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Laser micro-polishing of stainless steel for antibacterial surface applications

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Abstract

In this work laser micro polishing (LMP) of cold rolled 0.3 mm thick 304 stainless steel with a pulsed fibre laser is studied, for applications where antibacterial properties are required. Due to its production method, the initial surface roughness of the tested material was considerably low (Sa=85.3±2.8 µm), rendering a demanding case for the laser polishing process. Accordingly, process feasibility under three different atmospheric conditions, namely ambient, Ar and N_2 atmosphere, was investigated. A large set of process parameter combinations was tested and initial analysis was carried out to identify the polishing feasibility by inspection under an optical microscope. Once the feasibility window was determined, a primary characterization was made on selected surfaces for roughness and waviness. Results show that in some process conditions belonging to the explored feasibility range, surface roughness could be decreased by 50%.

Keywords: laser micro-polishing, stainless steel, antibacterial surface, surface roughness

1. Introduction

Austenitic stainless steel is widely used for different industrial purposes due to a combination of good mechanical properties and excellent corrosion resistance. Its anti-corrosive properties render it suitable for a variety of applications involving biological organisms. Grade 304 is one of the most versatile and most widely used stainless steel, available in a large range of products, forms and finishes. Typical uses are in architectural field, pharmaceutical and chemical industry processing equipment, heat exchangers, woven or welded screens for mining, quarrying and water filtration, food processing equipment [1,2]. Moreover this material is employed in applications where remarkable antibacterial properties are required, thanks also to its ability to withstand the corrosive action of various acids found in fruits, meats, milk, and vegetables. 304 stainless steel is used infact for domestic tools industry, such as sinks, troughs, table tops, stoves, refrigerators, other equipment and appliance; then it is also used in hospital environment for surgical and dental instruments.

Surface finish plays an important role regarding the interaction with the surrounding environment, hence with living organisms. Bacterial adhesion is an important concern in most of the related applications and are critically influenced by numerous variables [3], like surface morphology, physicochemical properties, environmental condition and type of pathogen. About surface morphology, it has been observed that the smoother surface implies the lower probability of bacterial attachment [4]. This occurs for three reasons: i) a higher surface area available for attachment, ii) protection from shear forces, iii) chemical changes that cause preferential physicochemical interactions [5]. On the other hand it seems that bacterial attachment is enhanced when the features of the surface have dimensions similar to bacterial size [4]. Hence, improved surface finish can prevent bacterial adhesion. For all of these considerations a polishing treatment could be appropriate to achieve antibacterial surface morphology properties. Several polishing technologies allow obtaining surface finishes in the nanometer range, for example abrasive polishing, lapping, mechanochemical, electrochemical polishing, ultrasonic, and magnetoabrasive polishing [6].

Laser micro-polishing (LMP) is one of the presently available options capable to attain high surface finish levels. In comparison with other polishing techniques, laser polishing based on surface remelting shows several advantages. It is a fully automated and controlled process, with the capability to polish a well-defined area. It is a single step and quick process, without any types of contact avoiding mechanical forces at tool-workpiece interface and wear of tool, both typical problem of conventional polishing techniques. Laser micro-polishing, moreover, can be used to modify the surface chemistry, for example capturing in the molten pool molecules from an eventual gas flow [1,7,8]. However, the process of LMP has some disadvantages compared to other technologies. It is a thermal process, resulting in the formation of a heat affected zone that shows different mechanical properties compared to bulk material. Then it is strongly influenced by material initial roughness and by the possible presence of some surface defects like scratches; infact the surface finish could vary the absorption or reflection of the incident radiation [1,7,8].

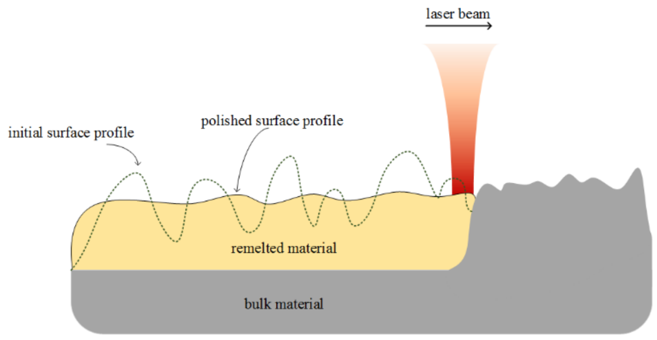

Fig. 1. Laser remelting process for polishing.

