DE GRUYTER Adv. Opt. Techn. 2020; 9(3): 121–130

Research Article

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Modification of Ti6Al4V surface properties by combined DLW-DLIP hierarchical micro-nano structuring

https://doi.org/10.1515/aot-2019-0071
Received December 20, 2019; accepted March 19, 2020; previously published online April 18, 2020

Abstract: The use of pulsed laser irradiation techniques has proven to be a clearly effective procedure for the achievement of surface properties modification via micro-/nano-structuration, different conceptual approaches having been the subject of research and extensively reported in the literature. Completing the broad spectrum of applications developed mostly involving the generation of structured surfaces (particularly of metallic materials) with specific contact, friction and wear functionalities, the application of laser sources to the surface structuration of metal surfaces for the modification of their wetability and corrosion resistance properties is considered. The particular problems found for the generation of the appropriate surface microstructure able to replicate the hydrophobic behaviour of some live structures present in nature, their long term stability and their amenability to macroscopic scale are discussed along with innovative methods to generate the required hierarchical micro-/nano-structures by a combination of the DLW and DLIP techniques.

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Keywords: combined DLW + DLIP; corrosion resistance; hierarchical surface structures; hydrophobicity; laser surface micro-/nano-structuration.

1 Introduction

The increasing availability of advanced fiber and DPSS lasers with characteristic pulse lengths ranging from ns to fs provides a unique frame in which the development of laser generated micro/nano-structures has been made possible for very diverse kinds of materials and applications [1–3]. Surface functionalization with different technical purposes has been thoroughly described [4–8]. In particular, surface micro-nanostructuring for achieving a super- or ultra-hydrophobic behaviour leading to self-cleaning [9–11], anti-corrosion [12, 13], anti-icing [14–16], and bacterial repellence [17, 18] have been specially considered.

For the achievement of this kind of surface properties, the use of pulsed laser irradiation techniques has proven to be a clearly effective procedure, different conceptual approaches having been the subject of research and extensively reported in the literature [19–22].

By means of the adaptation of different laser sources and processing techniques, the convenient alteration of the surface properties (especially its physico-chemical behaviour) by means of the introduction of micro-/nano-features for very different materials [23, 24].

The choice of the laser processing technique to perform the surface micro-/nano-structuring is dependent on the sought features. The achievement of a certain degree of hydrophobicity is related with both, the chemistry of the surface and its roughness. In addition, the absorption of atmospheric agents may result in a change in the hydrophobic response of the treated surface with time [25, 26].

From the point of view of laser processing, the structuring of a surface involves the removal of a prescribed amount of material by means of laser ablation.

This phenomenon is driven by either photo-thermal or photo-chemical effects, either of which can be activated as a function of the duration of the laser pulse, the wavelength, and the properties of the treated material [27, 28].

In the most straightforward approach, the technique known as direct laser writing (DLW) [29, 30] is a procedure to engrave micro-structures and micro-patterns on the surface of metals. In this case, a nanosecond focused laser beam causes photo-thermal ablation associated to the melting of the material under the laser track. In consequence, the evolution of the structures is driven by the Marangoni convection. DLW can be found in many different applications [31, 32]. Reference [33] illustrates the development of a micro-texture known as μ-cell which enables the treated surfaces to offer a super and near ultra-hydrophobic behavior. DLW is limited by the dimensions of the laser beam, the obtaining of structures with smaller resolution not being possible.

The engravement of sub-micro-structures requires procedures where the ablation effect is not limited by the size of the laser beam. In the process known as direct laser interference patterning (DLIP) [21, 34–36] the laser effect is adjusted by the interference between at least two coherently overlapped laser beams. With this configuration, ablation patterns of the scale of the laser wavelength can be manufactured, following the alternation between the maxima and minima of laser local interference pattern within the interaction region. From its initial proposal by Polushkin et al. [37], the DLIP technique has been demonstrated to be a fully versatile procedure for the micro-structuring of all kinds of materials, including metals, polymers, biomaterials, and soft materials.

In this work, the generation of hierarchical structures with features in the micro and nano scale (combination of DLW and DLIP) in Ti6Al4V (a material with demonstrated interest in the field of surface functionalization) is described. The obtained micro-/nano-structures have been analyzed with scanning electron and confocal microscopy in combination with static contact angle measurements. Additionally, the corrosion resistance behaviour of the produced structures has been analyzed. The obtained results highlight the capability of the DLW + DLIP combined technique to generate stable hierarchical structures with super-hydrophobic behavior and with enhanced corrosion resistance.

