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6th CIRP Conference on Surface Integrity

Electropolishing of 316L stainless steel parts elaborated by selective laser melting: from laboratory to pilot scale

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Abstract

Electropolishing is an effective technique for surface finishing of additively manufactured parts, compatible with complex geometries. It consists of an electrochemical dissolution in which the part to be treated is polarized anodically.

The present study focuses on the development of an electrofinishing process dedicated to 316 L stainless steel elaborated by SLM. A study, performed at laboratory scale, allowed to characterize the electrochemical behavior of raw substrates (produced according to different laser scan strategies) and to define the bests operating conditions for the levelling (electrolyte composition, temperature, electrical parameters, duration...) with acceptable dissolution rates (around 5 $\mu\text{m}/\text{min}$). The transposition to a pilot unit able to process samples of several square centimeters (plates or tubes) requires a precise recalibration. Difficulties are essentially due to the high roughness of the SLM substrates ($R_a \approx 30 \mu\text{m}$, $R_z \approx 200 \mu\text{m}$), but also to issues related to the scale-up such as the current lines distribution that cause an inhomogeneous dissolution. To fit with the double objective of roughness decrease and geometrical integrity preservation, the use of pulsed potential shows an excellent efficiency. In such conditions, a 90% roughness decrease was measured while better preserving the shape integrity.

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Keywords: Post-processing ; electropolishing; SLM; Stainless steel

1. Introduction

As additive manufacturing (AM) technologies are effective to produce customized component with highly complex shapes in a wide range of alloys (stainless steels, titanium alloys, nickel-based superalloys, aluminum alloys, ...), more and more industries are integrating this technology into their production lines. This is particularly the case in aircraft, aerospace and biomedical sectors [1].

However, while technological advances have led to AM components whose core mechanical and structural properties are almost equivalent to mechanically manufactured, there remains a major obstacle to the extension of the process, which

is due to the extremely degraded surface properties of the parts produced. AM components currently exhibit high roughness values (R_a up to 30 μm), a strong texture (directly connected to the process type and manufacturing parameters) and potentially detachable unmelted particles. These surface defects are responsible for a degradation of the material functional properties, such as an increased sensitivity to fatigue failure [2,3] and corrosion [4–6], which are incompatible with the desired applications. The use of a finishing step to improve overall surface integrity, including in channels and holes, is therefore essential for the deployment of AM in industry. It is also a significant cost item that is a key component of the value chain.

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Electrochemical polishing (EP), also known as electropolishing, is an electrolytic process of anodic dissolution, based on a controlled removal of materials, which induces roughness reduction and gives to the workpiece a smooth and shiny appearance. It is currently implemented as an ultrafinition process of AISI 300 stainless steels used in food, pharmaceutical, and biomedical industries. It is now experiencing a renewed interest for the surface finishing of parts produced by additive manufacturing as evidenced by the increasing amount of papers dedicated to this topic [7–10]. Aside from leveling the surface, electropolishing is promising because, as a non-contact technique, it leads to a stress relieved surface more resistant to fatigue [2]. Moreover, on passive materials such as stainless steels, the dissolution of the defective surface oxides layer and its replacement by a dense, and homogenous passive film (enriched with chromium) enhance the corrosion resistance [11]. EP is applicable to a large variety of alloys (even the more chemically resistant like Inconel 718 [12] or Ti-6Al-4V [13]) provided to adapt the electrolyte composition to each of them and to adjust the polarization electrical parameters (current, voltage or potential). It also allows the use of less harmful electrolyte than chemical polishing because the current passing through the part act as a dissolution promoter. Theoretically, according to Faraday's law, the amount of dissolved material is directly proportional to the current applied and to the duration of the process.

However the treatment of real AM parts of complex shape, at larger scale, reveals some shortcomings of the electropolishing process that are more rarely addressed [8,13–15]. High initial roughness of AM components requires to extend the electropolishing time to expect recovering a smooth surface. But EP can sometimes appear to be a self-limiting process that struggles to eliminate the residual undulation [16]. A long process is also associated to a temperature increase in the reactor and to excessive dissolution product build-up. The treatment of internal channels requires first a good penetration of the electrolyte and then the possibility to introduce a counter-electrode (cathode) [14]. Finally, achieving a uniform polishing on the entire surface is a key point that should be raised. Like in all galvanic processes, the local current distribution is dependent on the cell configuration, the electrodes geometries and spacing and on the stirring conditions. As current lines accumulate on the sharp edges of the parts, this leads to a loss of geometrical integrity [17,18].