During laser micro-polishing (Fig.1), laser beam energy is delivered to workpiece surface; the molten pool of material formed tends to redistribute around the area adjacent to each initial surface asperity under the action of surface tension. This phenomenon results in the reduction of the majority of peak to valley heights and surface asperities, after the quick solidification of melted layer [7]. In this way, Yermachenko et al. demonstrated that the irradiation at appropriate parameters of the laser radiation in argon atmosphere leads to a decrease in the surface roughness of the titanium samples by a factor of five in comparison with the roughness of the original samples [9]. The same authors observed also an increase in the microhardness of the surface layer. Mai et al. presented the results of an investigation on laser polishing of 304 stainless steel surfaces, with roughness reduction of 62% [1]. Perry et al. introduced the concept of critical frequency to predict the effectiveness of polishing in the spatial frequency domain [10], showing the importance of different spatial components on the surface profile. As a matter of fact, primary profile of a surface is conventionally divided in two parts: i) roughness profile, that is simply a collection of all high-frequency components, and ii) waviness profile, that instead is a collection of small-frequency components. Roughness profile can be obtained by filtering the primary profile using a high-pass filter, while waviness profile using a low-pass one [11]. Therefore the parameter that determines what is roughness and what is waviness is the cut-off wavelength, i.e. the length at which the filters are applied and provided by ISO 4288:1996 [12].

Despite numerous works, one of the biggest challenge of the LMP process remains the achievement of low surface roughness when the initial surface morphology has already a good finishing quality, as occurred for cold rolled sheets.

In this work a fiber laser source in ns pulse regime was used to study the polishing feasibility of stainless steel surfaces, in order to obtain anti-adhesiveness properties against bacteria.

2. Experimental details

2.1. Material and systems

Stainless steel 304 alloy sheets were used throughout the study. The sheets were cold rolled to 0.5 mm thickness. The surface average roughness Sa_r was 85.3±2.8 nm, instead the surface average waviness Sa_w was 56.4±5.6 nm. The material nominal chemical composition is summarized in Table 1.

Table 1. Nominal chemical composition of employed 304 stainless steel.

Element	C	Cr	Ni	Mo	Si	Mn	P	S	N
wt.%	0.047	18.1	8.04	0.29	0.48	1.2	0.029	0.003	0.06

Before LMP, the specimens were cleaned in ultrasonic bath cleaning with deionized water (10 minutes), ethanol (10 minutes) and deionized water (10 minutes). Then the samples were dried in nitrogen.

A Q-switched fibre laser (YLP-1/100/50/50 from IPG Photonics, Oxford, MA, USA) in fundamental wavelength (λ=1064 nm) was used coupled to a scanner head (TSH 8310 by Sunny Technology, Beijing, China). The scanner head was equipped with an f-theta lens (SL-1064-70-100 from Wavelength Opto-Electronic, Ronar-Smith, Singapore), with focal length of 100 mm; so the calculated beam diameter in focal point was 39 µm. The workpiece was positioned in Z-axis with L490MZ/M motorized lab jack from Thorlabs, inside a gas chamber. The main specifications of the employed laser system are summarized in Table 2.

Table 2. Main specifications of the employed laser system.

Wavelength	λ	1064 nm
Maximum average power	P_{max}	50 W
Pulse duration	τ	250 ns
Pulse repetition rate	PRR	20-80 kHz
Quality factor	M^2	1.7
Collimated beam diameter	d_c	5.9 mm
Focal length	f	100 mm
Focused beam diameter	d_0	39 µm

The chamber, designed and built in laboratory, allows working under inert atmosphere and avoiding in this way the material oxidation; infact a flux of gas is generated and passes through the sample during treatment. The design of chamber was focused particularly on dimensions, considering first of all the limitations due to the necessity to integrate the chamber in the employed laser system. Fig. 2 reports the assembled laser system.