2 Experimental

Samples of Ti6Al4V with a thickness of 2 mm have been used for laser surface micro-structuring by means of DLW and DLIP. The material was processed after cleaning with ethanol. The samples were kept on air atmosphere and no further treatments were applied after laser processing. The surface morphology of the sample was examined by scanning electron microscopy (SEM HITACHI S-3000N) and the corresponding topography assessed by confocal laser scanning microscopy (Leica DCM 3D). Additionally, static contact angle measurements were carried out to analyze the wettability behavior of the structures and potentiometric analysis according to ASTM G59-97(2014) by means of a Metrohm Autolab PGSTAT 302N potentiostat.

The DLW irradiation campaign was carried out in an ambient environment using a Q-switched pulsed Spectra-Physics nanosecond solid-state Nd:YVO_4 laser source (UPM Laser Centre) that generates pulses with a fixed duration of 15 nanoseconds, at a repetition rate of 20 kHz, with a central wavelength of 1064 nm. The fundamental wavelength is frequency-tripled by a nonlinear optical conversion to obtain pulses with a wavelength of 355 nm. The laser beam was focused onto the sample at normal incidence by means of a 250 mm focal lens to generate a spot size of 15 μm. The position of the sample was controlled using a translational stage with a resolution of 0.1 μm to create squared shaped micro-cells, by using a fixed scan speed of 10 mm/s according to the procedure defined by Ocaña et al. in [33] (see Figure 1). The distance between each micro-cell is named as hatch distance (HD). The process parameters used for the fabrication of the micro-cells are shown in Table 1.

The second laser micro structuring process was performed using DLIP with a Q-switched picosecond laser (EDGEWAVE solid-state Innoslab Nd:YVO_4, pulse duration: 10 ps at the FhG-IWS Dresden) at the fundamental wavelength of 1064 nm (Figure 2). The repetition rate was fixed at 1 kHz. For the DLIP set-up, the laser beam is split in two sub-beams and then overlapped onto the substrate (Figure 3). In the reported case, the patterned area corresponds to the superposition zone of the beams (approximately 160 μm). For a two beam DLIP

Figure 1: Experimental laser processing workstation AB-200 at UPM Laser Centre used for the DLW processing of Ti6Al4V samples.

Table 1: Laser processing parameters for the fabrication of micro-cells on Ti6Al4V by means of the DLW technique.

Average laser power (mW)	80–280
Repetition rate (kHz)	20
Scan speed (mm/s)	10–100
Beam spot size (μm)	15
Hatch distance (H.D.) (μm)	30–50

system, the interference pattern has a line-like distribution. A second irradiation perpendicularly to the first patterning with an angle of 90°allowed generating a periodic pillar-like pattern on top of the prior structures created via DLW and thus creating a dual-scale periodic surface structures. The process parameters used are shown in Table 2.

Table 2: Laser parameters for the application to the Ti6Al4V DLW fabricated samples of the DLIP process.

Average laser power (mW)	162
Repetition rate (kHz)	1
Fluence (J/cm^2)	0.8
Interference area diameter (μm)	160
Interference spatial period (μm)	1.1, 2.6 and 5.4

3 Results

3.1 DLW+Combined DLW+DLIP geometrical patternings

In the first DLW process, square-shaped micro-structures with five different hatch distances were produced in the range of process parameters as shown in Table 1. For all used laser fluence values, the DLW process allowed to create well-defined periodic surface structures. Figure 4 shows a scanning electron microscope (SEM) image (left) of one the processed surface areas (with wavelength: 355 nm, pulse duration: 15 ns, repetition rate: 20 kHz, laser fluence: 5.65 J/cm^2, scan speed: 80 mm/s).