This paper presents the methodology used to develop an electropolishing process for high roughness 316 L selective Laser Maelting (SLM) components (centimetric plates). After defining the optimal operating parameters in a laboratory cell, the process was upscaled in a pilot reactor. It highlights that the best electrical control to avoid surface and geometrical effects is to use potential regulation instead of current or voltage. Polishing quality evaluation metrics were defined to be the arithmetic surface roughness (R_a , S_a) and the shape preservation by local thickness loss measurements. Substrates electropolished under pulsed polarization were characterized and compared to the ones treated in direct mode.

2. Working methodology

2.1. Materials preparation and characterization

The process was first developed at laboratory scale on small cylindrical parts ($\varnothing = 8$ mm, 5 mm thick), to be inserted in a rotating disk electrode (RDE) holder. The samples, provided by Volum-e, were made of 316L stainless steel (1.4404) and manufactured by Selective Laser Melting (SLM).

Two build orientations were available to us, characterized by the angle between the surface to be studied and the manufacturing plate, as illustrated in Fig. 1 (replicate from [19]). For $\theta = 0^\circ$, the surface is parallel to the tray (up-facing surface) and for $\theta = 135^\circ$, it is relative to a down-facing surface.

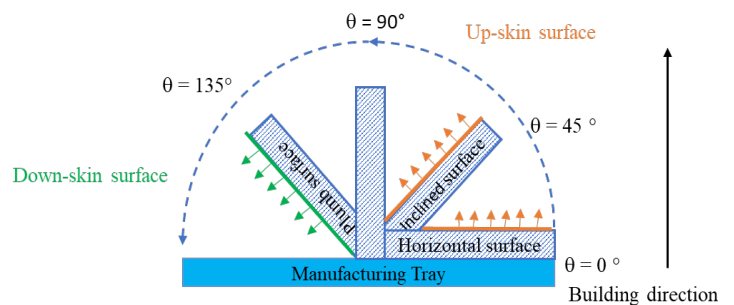


Fig. 1. Representation of the surface build orientation ($\theta = 0^\circ$, $\theta = 135^\circ$)

As shown in figure Fig 2, the surface morphology and the roughness of the samples are highly dependent on the building orientation. The horizontal surface presents misoriented macro streaks in which laser pass are visible whereas the inclined one exhibits a more homogenous texture but with numerous unmelted particles.

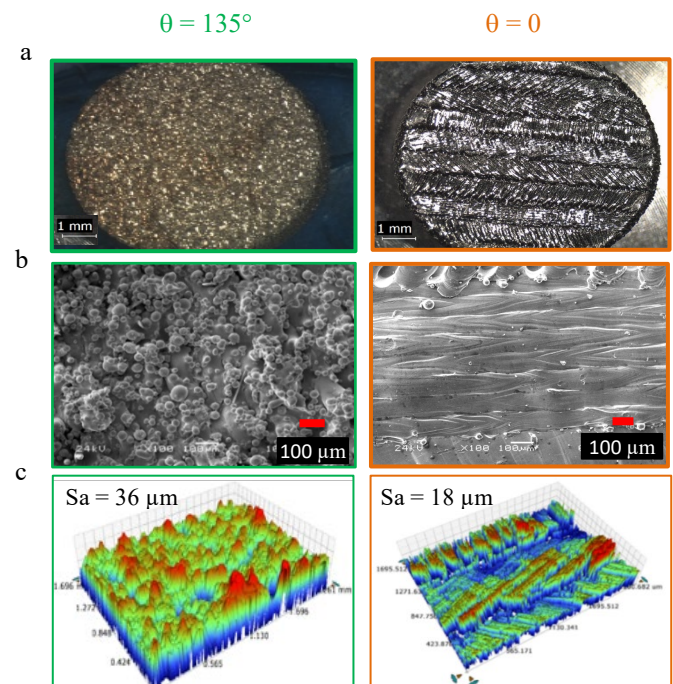


Fig. 2. Topography of surfaces elaborated at $\theta = 0^\circ$ (right), $\theta = 135^\circ$ (left). a) macroscopic views b) SEM pictures c) 3D view and S_a roughness values.

