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# POST-PROCESSING OF TITANIUM MICROPARTS THROUGH ELECTROCHEMICAL AND CHEMICAL POLISHING

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**Abstract.** *Cutting processes normally require post-processing in order to eliminate burrs on the surface. In the laser cutting process also exists formation of a re-melted layer in the cutting region due to the solidification of the ejected molten material. To remove burrs and remelted layers, surface finishing methods can be used, such as chemical polishing, electrochemical polishing, mechanical polishing and laser polishing. This work aims to analyze the electrochemical and chemical polishing as a post-processing technique to improve the surface integrity of 0.1 mm thick laser machined microparts. The microparts consists of a titanium clamp used to attach a sensor to the middle ear in the case of fully implantable hearing devices (hearing aids and cochlear implants). Due to geometry and thickness of the workpiece, the post-processing is hampered. The electrochemical polishing was carried out by a solution composed by sulfuric acid as the process electrolyte and a stainless-steel piece for the electrode. Chemical polishing was carried out by immersing the parts in a solution of hydrofluoric acid (HF) and nitric acid (HNO<sub>3</sub>), in two different concentrations, for about one minute. Characterizations through optical microscopy and scanning electron microscopy were performed to evaluate the part integrity before and after polishing. Results show an improvement of workpiece quality in relation to burrs and re-melted layer.*

**Keywords:** *surface integrity, chemical polishing, electrochemical polishing, laser cutting, titanium.*

## 1. INTRODUCTION

Laser machining includes processes such as cutting, texturing and drilling and could be used in different materials. The laser machining process is characterized as a thermal removal process in which surface material is removed through melting, ablation and vaporization (MDC, 2011).

However, despite being the process with thermal removal that least affects the surface, the laser machining process can result in a rough surface with a remelted layer in the cut region. This layer is due to the solidification of the molten material ejected from the cutting region and the high energy density imposed (MDC, 2011).

Polishing can be used for the removal of burrs and remelted layers produced after laser material removal. Polishing processes can be classified as mechanical, laser, electrolytic and chemical. Electrochemical polishing is an electrochemical treatment method that consists of submerge an electrode into an electrolyte, where different chemical reactions occur. In this process there is a controlled surface dissolution, being considered as a method of surface finishing (BRUNETTE *et al.*, 2001).

Dong *et al.* (2019) investigated the use of electrochemical polishing and engraving on an additive manufactured Ti-6Al-4V alloy, in relation to dimensions and average roughness. The authors observed that the average surface roughness is related to the polishing time and voltage used during the process. Results show lower roughness values with the use of longer time and higher voltages. Another result obtained from the experiment is the so-called edge effect, this effect is due to a rapid dissolution of the metal on sharp edges that causes a variation in roughness along the part. This effect can be minimized through electrochemical pre-engraving on the workpiece.

During the electrochemical polishing process, the use of an electrolyte is necessary. For Titanium and its alloys, the authors Madore *et al.* (1999) claim that the use of sulfuric acid – methanol is viable. The use of this electrolyte was applied by Birch *et al.* (2012), the authors evaluated the chemical and surface topography changes of an electrochemically polished Titanium alloy. For this, chemical and physical characterization techniques were performed to verify the part properties before and after the electrochemical polishing process. As well as Dong *et al.* (2019), the authors found a

relationship between polishing time and surface roughness, where lower values are found at longer polishing times. Furthermore, the electrochemical polishing process did not change the chemical composition of the alloy.

Choungthong *et al.* (2019) removed the remelted layer through an electrochemical polishing process. The titanium alloy (Ti-6Al-4V) surface was previously submitted to a laser-milling process, with a nanosecond pulse, to create a cavity that has been studied. The authors used a solution of sulfuric acid and distilled water as electrolyte, and a stainless steel piece as electrode. The electrochemical process was used to remove the remelted layers, generated from the previous laser processing. The results demonstrate that the polishing process was able to remove the remelted layers. Also deburring and surface finishing through electrochemical polishing can be beneficial.

Another technique of the surface finishing is chemical polishing. The chemical polishing consists of using a corrosive solution where the piece is submerged and a polished surface is obtained. The solution is composed of oxidizing agents, such as nitric acid or hydrogen peroxide, which act on corrosion by replacing, for example, the electrical current required in an electrolytic polishing. The solution varies depending on the piece material (Voort, 2004). For titanium pieces a solution composed by nitric acid (HNO<sub>3</sub>) and hydrofluoric acid (HF) in varied proportions are commonly used, in most cases they present satisfactory results (Balyakin *et al.*, 2019a; Balyakin, 2019b; Łyczkowska *et al.*, 2014).