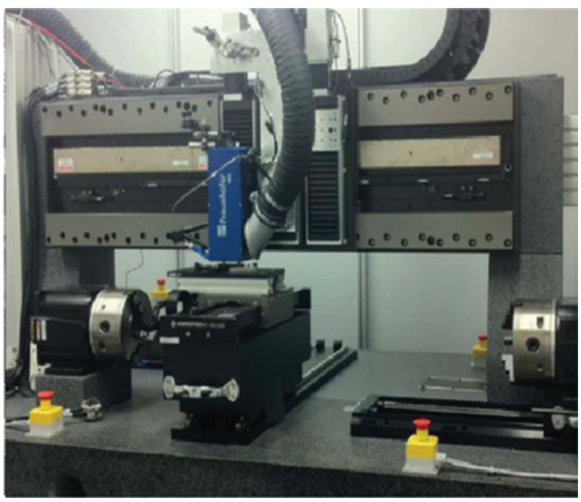

Figure 2: Experimental laser processing workstation at FhG-IWS Dresden used for the DLIP processing of Ti6Al4V samples.

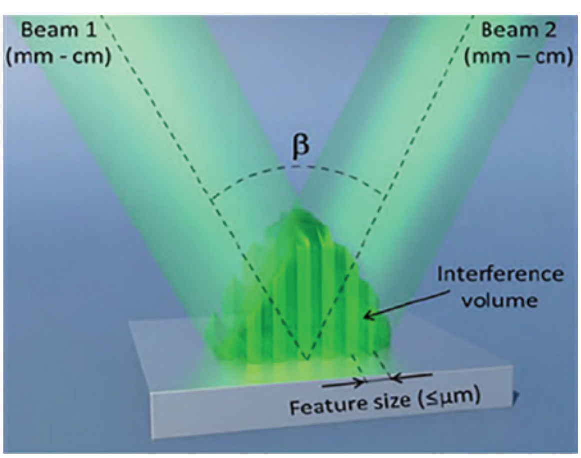

Figure 3: Schematic for the two-beams Direct Laser Interference Patterning used in the treatment of the reported samples (*β* corresponds to the angle between the laser beams and controls, together with the laser wavelength, the resulting spatial period). Adapted from reference [36].

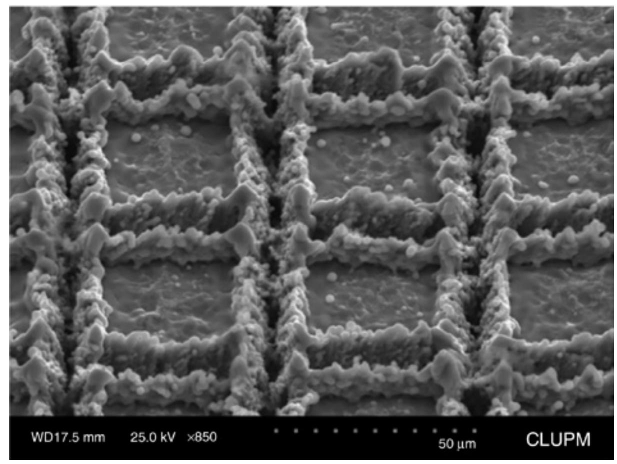

Figure 4: Sample SEM image of a patterned Ti6Al4V μ-cell produced by DLW using nanosecond pulses.

Under the application of ns lasers to microprocessing applications, typical thermal can be appreciated, especially recast formation of the molten material. During the laser ablation, the molten material recast as the laser scan the sample, inducing the formation of micro-walls around the original surface forming a closed packet or μ-cell. These DLW structures are of great interest due to the potential application on enhancing the wettability behavior of the titanium alloy by means of a micro-structured surface as it has been showed that a μ-cell type structure can be used to create a super-hydrophobic surface [33, 37].

In addition to the characterization by SEM, the laser irradiated areas were also analyzed by confocal microscopy. Figure 5 shows an example of reconstructed 3D profile of the same μ-cell obtained by DLW using this technique and in Figure 6 a typical cross-sectional profile of the sample is provided.

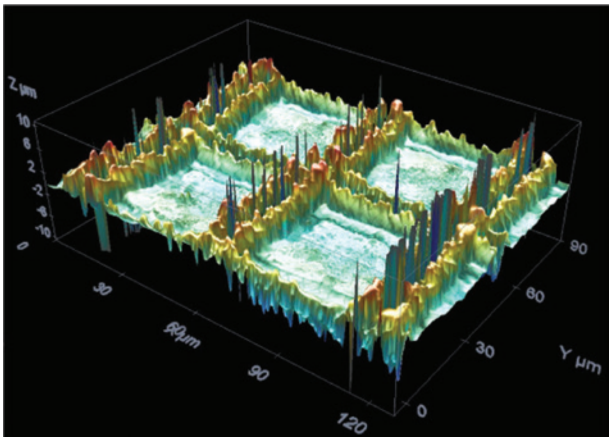

Figure 5: Sample confocal reflection scanning microscopy (CRSM) image of a a patterned Ti6Al4V μ-cell produced by DLW using nanosecond pulses.