Initial surface roughness parameters were determined thanks to a green light interferometric microscope (Bruker Contour GT) with only a shape correction and then compared to stylus profiler values (Veeco Dektak 150) according to EN ISO 4288 standard (measurement length 40 mm, cut-off = 8 mm). Raw samples surface roughness parameters are respectively of $Sa/Sz = 18 \pm 1 \mu\text{m}/181 \pm 2 \mu\text{m}$ and $Sa/Sz = 36 \pm 1 \mu\text{m}/375 \pm 6 \mu\text{m}$ for horizontal and inclined surface. This corresponds to $Ra/Rz = 13 \pm 1 \mu\text{m}/157 \pm 3 \mu\text{m}$ for the horizontal surface and $Ra/Rz = 30 \mu\text{m} \pm 2 / 330 \pm 8 \mu\text{m}$ for the inclined one.

The tests pieces electropolished in the pilot were SLM 316L plates provided by another supplier (see Fig. 3). They were all manufactured along a vertical build direction (BD). Their initial roughness was determined on 3 plates and 6 measurements by plates leading to $Ra = 32 \mu\text{m} \pm 2 \mu\text{m} / Sa = 27 \pm 1 \mu\text{m}$.

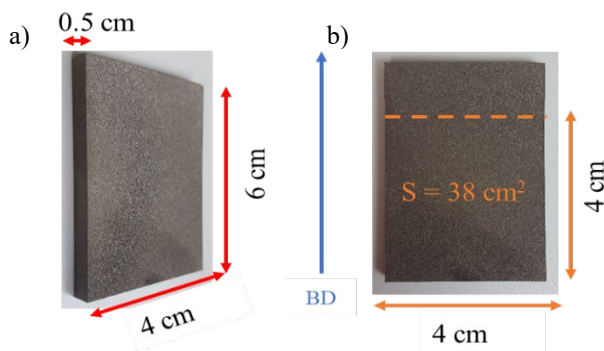


Fig. 3. Plates of 316 L SLM a) side view b) front view. Dashed line indicates the immersion line and the corresponding electropolished surface

All samples present a chemical composition in accordance with AISI 316 L alloy specifications, namely 16.5 – 18.5% chromium, 10 – 13% nickel, 2 – 2.5% molybdenum, less than 2 % Manganese, trace elements and balance iron.

2.2. Electrochemical procedure at laboratory scale

The laboratory electropolishing set-up consists in a double wall glass cell equipped with a three electrodes system including:

- a saturated calomel reference electrode (SCE, $E = 0,240 \text{ V/ESH}$)
- a platinum-coated titanium mesh as the counter electrode
- a rotating disc working electrode (500 rpm) with a sample holder featuring a $0,283 \text{ cm}^2$ working surface.

Samples embedded in the rotating tip are those presented on Fig. 2. They were only degreased in alcohol then dried to keep their raw original surface roughness.

The electropolishing solution is a mixture of 45 wt% H_3PO_4 , 35 wt% H_2SO_4 and 20 wt% H_2O , kept at $70 \text{ }^\circ\text{C}$, which has already been used for 316L SLM parts finishing [20]. To study the electropolishing mechanism and its kinetics, linear sweep voltammograms were recorded thanks to a potentiostat (Biologic SP 300). The resulting current density vs potential curve (j-E curve) presents a current plateau corresponding to the potential range where smoothing occurs. Horizontal and inclined samples were then electropolished during 20 min at different potential to determine for each one the best

conditions. For each trial, weight loss, roughness parameters (Ra , Sa) and microscopic top view (SEM) were recorded.

2.3. Electropolishing process at pilot scale

Electropolishing of plates was performed in a pilot unit (1,5 L) equipped with a temperature regulation. The electrolyte circulation and renewal are achieved by a pump with a flow rate of 17,2 L/min. Inside the vessel, the SLM part to be treated (38 cm^2 area) is placed between to cathodes made of platinum-plated titanium grid (Fig. 4). A power potentiostat 40 A/30 V (SBL, Micronics Systems) allows to drive the process either in current (I), voltage (U) or potential (E) mode. In this last configuration a reference electrode (SCE) is also introduced in the vicinity of the anode and connected to reference output of the generator. The generator can also work in pulsed and reverse pulsed modes.