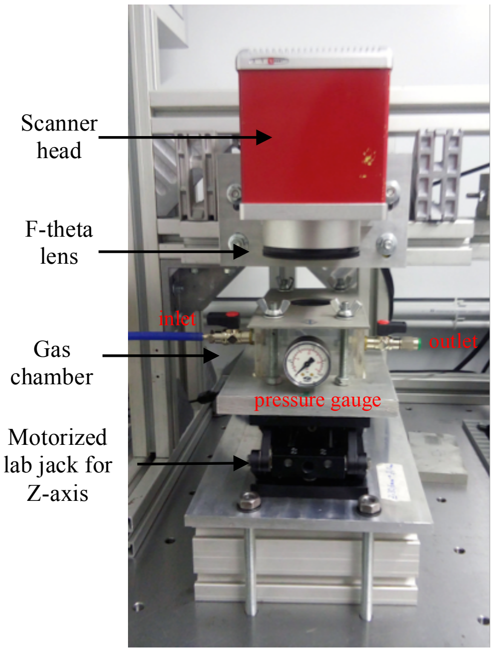

Fig. 2. The assembled laser system.

For surface characterization, a first visual analysis was conducted to classify treatments in polished and unpolished surfaces. Then focus variation microscopy was tentatively used for surface morphology acquisitions and roughness measurements. Due to the high reflectivity of the polished surface the focus variation microscopy was not appropriate to operate directly on stainless steel samples. Therefore the surfaces were replicated on silicon-based rubber and measured through the replicated rubber surfaces. Parameters values were calculated applying an high-pass filter (for roughness Sa_r and Sz_r) and a low-pass filter (for waviness Sa_w and Sz_w) to the surface profile at a cut-off wavelength of 29.14 µm, according to the ASME B46.1-2002 [13]. In particular parameters were calculated over a selected area of 110.61x145.81 µm^2, through area averaging method.

2.2. Experimental plan

An experimental plan was conducted to define the feasibility of LMP. In this explorative work laser beam was focused 2 mm above the surface to generate a larger spot and to reduce energy intensity on the material surface; in this way material removal was avoided and processing conditions yielded melting of a superficial layer. The calculated beam diameter d_s on the workpiece was 124 µm. Laser pulse energy (E) was maintained between 0.10 and 0.64 mJ.

LMP surface structuring consists of scanning the laser beam on a two dimensional plane to overlap laser pulses on the material. Scanning speed (v) was changed from 25 to 6000 mm/s . The overlapping of the successive scan lines depends on the pitch (p) which was set as 10 µm in all experiments. It is also possible to increase the number of passes (N) on the scanned area, with different angles to eliminate directionality on the surface. Within this study, single passes (N=1) were done at 0°, which was defined as perpendicular to the grinding traces on the material. In multiple passes, the scan angle followed 0° and 90° (N=2) or 0°, 90°, 45° and 135° (N=4). A schematic representation of scanning strategy can be seen in Fig. 3.

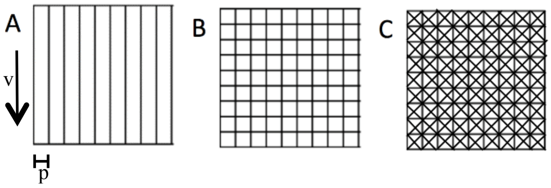

Fig. 3. Scheme of the employed scan angle strategies with single pass (A), double passes (B) and four passes (C).

The experiments were conducted under atmospheric conditions (without shielding gas), under argon and under nitrogen; however, with argon and nitrogen, the gas pressure was set constant at 0.3 bar and measured through a pressure gauge placed inside the gas chamber. Different processing conditions were applied in 3x3 mm^2 squares.

The Table 3 summarizes the defined experimental plan, classifying fixed, varied parameters and measured variables.

Table 3. Defined experimental plan.

Fixed parameters		
Pulse repetition rate	PRR	65 kHz
Pitch	p	10 µm
Focal position	Δz	2 mm
Gas pressure	-	0.3 bar
Varied parameters		
Pulse energy	E	7 levels: 0.10-0.64 mJ
Scanning speed	v	9 levels: 25-6000 mm/s
Number of passes	N	3 levels: 1, 2 or 4
Gas type	-	3 levels: None , N_2, Ar
Measured variables		
Categorically classification in polished and unpolished		
Sa_r, Sz_r, Sa_w and Sz_w on chosen surfaces		

In order to assess the effect of laser process parameters and define the feasibility of LMP, results were analyzed categorically through visible inspection under optical micorscope, with polished (P) and unpolished (U) states as output. Fig. 4 reports optical microscopy images showing example surfaces belonging to the defined categories. As can be observed in Fig. 4.a, a polished surface is smooth, free of surface macro-defects, and surface irregularities from the previous manufacturing processes are absent. On the other hand, an unpolished surface corresponds to surfaces with excessive melting, often accompanies by oxidation and roughnening, as depicted in Fig. 4.b. For a preliminary characterization, on chosen surfaces Sa_r, Sz_r, Sa_w and Sz_w were measured.