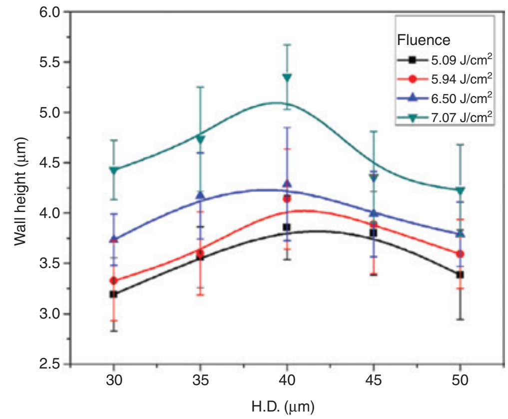

Figure 7: Wall height values for four different laser fluences (5.09–7.07 J/cm²) as function of the hatch distances (30–50 μm).

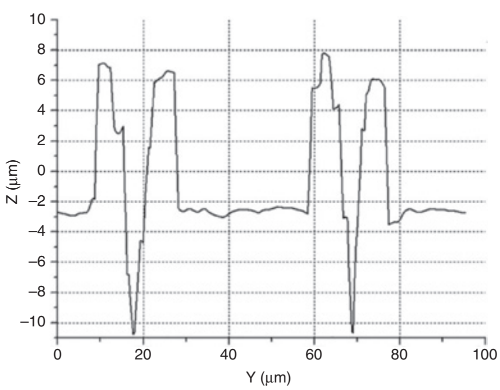

Figure 6: Sample confocal reflection scanning microscopy (CRSM) profile of the reference patterned Ti6Al4V μ-cell (Process parameters: HD: 40 μm; Laser Fluence: 7.07 J/cm²).

The dimensions of these micro-structures strongly depend on the laser parameters used for the ablation of the micro-channels. Figure 7 shows the relation found between the hatch distances and the height of the walls of the μ-cell for four different laser fluence values. It can be observed that, as the laser fluence increases, the material is removed in a higher extent inducing the recast on the top while also re-filling the patterned micro-channel with the molten material. This can be observed by noticing that the horizontal and vertical micro-channels are not created in a symmetric way, as one of them is open in the direction of the laser writing while the other is blocked due to the recast of material as the perpendicular channels are made. For low fluence values, the amount of ablated material is lower and well-defined structures are produced. As the average laser fluence increases, more recast material starts to accumulate, giving place to higher walls, but less defined structures, due to the amount of uncontrolled recast. As the fluence increases, the amount of molten material is higher, thus growing the volume of recast material and the height of the micro-wall, reaching a maximum value of more than 5 μm for a HD of 40 μm. Additionally, it can be observed that micro-walls' height is influenced by the periodic distance between channels. When the distance is lower, there is an accumulation of recast material on adjacent walls, thus increasing height values. For higher hatch distances, there is less accumulation of recast material on adjacent walls, due to the large gap between the structures. The highest wall height values were measured for 40 μm of HD, where the periodic gap promotes both accumulation of recast and fewer ablation of previously formed micro-walls.

After the treatment by DLW processing, microstructured samples were subsequently treated by a DLIP process according to the parametric experimental space defined in Table 2. The combination of both techniques has been investigated in view of their very different spatial scaling and with the objective of generating hierarchical micro-/nano-structures as recomended by different studies on the feasibility of superhydrophobic self-cleaning surfaces [23, 33].

For this purpose, firstly squared shaped micro-cells were generated by means of DLW according to the procedure just described with nanosecond pulses using the parameters reported in Table 1 for a HD of 50 μm. After that, a pillar-like pattern was produced using DLIP with a spatial period of 2.6 μm using the referred process parameters. The result can be observed in Figure 8A and B where a dual hierarchical structures (nano- and micro-features)