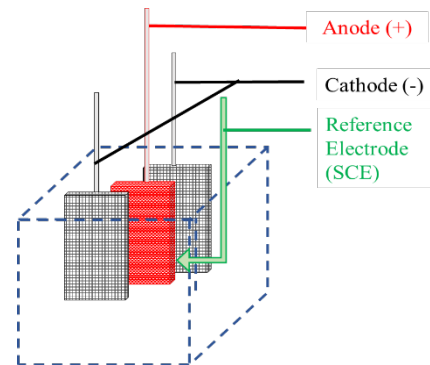


Fig. 4. Scheme of the electrodes set-up in the pilot.

2.4. Test-parts characterizations after EP in the pilot

Roughness remains the predominant criterion for assessing quality and defining the duration of treatment. In this study, like for laboratory samples, this was measured either by stylus profilometer (Veeco Dektak 150) to give arithmetic roughness value (Ra), and other amplitude parameters (Rz , Rv , Rp , according to *ad hoc* standard) or by green light interferometric microscopy to obtain equivalent surface roughness parameters (Sa , Sz , $Sp...$).

For industrial polishing, the single criterion of target roughness is no longer sufficient to qualify the process. Additional dimensional criteria must be considered, one is the average thickness reduction, and the other is the shape preservation of the part. The dissolution, which leads to the desired reduction of roughness, must be as homogeneous as possible. This point is barely mentioned in the literature dedicated to the finishing of additive manufacturing parts. It particularly concerns parts with sharp edges such as plates. Dimensional surveys by 3D laser scanner allow to evaluate the respect of the geometric integrity by measuring the deviation at each point before and after the leveling treatment. Numerical models of the parts were recorded thanks to a Solutionix Rexcon DS3 3D scanner (0,01 mm resolution). Then, 3D images were numerically compared using the Geomagic Control X (3D Systems) software (Fig. 5). From data processing, an average thickness reduction was first measured (Fig 6.a), as it is the most obvious dimensional criteria.

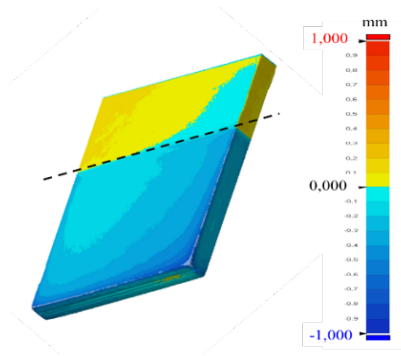


Fig. 5. 3D numerical comparison between raw and electropolished test plates. Dashed line indicates the limit between unpolished and polished zones (dip line)

A 2D integrity coefficient was also extracted, corresponding to the extra consumption of material in the sharp edge of the parts (Fig.6b).

It was calculated as follow:

$$\gamma = \frac{L - l}{L} \times 100$$

Where :

γ : the integrity coefficient%

L : the distance between the initial angle and the real angle after EP.

l : the distance between the theoretical angle (supposing a uniform thickness loss) and the real angle after EP

As defined, γ tends toward 100 %, when no distortion of the initial angle is observed.

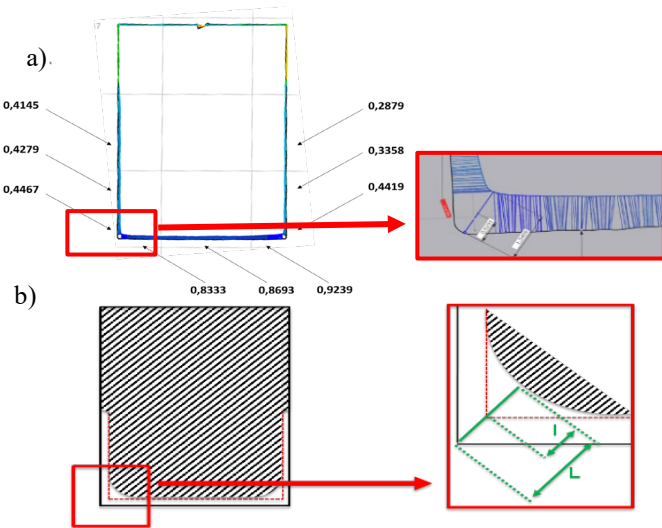


Fig. 6. a) 2D numerical comparison between raw and electropolished plates (values of local thickness loss in mm), b) Scheme of the integrity coefficient determination

