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Research Paper

Micro-fabrication of high aspect ratio periodic structures on stainless steel by picosecond direct laser interference patterning

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ABSTRACT

We have studied the fabrication of line-like and pillar-like periodic microstructures on stainless steel by means of direct laser interference patterning. A picosecond (10 ps) pulsed Nd:YAG laser operating at 1064 nm wavelength was used to produce the microstructures with spatial periods ranging from 2.6 μm to 5.2 μm. By varying the laser parameters (laser fluence, pulse-to-pulse overlap) structure depths ranging from 500 nm to nearly 11.5 μm could be obtained. Furthermore, low and high frequency laser induced periodic surface structures (LIPSS) have been generated, resulting in three-level multi-scaled patterns. The orientation of the laser induced periodic structures with respect to the interference patterns could be adjusted by controlling the laser beam polarization. Finally, static water contact angle measurements are performed to investigate its correlation with the surface morphology. The treated surfaces are characterized using confocal and scanning electron microscopy.

1. Introduction

The arising usages of metals in the automotive, aeronautics and appliances industries require new innovative surface properties such as superhydrophobicity, oleophobicity or antibacterial behavior. Several solutions for these specific problems can be already found in natural examples such in plants and animals and have been studied by different scientist such Webb et al. (2014). Hence, researches worldwide have approached in mimicking the topography of these natural surfaces using different fabrication methods. For example, Liu and Li (2012) used a micro-replication method to mimic the shark's skin for obtaining a superhydrophobic surface. Additionally, Baum et al. (2014) carried out an analysis of frictional properties of a snake-skin replica. Furthermore, significantly enhanced surface properties can be produced by combining a micrometer scaled structure with nano- or sub-micrometer features. Barthlott and Neinhuis (1997) and Ensikat et al. (2011) have pointed out the morphologies as in the case of Nelumbo nucifera and Colocasia esculenta, which consist on pillar and dot-like micro- and nanostructures and are responsible of self-cleaning property.

In addition to the surface topography, also the chemistry plays an important role on the surface properties. In this context, a large number of methods can be used, such as the process developed by Zhu et al. (2012) based on an immersion of steels in a hydrophobic treating solution or other coating deposition techniques consisting for example on the fabrication of nanoparticles and its subsequent chemical modification as reported by Geissler et al. (2013). Therefore, not only microstructure govern the solid surface properties but also the chemical content. Thus, the manufacturing routes are unlimited, and offer more alternatives to be scale-up, which represent an interesting and potentially area of research.

Recently, ultrafast lasers have been used to fabricate microstructured surfaces on metals to create for example superhydrophobic properties. Moradi et al. (2013) have examined the influence of laser parameters in relation with the resulted surface topology and the water contact angles (WCA) that can be obtained in stainless steel 316L. Using this process, he has been capable of achieving WCA up to ~160°. In addition, it has also been observed that after laser processing, a further increase of the WCA can occurs after a certain period of time. Kietzig et al. (2009) has explained this behavior in relation with an increasing amount of carbon in the processed surface when exposed to normal environment conditions. However, the main drawback of this technology is the long processing times that are required to treat the material surface. In general, during the structuring process, the component surface is scanned with the laser beam using galvanometric mirrors and several over-scans are required to get the final structure. For example, Del Cerro et al. (2010) required 19 min/cm^2 to obtain contact angles of 150° on top-coated stainless steel (laser power: 180 mW, linear speed: 20 cm/s, 50 over-scans, repetition rate: 400 kHz). Similarly, 55 min/

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cm^2 were required to obtain WCA over 140° on stainless steel 316 L by Wu et al. (2009) and 12 min/cm^2 on cooper by Long et al. (2010). Furthermore, achieving resolutions better than 20 µm is normally associated with an increased technical complexity and long processing. For example, pillar-like patterns with a lateral spacing of 19 µm and a structure depth of 20 µm required approximately 27 min/cm^2 according to Del Cerro (2014).

In the last years, Direct Laser Interference Patterning (DLIP) has become one of the most effective techniques for texturing materials down to the sub-micrometer level with high quality, throughput and flexibility. Regarding these advantages, Lang et al. (2016) used a high speed DLIP configuration to produce periodic patterns with throughputs up to ~0.0001 min/cm^2 (1.11 min/m^2) in polymers and ~0.0003 min/cm^2 (2.78 min/m^2) in metals. The DLIP process makes use of interference patterns that are created by overlapping two or more coherent laser beams and thereby, producing a periodic variation of the laser intensity. The versatility of the method in terms of pattern geometries that can be produced has been realized by numerical simulations of the interference patterns that can be obtain as function of the number of laser beams used, the laser light polarization, intensity and incident angles for each sub-beam by different authors, such Tavera et al. (2011) and Rodriguez et al. (2009). Recently, Alamri and Lasagni (2017) has also shown the possibility of producing more complex patterns based on both ablation and swelling processes. Furthermore, an overview of applications is described by Lasagni et al. (2015) explaining the maturity of the DLIP technology to be scaled-up as well as different systems that have been produced.