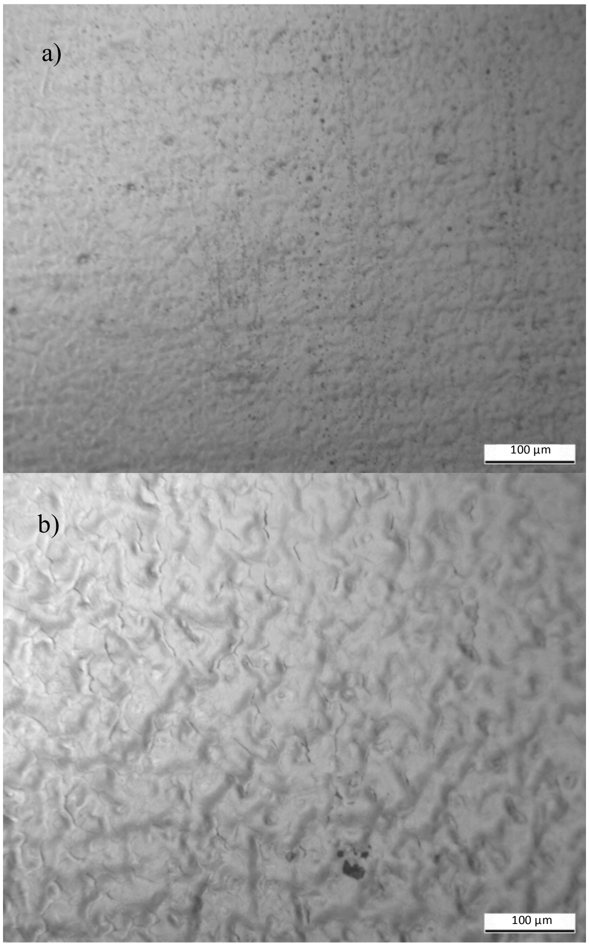

Fig. 4. Optical microscopy images of a polished (a: N_2 E 0.19 mJ v 400 mm/s N 2) and an unpolished (b: None E 0.19 mJ v 200 mm/s N 1) surface.

3. Results

Fig. 5 collects the feasibility maps belonging to different atmospheric conditions. All treatments in ambient atmosphere were excluded, since they were oxidised and showed macro-defects. Other authors have also observed that LMP could generate some defects on surfaces, as cracks and pores, when process was carried out in atmospheric air, due to the large thermal gradients and residual stresses generated by the oxides formation at the material surface [10,14]. It can be observed that the energy level of 0.64 mJ is too high and also under inert gas the laser processing seems to be too strong, generating surface colorization and macro-defects, despite the use of the process gas. The same considerations can be done for scanning speed levels of 25, 50 and 100 mm/s that result in high overlapping value and so in very energetic treatments. Instead, the energy level of 0.10 mJ seems to be too low, resulting infact only in a soft heating of material.

Thus excluding these levels, the number of conditions that can be defined as polished was reduced and the main guidelines for polishing were defined:

- Treatment should be carried out under shielding gas (argon or nitrogen);
- Pulse energy in a range from 0.15 mJ to 0.47 mJ should be employed;
- Scanning speed in a range from 200 mm/s to 6000 mm/s is suitable

For a preliminary characterization within the feasibility window, four surfaces were chosen, two obtained in argon and two in nitrogen respectively, maintening the same laser parameters for both gas conditions. In particular the two extreme values of energy were selected, while for the other factors (v and N) intermediate ones.