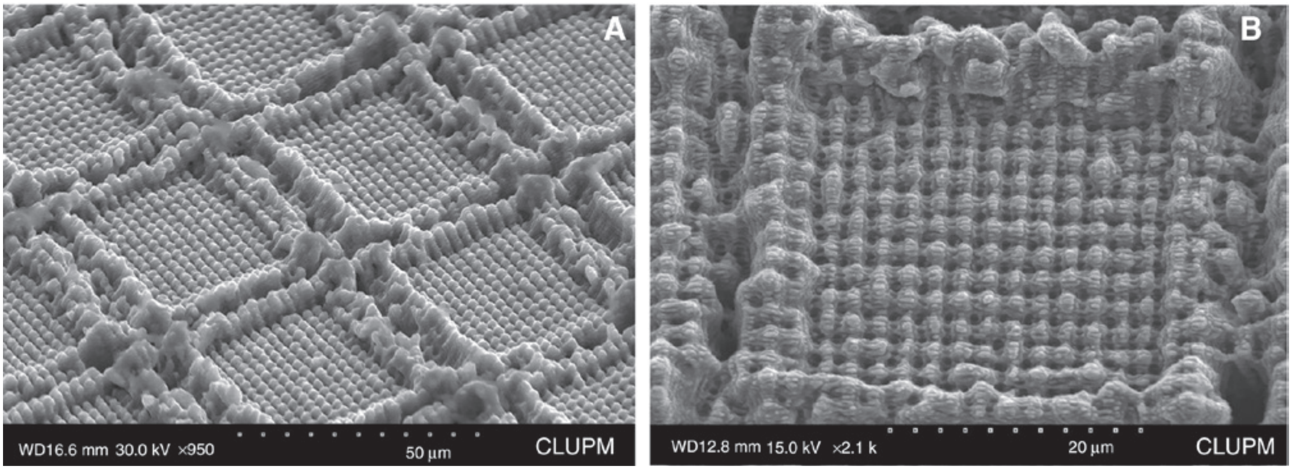

Figure 8: SEM micrographs showing the dual micro-/nano-scale technique obtained in Ti6Al4V by the application of a combined (DLW + DLIP) treatment. A: Magnification x 950; B: Magnification x 2100 (Process parameters: DLW HD: 40 µm; Interference spatial period: 2.6 µm; DLW Laser Fluence: 7.07 J/cm^2; DLIP Laser Fluence: 0.8 J/cm^2).

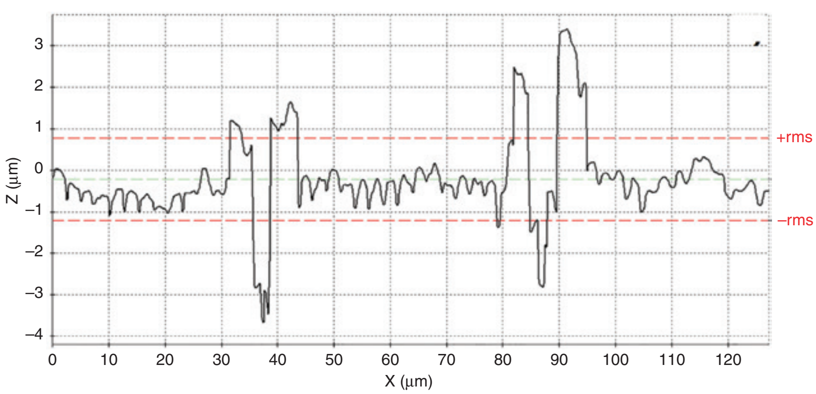

Figure 9: Confocal reflection scanning microscopy cross-section profile of the dual micro-/nano-scale technique obtained in Ti6Al4V by the application of the referred combined (DLW + DLIP) treatment. (Process parameters: DLW HD: 40 µm; Interference spatial period: 2.6 µm; DLW Laser Fluence: 7.07 J/cm^2; DLIP Laser Fluence: 0.8 J/cm^2).

generated by the combination of both techniques is visible. A cross-section profile of the obtained surface pattern is shown in Figure 9. The micrograph shows clearly that the second process with DLIP does not damage the previously fabricated µ-cell using DLW. Much in the contrary, in the center of the µ-cell, where the original topography of the sample along with some recast particles was observed after the individual DLW, a well-defined periodic structure has been fabricated.

