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Picosecond laser treatment production of hierarchical structured stainless steel to reduce bacterial fouling

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ABSTRACT

The design of surfaces that prevent biofouling through their physical structure and chemical properties provides a potential solution to increase their hygienic status. A picosecond laser was used to produce hierarchical textures on stainless steel. The surface topography, chemistry and wettability were characterised. The S_a, and wettability of the surfaces all increased when compared to the control following laser treatment. The S_a, S_q and S_{pv} values ranged between 0.02 µm–1.16 µm, 0.02 µm–1.30 µm and 0.82 µm–9.84 µm respectively whilst the wettability of the surfaces ranged between 99.5°–160°. Following microbial assays, the work demonstrated that on all the surfaces, following attachment, adhesion and retention assays, the number of *Escherichia coli* on the laser textured surfaces was reduced. One surface was demonstrated to be the best antiadhesive surface, which alongside being superhydrophobic (154.30°) had the greatest S_a and S_{pv} (1.16 µm; 6.17 µm) values, and the greatest peak (21.63 µm) and valley (21.41 µm) widths. This study showed that the surface roughness, feature geometry, chemistry and physicochemistry all interplayed to affect bacterial attachment, adhesion and retention Such a modified stainless steel surface may have the ability to reduce specific fouling in an industrial context.

1. Introduction

Biofouling on surfaces can produce a number of economic and potential contamination problems in a variety of industries including the food industry (Whitehead and Verran, 2009). Bacterial attachment is the prerequisite to such fouling and is followed by bacterial adhesion and retention on a surface. This may result in the decline of the hygienic status of a surface resulting in potential risks to food quality, product contamination and/or spoilage and blockages of mechanical components (Whitehead et al., 2015). In 2011, it was reported that foodborne disease caused a projected 48 million illnesses, 128,000 hospitalisations and 3000 deaths annually in United States between 1996 and 2010 (Nyachuba, 2010; Schlisselberg and Yaron, 2013; Srey et al., 2013).

The modification of substratum topography, chemistry and/or physicochemistry can be used to reduce microbial biofouling. Many studies have been carried out to determine the effect of surface properties on bacterial attachment and retention (Hilbert et al., 2003; Jullien et al., 2003; Whitehead et al., 2005; Whitehead and Verran, 2006; Wang et al., 2009; Milledge, 2010; Dantas et al., 2016; Tetlow et al., 2017). Some studies have reported that there is a correlation between surfaces roughness and bacterial attachment whereby the retention of

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microorganisms increased with increasing surface roughness (Jullien et al., 2003; Wang et al., 2009; Whitehead et al., 2011; Dantas et al., 2016). However, others have reported that there was little or no relationship between surface roughness and bacterial attachment (Hilbert et al., 2003; Milledge, 2010). The effect of surface wettability on bacterial attachment has also been carried out and it has been reported that the number of adhered bacteria was dramatically decreased with increasing surface hydrophobicity and bacteria adhered to hydrophobic materials were more easily removed by an increased flow or an air-bubble jet (Bos et al., 2000; Fadeeva et al., 2011; Privett et al., 2011; Dou et al., 2015). However, others have reported that there was no relationship between surface wettability and bacterial attachment (Cunha et al., 2016).

Surface topographies can be grouped into three main structural types namely irregular, regular or hierarchical. Although changes in the surface topography are usually described using the average roughness value (R_a), it has been suggested that an in depth evaluation of the shape of the surface features also needs to be described (Whitehead et al., 2005). Several studies have been carried out to study the effect of topographies, for example grooves (Verran et al., 2010), squared-features (Perera-Costa et al., 2014) or pits (Whitehead et al., 2005; Whitehead and Verran, 2006). In nature, there are many plants with hierarchical surface structures that are considered as self-cleaning surfaces such as the lotus leaf. These surfaces are superhydrophobic with contact angles ≥150° and sliding angles <5° (Yan et al., 2011). Several studies of bacterial attachment and retention on such biomimetic type features for example those which replicate the lotus leaf (Fadeeva et al., 2011) or taro leaf (Crick et al., 2011) have been carried out.

Stainless steel is a used in a wide range of industrial applications due to its unique properties such as ease of fabrication and modification, corrosive resistance, inertness and ease of cleaning. Different techniques such as lithography (Gold et al., 1995), moulding (Chou et al., 1995) and photolithography (Green et al., 1994) have been used to produce different micro/nano structures, but most of these are not compatible with stainless steel surfaces. Laser surface modification has been extensively studied for the production of surfaces to be used in a range of different applications (Dobrzański et al., 2008; Cunha et al., 2013; Long et al., 2016, 2015b). This paper focuses on the production of a range of hierarchical (macro/micro/nano) topographies generated using a novel picosecond laser ablation process and the effect of the altered surface properties on bacterial attachment, adhesion and retention.