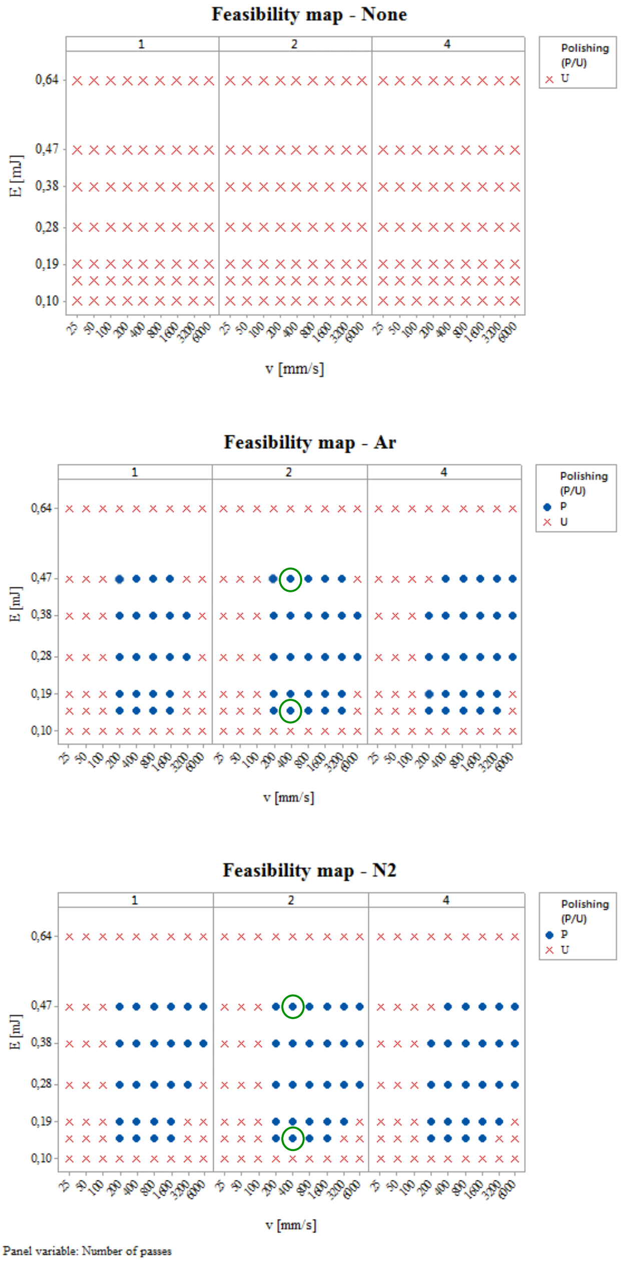

Fig. 5. Polishing map, where green circles represent the selected surfaces for a preliminary characterization.

In Table 4, the measured roughness and waviness values, with relative standard deviation, are reported. Fig. 6 reports the relative acquisitions through focus variation microscopy of selected surfaces and of bulk material. It can be observed that both Ar and N_2 are effective in reducing the surface roughness, once the laser parameters are opportunely regulated. In these conditions surface roughness could be reduced approximately by 50%. On the other hand, with increased energy level, surface roughness was found to be higher than the initial value, showing that the LMP can produce higher surface roughness, despite the fact that surface is visually brighter and smoother.

Overall, it can be observed that LMP treatment consists first of all in the removal surface defects inherent from the previous cold rolling process. Doing so, the reduction of peaks was possibly. This is attributed to the fact that during laser remelting process, the molten pool tends to fill the initial surface asperities, resulting in a roughness decreasing. However the process can also induce higher roughness and waviness

Another important aspect is the variability of the surface roughness and waviness indicators among the surface. As a matter of fact, surface inhomogeneity manifests not only in terms of high values of surface roughness and waviness but also high values of standard deviation. As can be observed from Table 4, in improved conditions (Ar, E=0.15 mJ, v=400 mm/s, N=2) surface roughness and waviness decreases. More remarkably, the variability within the surface especially for Sz_r and Sz_w decreases in this condition. On the other hand, with higher energy conditions (Ar, E=0.47 mJ, v=400 mm/s, N=2) the LMP process induces an increase in surface variability as seen by the highly increased standard deviation in waviness indicators. Such variability, especially in the waviness profile can be attributed to the liquid instabilities and defect generations due to high energy input. In this regard Nüsser et al. [15] underlined that surface micro surface defects can occur during laser treatment, in the form of undercuts, holes or increased waviness. Finally, in the experimented example conditions, LMP process was found to be more efficient in terms of roughness reduction, rather than waviness.

Table 4. Examples of obtained roughness and waviness values, compared to bulk material.

