s and pulse repetition rates, f, from 50 kHz to 500 kHz. The distance between two consecutive spot centres is therefore given by:

$$
d = v/f \tag{1}
$$

The effective number of pulses per beam spot is calculated by  $2\omega_0/d$  while the effective number of pulses per unit area, N, is:

$$
N = \frac{\pi \omega_0^2}{dh} \tag{2}
$$

The overlap, O, between two consecutive circular pulses can be approximated by the geometrical equation [\[34\]](#page--1-0):

$$
O = \frac{1}{\pi \omega_0} \left\{ 2\omega_0 \cos^{-1} \left( \frac{d}{2\omega_0} \right) - d \sqrt{1 - \frac{d^2}{2\omega_0^2}} \right\}
$$
(3)

Surfaces were textured with varying average power, P, and pulse repetition rate, f, and thus the pulse fluence could be calculated as  $\varphi_0 = P/(\pi \omega_0^2 f)$ . The effective accumulated fluence per unit area,  $\varphi$ , can then be approximated as follows:

$$
\varphi = N\varphi_0 = \frac{P}{f d h} \tag{4}
$$



Fig. 1. Beam line components and scanning strategy.

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