3. Results

3.1 Determination of the best EP conditions at laboratory scale

Figure 7 below presents the polarization curves obtained on inclined and horizontal raw SLM samples. The j-E curves exhibit the typical behavior of the electropolishing phenomenon characterized by a current plateau between 1.6 V/ECS and 2.2 V/ECS.

The current density on the plateau (0.3 A/cm²) calculated by dividing the current by the geometrical area of the electrode, appears constant from one sample to another.

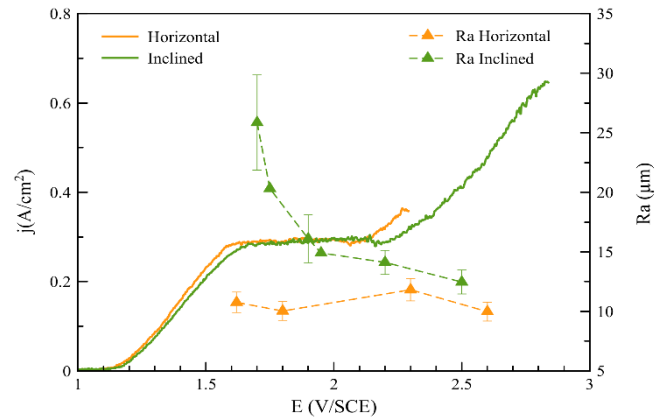


Fig. 7. Polarization curves of inclined and horizontal raw SLM samples at 70 °C in H₂SO₄/H₃PO₄ electrolyte and corresponding roughness after EP at different potentials during 20 min.

On the contrary, the roughness evolution vs treatment potential (Fig 7, right axis), shows a dependence to the building direction. For inclined samples of high initial roughness (Ra =29.7µm), an exponential decay is observed. A roughness reduction of 50% is obtained after 20 min at E = 2.2 V/SCE. For horizontal specimens of less than 20 µm initial roughness, the roughness reduction is only 20 % and remains constant with the potential. These observations are to be correlated with the surface topography of the samples (Fig. 2). Horizontal samples profile is characterized by defects of low amplitude and low spatial frequency that are therefore less sensitive to electropolishing, even if an increase of brightness is observed. Conversely, inclined surfaces exhibit a profile of high amplitude and a high spatial frequency. They respond better to the leveling phenomenon that is associated with a concentration of current lines on the profile peaks. The final roughness after 20 min of EP, is between 10 µm (for horizontal samples) and 15 µm (for inclined ones) in connection with initial roughness.

Surface profilometry after treatment also shows that electropolishing hardly eliminates low spatial frequency waviness (Fig. 8). On both samples, best results are obtained at potentials starting from the end of the EP plateau. Higher potentials, in the range of solvent oxidation also give good results. The dissolution rate measured experimentally is between 3.5 µm/min and 5 µm/min as the potential increases on the polishing plateau. This is in relative agreement with theoretical calculations (4 µm/min).

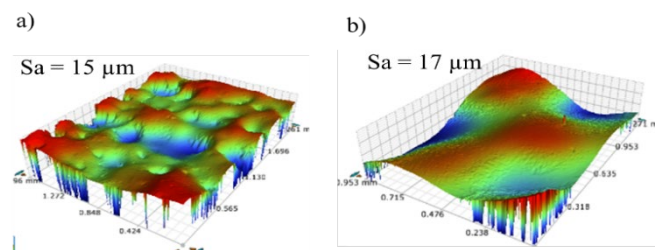


Fig. 8. 3D view and Sa roughness value of a) inclined and b) horizontal samples after a 20 min electropolishing at the end of the EP plateau (2,2 V/SCE).

All these results have shown that it is possible to electropolish raw additive manufacturing parts by adjusting the operating parameters, particularly by controlling the potential. Initial surface topography, both in amplitude and spatial frequency, appears to be a major element that determines the optimization of the process. The transition to pilot scale, on larger specimens, will therefore require a recalibration of the process in potential as well as in duration.