Gas type	Non treated	Ar	N_2	Ar	N_2
E [mJ]		0.15	0.15	0.47	0.47
v [mm/s]		400	400	400	400
N		2	2	2	2
Sa_r [nm]	85.3 ±2.8	42.0 ±4.1	52.8 ±3.5	77.3 ±9.2	71.8 ±7.8
Sz_r [nm]	1113.6 ±233.0	544.1 ±40.0	680.9 ±105.7	812.5 ±188.0	615.1 ±47.0
Sa_w [nm]	56.4 ±5.6	40.0 ±6.9	56.6 ±7.3	199.4 ±125.7	218.4 ±65.6
Sz_w [nm]	625.3 ±70.2	344.5 ±41.1	392.2 ±71.3	1749.1 ±1110.5	1555.3 ±550.4

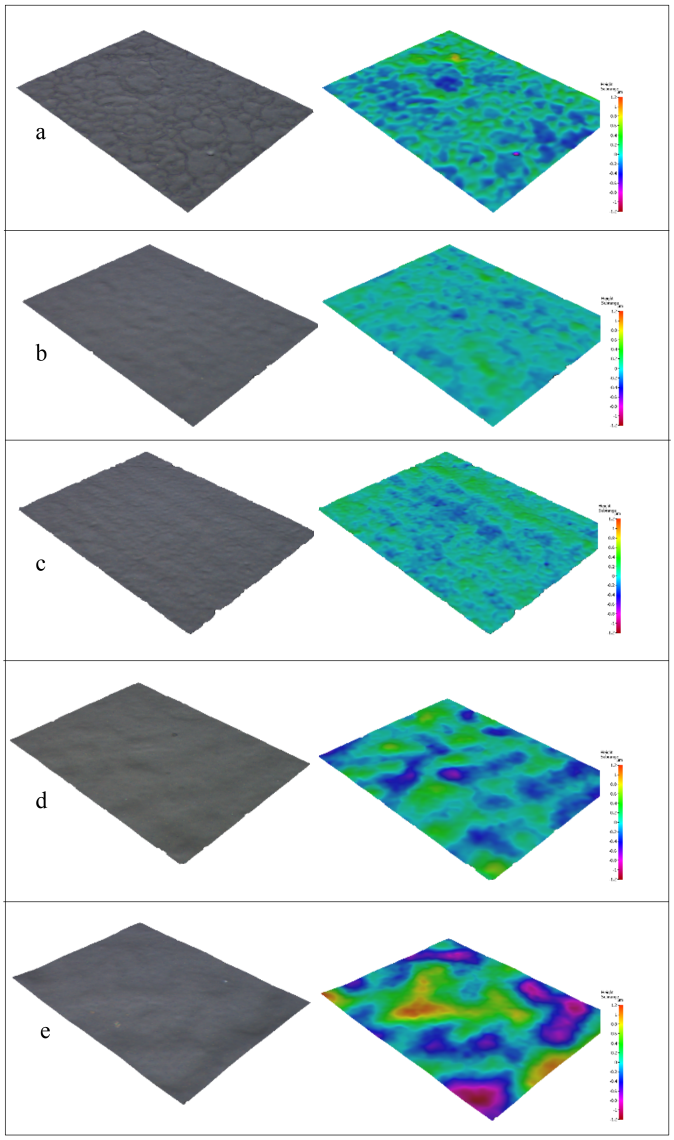

Fig. 6. Examples of laser polished surfaces acquisition through focus variation microscopy, both in real colors and in color map: a) bulk material; b) treatment under argon E=0.15 mJ, v=400mm/s, N=2; c) treatment under nitrogen E=0.15 mJ, v=400mm/s, N=2; d) treatment under argon E=0.47 mJ, v=400mm/s, N=2; e) treatment under nitrogen E=0.47 mJ, v=400mm/s, N=2.

4. Conclusions

LMP of 304 stainless steel was investigated using different process parameters such as laser pulse energy, scanning speed, shielding gas flow and scanning strategy. The perfomance of LMP treatment in improvement of the surface finishing was successfully demonstrated, also for an already good initial surface roughness. As observed with focus variation microscopy, this is due to the filling of surface asperities and grain boundaries with the molten material surface during remelting process.

In this study the surfaces were first classified in polished and unpolished, through a simple visual analysis, allowing the definition of a process feasibility map. Polished surfaces can be obtained, in particular, working under both Ar and N_2 and thus avoiding the material oxidation. In this preliminary analysis under Ar, roughness and waviness could be decreased by 50% and 29% respectively. With the optimisation of the inert gas and process parameters the decrease in roughness is expected to further increase.

A part the optimisation of the process, future works will be dedicated to evaluate the functionality of laser micro-polished surfaces in the context of antibacterial applications.

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