If pulsed laser systems with sufficient energy (or power) are used, the materials can be locally ablated at the interference maxima positions either by photo-thermal or photo-chemical processes, depending on the material used, the laser wavelength and the pulse duration of the laser system. In consequence, periodic surface patterns can be produced in a one-step process with controllable pitch and geometry. This characteristic has allowed DLIP to directly process different metallic surfaces for a wide number of application fields. For instance the coefficient of friction and its correlation with different surface parameters as the structure aspect ratio (AR) and the geometry shape were investigated by Bieda et al. (2014) and the lifetime of produced structures on metals was investigated by Rosenkranz et al. (2015). Other applications have been developed by other authors. For example, Gao et al. (2016) demonstrated the increasing of the WCA in coronary stents and Zhou et al. (2015) shown the fabrication of variable-sized platinum nanoparticles for enhancing photocatalysis and solar energy conversion. Also Yuan et al. (2010) developed a method to produce ZnO nanowires on GaN for sensor arrays while Raillard et al. (2013) demonstrated the change of the wetting properties of martensitic bearing steel (100Cr6) as function of the pattern orientation and periodicity for oil and destilled water.

Independently of the number of laser beams used to produce the interference patterns, the repetitive distance of the periodic intensity distribution (called spatial period) can be changed by varying the angle between the beams. For a two-beam interference setup, the spatial period (Λ) is given by Eq. (1):

$$\Lambda = \frac{\lambda}{2\sin(\theta)} \tag{1}$$

where λ is the wavelength of the laser beams and θ is the half angle between the two incident beams.

However, the minimal achievable spatial period that can be transferred to a certain material (such metals) is also controlled by the thermal properties of the material as well as the laser pulse duration. For example, when nanosecond pulses are used, the structuring mechanism is mainly based on local ablation and/or Marangoni convection. In particular, if the used spatial period of the interference pattern (Λ) is in the same order as the thermal diffusion length (l_T) (for example 1–2 µm for metals irradiated with 10 ns laser pulses, see Eq. (2)), a low thermal contrast is observed (temperature difference between maxima and minima positions over the spatial period) which inhibits the local treatment of the material at the maxima positions and thus the material is i.e. molten over the whole irradiated area.

$$l_T = \sqrt{\frac{\lambda_{th}\tau}{\rho c_p}} \tag{2}$$

where λ_{th} is the thermal conductivity, τ is the pulse duration, ρ is the density, and c_p is the specific heat capacity.

Furthermore, Bieda et al. (2010) observed that the structure heights for short spatial periods (< 2–3 µm) are generally limited to a few nanometers on several metallic surfaces. As it can be deducted from Eq. (2), for a certain material the thermal diffusion length can be only shortened by reducing the duration of the laser pulse. Therefore, if picosecond pulses are used (i.e. 10 ps instead of 10 ns), the material can be precisely heated mainly only at the interference maxima positions which reduces the amount of produced melt and allowing the fabrication of patterns even in the sub-micrometer range. The last was demonstrated theoretically and experimentally by Bieda et al. (2016), by fabricating 700 nm surface structures on Cu, Fe, and Ti.

In addition to line-like patterns, a two-beam configuration can be also used to produce more complex pattern geometries. For example, if the target material is irradiated more than once and the substrates are rotated a certain angle (i.e. 60 or 90°) between the irradiation steps, pillar-like patterns can be produced. However, due to the relative strong melting taking place when using ns-pulses, the first structure is commonly deleted by the second one as shown in the study performed Bieda et al. (2010), where this strategy was explored together with thermal simulations of the performed experiments.

In the present study, we focus on the fabrication of high aspect-ratio line-like and pillar-like periodic structures on stainless steel using picosecond Direct Laser Interference Patterning. A strategy for this objective is introduced. The produced structures are characterized using scanning electron microscopy, confocal microscopy and finally water contact angle measurements are performed as function of the geometrical parameters of the produced patterns.

2. Experimental

2.1. Materials

Sheets of X6Cr17 corrosion resisting ferritic steel (also called 1.4016), with 0.98 mm in thickness, were used as substrates in this work. The samples were electro-polished obtaining an average surface roughness (Ra) of 60 nm. Prior to the laser process, the substrates were cleaned from contaminations using isopropanol.

2.2. Direct laser interference patterning

The structuring of the metallic substrates was performed using a two-beam DLIP configuration. The experimental setup (presented in Fig. 1a) consists of an infrared (IR) pico-second laser (solid-state Q-switched Innoslab Nd:YVO_4, pulse duration: 10 ps), a DLIP optical head (Fraunhofer IWS) and a three-axis positioning stage system with an accuracy of ± 2.5 µm. The used wavelength was 1064 nm and a repetition rate 1 kHz. The beam was vertical polarized. In the DLIP optical head, the primary beam is split in two beams by means of a diffractive optical element with a diffraction angle for the first order of 9.21°. Then, the beams are parallelized using a prism with an angle of 19.93° and are finally overlapped with a converging lens (with a focal distance of 40 mm). By controlling the position of the prism it is possible to automatically change the spatial period. Further information about the experimental setup has been already published elsewhere by Bieda et al. (2016). The fluence was calculated on the basis of the laser beam radius ($1/e^2$), using the D-squared method described by Liu