3.1. Electropolishing at pilot scale

Pilot calibration

Thanks to the power potentiostat, it was also possible to plot $j = f(E)$ and $j = f(U)$ polarization curves on large test plates directly in the pilot unit. Polarization curves (Fig 9.) obtained for different inter-electrodes gaps clearly showed that the j - E curves are almost independent of the pilot configuration contrary to the current-voltage curves that are offset by the ohmic drop between the electrodes. Monitoring the process in potential rather than in voltage allow to get rid of the pilot configuration and to the initial area of the electrode.

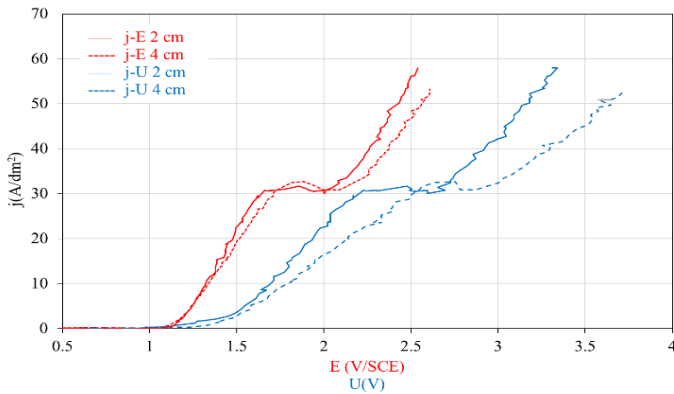


Fig. 9. Polarization curves $j = f(E)$ and $j = f(U)$ for different inter-electrodes distances ($d = 2$ cm or 4 cm) in the pilot (70°C , same electrolyte)

For further experiments, the distance between each side of the anode and the facing cathodes was fixed at 4 cm and the cathode-anode surface ratio (Sc/Sa) was 1.5 .

Electropolishing at constant potential

Preliminary investigations have shown that the best EP potential in the pilot is 3V/SCE . Fig 10a is related to roughness and thickness loss evolution as a function of time. An exponential like decrease of roughness is observed as often reported in literature [21],[22]. After a 60 minutes electropolishing, the roughness is still $5\ \mu\text{m}$ and the treatment must be pushed to 120 minutes to reach the target roughness (between 1 and $2\ \mu\text{m}$). This low roughness is associated with a high thickness loss (about $200\ \mu\text{m}$) which logically corresponds to the maximum roughness (Rz) of the profile. However, this reduction varies very little after 40 min of treatment, while the roughness continues to decrease. This is related to the dissolution rate which decreases over time (from 10 to $1\ \mu\text{m}/\text{min}$).

The integrity coefficient, relative to an extra consumption of material in the angles of the specimen (Fig. 10b), shows a sharp drop after 40 minutes (from 80 to 40%) before stabilizing like it does for the thickness reduction.

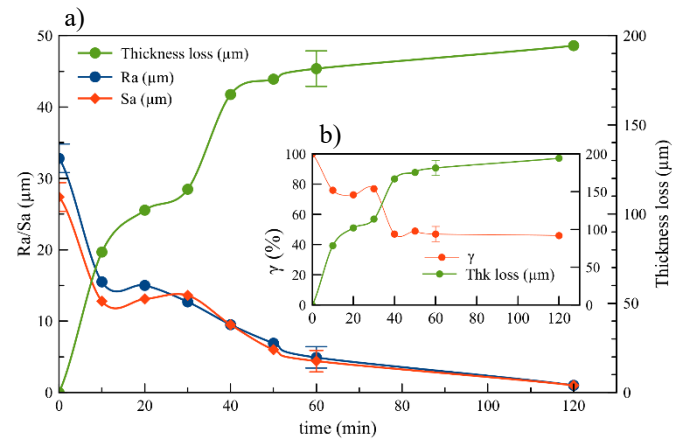


Fig. 10. a) Roughness and thickness loss evolution versus EP duration at $E = 3\text{V/SCE}$. b) Inset: associated integrity coefficient variation.