The results obtained for the fabrication of two different types of micro-structures generated with the two micro-machining techniques shows that, the DLIP technique can reach a smaller spatial resolution between adjacent micro-structures in comparison with the resolution of the DLW technique, which is limited by the spot size of the laser beam. Although the final topographical structure of the samples treated by this combined procedure is agreed to be predominantly in the micron domain (thus, predominantly a micro-structure), at the same time, as a consequence of the combined treatment, a certain lower scale degree of structuration is achieved (an approach to a true hierarchical micro-/nano-structure) that is considered to be the main responsible for the change in surface properties (as discussed, i.e. in references [12] and [33]).

3.2 Surface wettability modification

After the fabrication of the reported hierarchical micro-/nano-structures, the evaluation of their wettability

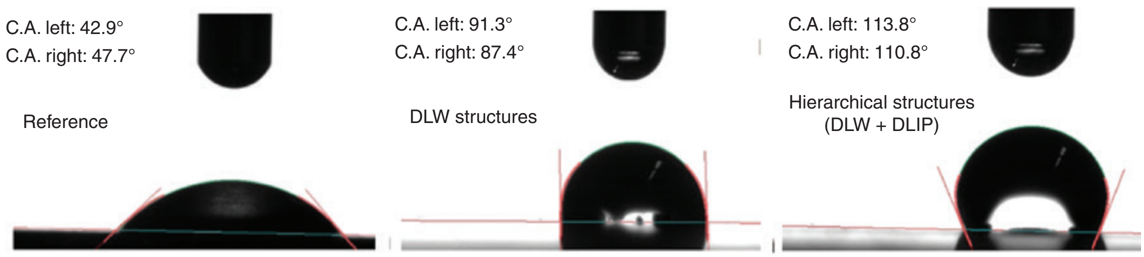

Figure 10: Long term static contact angle (SCA) results obtained for reference (left), DLW (center) and combined DLW+DLIP (right) processed samples.

properties has been performed by means of the determination of their static contact angle (SCA) measurements.

The reported SCA measurement were performed using a Biolin Scientific Theta Basic optical tensiometer dispensing a 3 μl droplet volumen. The non-patterned sample was measured as a reference and recorded a SCA value of 45.3±2.4° (Figure 10 left) thus showing highly hydrophilic character. The DLW patterned samples were hydrophilic after the laser processing, reaching a slightly hydrophilic/hydrophobic long term SCA value of 89.35°±1.95° (Figure 10 center), showing that the DLW-generated μ-cell structures, although sensitively increasing the hydrophobic character of the sample by effect of the formed μ-cells (as also recognized in previous publications by the authors [26, 33]), did not increase drastically, for the set of process parameters chosen in this case), the hydrophobic character of the material.

On their turn, the micro-/nano-scale hierarchical structures generated by means of the combined DLW+DLIP process were also moderately hydrophilic immediately after the laser process, but became clearly hydrophobic in the long term, recording a SCA value higher than 110° (Figure 10 right), thus improving the SCA value on comparison with the puwre DLW treatment. This increase on the SCA must be related to the presence of the newly generated hierarchical structures considered by the Cassie-Baxter model, as discussed, i.e. in reference [33]. Although, effectively, this new lower-scale features do positively improve the surface hydrophobic behaviour, the cause for this behaviour is mostly attributed to the initial ns-DLW treatment as shown, i.e. by Jagdheesh et al. in references [10, 33]. By means of this ns-DLW that is, essentially, a material ablation process with thermal affection causing occasional material remelting, an array of mixed μm/nm-scale microcells is generated able to efficiently trap air 'cushions' in the surface topographic structure that definitely push-away liquid dropplets and preclude the conditions for a Wentzel case, thus enabling a Cassie-Baxter one by means of a practical reduction of the effective area of the liquid-solid interface [38, 39]. In this sense, the obtained results are somehow disruptive with relation to the well known evolution of hydrophobicity with roughness in DLIP generated microstructures (as reported, i.e. by Cuello et al. [40] and Guenther et al. [41]).

3.3 Surface corrosion resistance modification

In addition to the preceding wettability analysis of samples processed by pure DLW and combined DLW+DLIP, linear polarization studies were performed on the fabricated structures to investigate the corrosion resistance behavior of both samples after either treatment.

The measurements were made using a three-electrode cell using a 0.5 M NaCl solution as electrolyte, a 316L stainless steel was used as the counter electrode, saturated sodium chloride 3 M was used as the reference electrode and the Ti6Al4V samples were used as the working electrode. The area surface exposed to the solution was 1 cm^2 and the polarization analysis was performed by varying the voltage from −0.3 to 8 V with an increment of 5 mV.