2. Material and methods

2.1. Laser surface texture preparation

Stainless steel surfaces (316L, S_a 20 nm ± 0.1 nm finish) with a 0.7 mm thickness was used in this work to produce laser etched areas 5 mm × 5 mm in size. Before laser treatment, the surfaces were cleaned ultrasonically with acetone followed by ethanol then deionised water for 10 min each. The experiment was performed using an EdgeWave Nd:YVO_4 picosecond laser of 10 ps pulse duration, with a 103 kHz repetition rate, 1.06 μm, 125 μm beam size in ambient air using a range of scanning parameters (Table 1). The scanning was performed using either parallel or cross lines patterns. After laser treatment, the surfaces were cleaned ultrasonically with ethanol for 5 min then dried using compressed air for 5 s–10 s to remove any ablated debris or contamination. The surfaces were immersed into a 1% hetadecafluoro-1,1,2,2-tetrahydrodecyl-1-trimethoxysilane ($CF_3(CF_2)_7(CH_2)2Si(OCH_3)_3$) (Gilest Inc., USA), (referred to as FSA) methanol solution for 2 h followed by rinsing with ethanol and drying in an oven at 80 °C for 30 min (Long et al., 2015a).

2.2. Surface characterization

After laser treatment, the macrostructure of the surfaces was imaged using a scanning electron microscope (SEM) (Carl Zeiss Ltd. UK). The microtopography and roughness values of the surfaces were also characterised using laser profilometry (Keyence, UK). Values of S_a (Arithmetic mean height), S_q (Root mean square height) and S_{pv} (Maximum height of the surface) were recorded for each of the surfaces. Selected line scans were used to determine the height, depth and width of the peaks and valleys. Atomic force microscopy (Veeco Instruments Inc., UK) was used to examine the nanotopography of the surfaces. Image processing was carried out using the Scanning Probe Image Processor. Selected line scans were used to determine the height, depth and width of the peaks and valleys. Chemical analysis was carried out using Energy Dispersive X Ray (EDX) on the SEM instrumentation (n = 3).

2.3. Confocal laser microscopy (CSLM) and atomic force microscopy (AFM)

For CSLM, the surfaces were examined using a 150× objective (Keyence X200K 3D Confocal Laser Microscope, USA with VK analyser software) to determine the substratum macro and micro topographies. The S_a, S_q and S_{pv} (average surface roughness, root-mean square roughness and peak to valley height respectively) was measured for the surfaces. To determine the shape height, depth and width of the peaks and valleys line profiles were used.

AFM measurements were carried out to determine the nanotopographies of the surfaces using a Dimension 3100 AFM (Veeco Instruments Inc., UK).

2.4. Physicochemistry

The physicochemistry of the surfaces was obtained by measuring the contact angle via the sessile drop method (FTA 188, UK). By measuring contact angles for three fluids with known γ_L values, the three variables, $\gamma_s{}^{LW}$, γ_S^+ and γ_S^- could be determined (n = 10). Six microliter droplets of deionised water, formamide or α-bromonaphthalene were dropped onto the surface. The approach of Van Oss et al. (1989) was used to calculate the surface hydrophobicity. The degree of hydrophobicity of the surfaces was expressed as the surfaces free energy of interaction of the material when immersed in water (w) (ΔG_{iwi}). The material was considered hydrophobic when the surface $\Delta G_{iwi} < 0$ and hydrophilic when $\Delta G_{iwi} > 0$. ΔG_{iwi} was calculated according to Eq. (1) (Van Oss et al., 1989),

$$\Delta G_{iwi} = -2\gamma_{sL} \tag{1}$$

The SFE (γ_S) was determined from the polar or Lewis acid base component ($\gamma_s{}^{AB}$) and apolar Lifshitz–van der Waal component ($\gamma_s{}^{LW}$) where;

$$\gamma_s = \gamma_s{}^{LW} + \gamma_s{}^{AB} \tag{2}$$

where γ^{LW} was the Lifshitz–van der Waals component of the surface free energy and the Lewis acid–base component γ^{AB}.

$\gamma_s{}^{AB}$ was calculated from;

$$\gamma_s{}^{AB} = 2\sqrt{\gamma^+\gamma^-}. \tag{3}$$