Electropolishing under pulsed potentials

Similar experiments were reproduced using pulsed potentials. The sequence consists in interrupting the anodic polarization by an off-time at open circuit potential (OCP).

The signal is defined by its peak potential (3V/SCE), its frequency f and its duty cycle (DC):

$$f = \frac{1}{t_{on} + t_{off}} \quad DC = \frac{t_{on}}{t_{on} + t_{off}}$$

where t_{on} and t_{off} are respectively the on and off time.

Nine polishing sequences were tested corresponding to three Duty Cycles (DC) 25% , 50% and 75% and three frequencies of 0.5 , 5 and 50 Hz. Samples were electropolished for one hour. It appears that DC is the major factor that influences roughness reduction (Fig. 11). For a given DC, decreasing the frequency allow to reach lower roughness. The best electropolishing conditions were determined to be DC 75% and $f = 0,5$ Hz, for which a $2\ \mu\text{m}$ roughness is reached in 60 min.

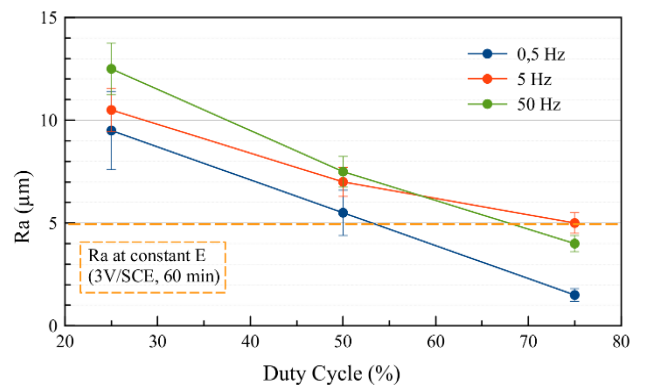
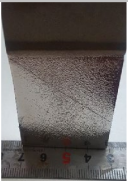
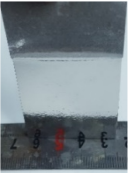


Fig. 11: Influence of pulse parameters on roughness reduction.

Comparison of test plates after EP in direct and pulsed mode (Table 1) shows that in addition to a lower roughness, samples electropolished under pulsed potential exhibits a slightly higher integrity coefficient.

From the point of view of energy consumption, this mode is also more favorable as the electrical charge required for the same results is 67% lower than in direct mode.

Table 1. Comparison between specimens electropolished at constant and pulsed potential during 60 min.

	Roughness Ra (μm)	Integrity coefficient γ (%)	Charge quantity (A.h)	Specimen picture
After EP (3V/SCE)	$4.9 \pm 1,5$	47 ± 5	9.2	
After Pulsed EP (3V/SCE, DC75%, 0,5Hz-)	2.0 ± 0.5	64 ± 5	6	

4. Conclusion

The aim of this study was to develop an electropolishing process dedicated to 316 L SLM parts of high roughness. It was first developed at laboratory scale then up-scaled in a pilot unit able to process parts of larger areas (several tenth of cm^2).

- At laboratory scale an electrochemical three electrodes assembly was used. The polarization curve (I-E) is a useful tool to select the best range of operating parameters, particularly the electropolishing potential.

- Increasing the potential along the plateau where the current density remains constant (0.3 A/cm^2) on the j-E curve provides good leveling and better brightness. The initial surface roughness of samples also appears to be a major element that influences the process optimization.

- A pilot unit (1.5 L) was developed allowing to perform EP on test plates of centimetric size. A dedicated power generator (40 A/30 V) including current, voltage and potential control enabled to confirm the interest of monitoring the process in potential rather than in voltage which paves the way to further implementation at industrial scale.

- Plates of high initial roughness (Ra about $30 \mu\text{m}$) were successfully electropolished in 60 min at 3 V/SCE to obtain Ra close to $5 \mu\text{m}$. However, the roughness reduction is associated to a loss of geometric integrity particularly at sharp edges of the samples.

- This drawback has been partially overcome by using a pulsed potential sequence (3 V/SCE, DC 75 %, $f = 0,5 \text{ Hz}$) which improves both roughness reduction and integrity coefficient while saving electrical energy.

Further studies, devoted to roughness characterization by power spectral density (PSD), will explain how pulsed potentials act on both surface roughness and shape preservation.

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