Figure 11 shows the cyclic polarization curves or Tafel plots for a representative set of processed samples (with three representative DLW HD values) in order to compare the difference in the corrosion behavior between the reference sample, the DLW-generated micro-structures and (DLW+DLIP)-generated micro-structures. The plots provide valuable information for corrosion characterization and several parameter like the pitting potential (E_{pitt}), the corrosion current density (i_{corr}), the corrosion potential (E_{corr}) and the corrosion rate can be obtained from them. Table 3 shows the values for the corrosion current density, the corrosion potential and the pitting potential for the measured samples.

According to the values displayed in Figure 11 and Table 3, it is observed that the corrosion current density for the considered DLW samples is generally much lower

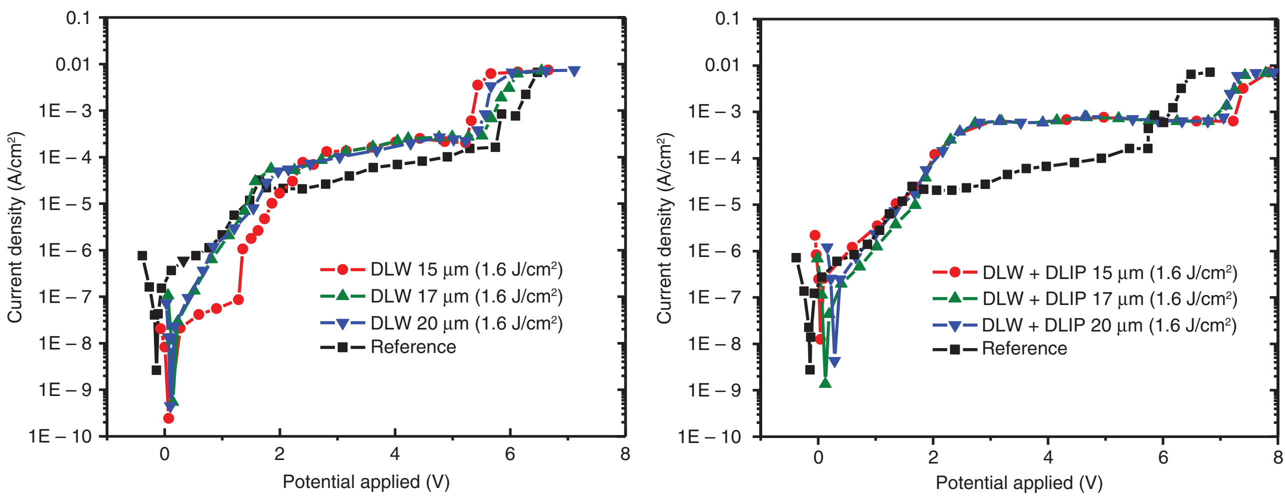

Figure 11: Polarization curves for reference sample and three values of DLW HD (15μm, 17μm and 20μm) processed at a DLW or (DLW+DLIP) fluence adjusted to a representative value of 1.6 J/cm².

Table 3: Results for the corrosion current density (i_{corr}), corrosion potential (E_{corr}), pitting potential (E_{pitt}) and corrosion rate for the considered representative samples.

Sample	i_{corr}(nA/cm²)	E_{corr} (mV)	E_{pitt} (V)	Corr. rate (mm/year)	Corr. rate (relative to reference)
Reference	11.55	−137.07	5.73	9.92E-05	100%
DLW 15 μm (1.6 J/cm²)	0.63	75.09	5.29	5.46E-06	5.51%
DLW 17 μm (1.6 J/cm²)	3.22	131.92	5.59	2.77E-05	27.93%
DLW 20 μm (1.6 J/cm²)	2.11	97.45	5.37	1.81E-05	18.29%
DLW+DLIP 15 μm (1.6 J/cm²)	36.55	39.4	7.23	3.10E-04	312.56%
DLW+DLIP 17 μm (1.6 J/cm²)	8.36	129.42	7.07	7.18E-05	72.39%
DLW+DLIP 20 μm (1.6 J/cm²)	24.65	280.58	7.05	2.11E-04	212.74%

than the reference values and, consequently, so does the corrosion rate. In the contrary, for the samples treated by the combined (DLW+DLIP) treatment, the values of this parameter is observed to be subject to a very sensitive wide variation against the DLW HD parameter, seeming to present an optimized value lower than the reference one for an intermediate choice of such parameter. Considering that the feature size characteristic of the DLIP treatment is much smaller than the characteristic feature size of the DLW process, it is presumable that the existence of a finely-tuned optimum range of values for the nano-pitch achievable by the DLIP process leading to a clear advantage over the reference value. The existence of this range is a point to be further elucidated in future research.

This advantageous behaviour against the reference can be clearly observed through the values of the pitting potential that is clearly improved by the combined (DLW+DLIP) treatment to values far exceeding the reference and DLW values. Provided that the pitting potential is defined as the potential at which a sudden increase of the current occurs after a steady behaviour of the potential-current relation, and is considered as the potential at which the material corrosion is hardly controllable, because of the appearance of multiple local corrosion hot points (pits), this improvement (increase) of the pitting corrosion potential (from about 6.0 V for the reference case to about 7.0 V for the treated samples) is considered to be both quantitatively and qualitatively very significant. The pitting process for these hierarchical structures seems to start on random places and begin to spread from there in a directional manner, but the damage to the surface is less pronounced that in the case of DLW generated microstructures (previously analyzed in reference [12]). This may be caused by the presence of the smaller DLIP micro-pillar on the center of the DLW micro-cells, which has been proven to be beneficial to prevent in some manner that water droplets make contact with the original material, thus reducing the surface area that is in contact with the electrolyte.

4 Conclusions

Multiple-scale periodic surface patterns were fabricated on Ti6Al4V alloy combining two laser micro-structuring techniques, starting from nanosecond-pulsed (DLW) to create micro-cells (50 μm width, 4–10 μm depth and 4–10 μm high) and subsequently applying a DLIP treatment. Based on the material melting due to the prevalent thermal ablation process in ns laser interaction, the initial DLW generation of μ-cells regularly distributed on the sample surface according to the procedure described by Ocaña et al. [33] was accomplished. Subsequently, picosecond-pulsed DLIP according to the procedure described by Lasagni et al. [21] was used to create micro-structures with a period of 2.6 μm. The structuring was mainly free of thermal effects and has been applied in order to create pillars with a height of 1.1 μm. The applied procedure is considered to be highly original and opening a broad field of hierarchical laser surface structuring in the micro-/nano-scale with high potentialities from the point of view of hydrophobicity and self-cleaning capability recalling well-known examples found in nature.

The microscopy analysis of the processed specimens shows clearly that the second process with DLIP does not damage the previously fabricated μ-cells using DLW. In the center of the μ-cell, where the original topography of the sample along with some recast particles was observed before, a well-defined periodic structure with much finer spatial resolution is fabricated. Although the final topographical structure of the samples treated by the combined procedure is predominantly in the micron domain, a certain lower scale degree of structuration is achieved (an approach to a true hierarchical micro-/nano-structure), so that the resulting structures present a dual micro-/nano-structure, similar to examples present in nature and are a promising texturing method for the generation of super-hydrophobic surfaces.

Concretely, in line with the previous results relative to DLW treatment published by the authors [33], static contact angle (SCA) measurements have demonstrated that the developed hierarchical structures exhibit a clear long-term hydrophobic behaviour, recording SCA values over 110°, thus allowing the creation of extense hydrophobic surfaces offering a great potential application on wettability technology.

Additionally, the fabricated hierarchical micro-/nano-structures exhibit an improved corrosion resistance behaviour relative to the reference untreated samples of the same material, especially in what concerns the pitting corrosion potential, a result which is being fully in line with previous results published by the authors [12] is considered to be as fully relevant, both from a quantitative and qualitative point of view.

Consequently, the originally developed reported dual treatment is envisaged as a really promising one for the generation of corrosion stable, truly hierarchical in the Cassie-Baxter sense surfaces with great application potential in hydrophobic self-cleaning industrial and biomedical applications.

Acknowledgements: Work partly performed in the frame of the EU funded H2020 ITN LASER4FUN Project (www.laser4fun.eu).

Funding: Funder Name: European Commission, Funder Id: http://dx.doi.org/10.13039/501100000780, Grant Number: EU funded H2020 ITN LASER4FUN 675063

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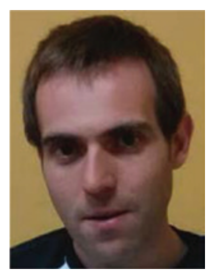

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