Bezuidenhout *et al.* (2020), performed the chemical polishing on a sample of titanium alloy material (Ti6Al4V) using an HF-HNO<sub>3</sub> solution, in 1:10 mass percentages (1M), 1:5 (2M) and 1:2.5 (4M). The samples were submerged for a period of 60 min, with breaks between 5, 10 min, 15 min and 30 min. Variations in time and proportion generated different results in relation to material reduction rate and HF consumption in the solution. Higher material removal and HF consumption rates were achieved using the 4M ratio.

Vlcak *et al.* (2020) analyzed the use of several surface polishing treatments on commercially pure titanium grade II, including mechanical, chemical, electrochemical and ion sputter etching. Polishing occurred in a solution composed of 1 ml HF + 2 ml HNO<sub>3</sub> + 100 ml H<sub>2</sub>O. The results showed an irregular surface and grain contours with varied height, which resulted in a high surface roughness in relation to the other techniques. Also, according to the authors, this roughness would be due to the removal process carried out by chemical polishing, in which there is no sensitivity in the crystallographic orientation and in the removal of grains.

Wysocki *et al.* (2019) applied chemical polishing on commercially pure titanium surfaces produced by additive manufacturing. The solution used was composed of HF and HNO<sub>3</sub> in three different proportions. The authors observed that for the best results of chemical polishing occurred a weight reduction of 35 % and 30 % for structures in smaller and larger sizes, respectively. Regardless of the polishing time, the smaller samples suffered greater weight reductions. The authors also observed a relationship between the number of free HF in the solution, which is greater for small samples, and the HF/HNO<sub>3</sub> ratio with sample dissolution.

The workpiece under study consists of a titanium clamp that is used to attach hearing aids and cochlear implants in the middle ear for cases of moderate to severe hearing loss. The system is fixed in the temporal bones and they are subjected to mechanical vibrations, which require a good mechanical and functional coupling (Schraven *et al.*, 2016). Due to the small size of the clamps the laser manufacturing method was selected. In this context, the aim of this work is to characterize the surface quality of laser machined components before and after polishing. Electrochemical and chemical polishing was used in order to remove burrs and remelted layers on a titanium clamp. Results show that after the surface finishing process, there was a considerable workpiece quality improvement.

## 2. MATERIALS AND METHODS

The microparts under study consist of a titanium clamp that is used to attach a sensor to the middle ear in the case of totally implantable hearing devices (hearing aids and cochlear implants). The material of the part is pure Titanium grade II and the geometry of the part is shown in Figure 1. The clamp is 4 mm length, 2 mm width and 0.1 mm thickness. The workpiece was cut previously in a WELLE LASER LS3000 machine. This configuration allows the emission of energy pulses of 0.08 mJ, the pulse frequency was 26 kHz and a pulse time (FWHM) of 10 ns. The power was 90 %, therefore the energy used was 0.072 mJ. The beam diameter at the focal plane was 100 μm. The laser scanning speed on the part was 300 mm/s. Several passes were performed until cutting the workpiece.

Electrochemical polishing was performed using a sulfuric acid solution as the process electrolyte and a stainless-steel piece for the electrode. The clamp and the piece were suspended and immersed in the electrolyte under a voltage of 9 V and a distance between the electrodes of 20 mm. The time used during the process varied between 5, 15 and 23 min.

The chemical polishing method was selected for the removal of burrs and remelted layers of the clamps surface. The solution used to perform the polishing was composed by hydrofluoric acid (HF) and nitric acid (HNO<sub>3</sub>) in a proportion of 30 ml and 70 ml respectively (Bezuidenhout *et al.*, 2020). Two situations were analyzed. The first sample was immersed in the 100 % solution for a period of 1 minute. The second sample was submerged in a diluted solution using 50 % of the previous solution and 50 % of distilled water. The samples were submerged in the solution hanging in a polyethylene and polypropylene wire. The polishing results are presented and compared with the samples before polishing.

Optical and Scanning electron microscopy images were used to characterize samples quality. For that, it was used an optical microscope, model LEICA DM4000/DFC450 with until 3000 x of magnification. Also, a scanning electron microscope, model TESCAN VEGA-3 LMU with voltage of acceleration of 0.5 and 30 kV. The device allows to obtain

images with secondary and backscattered electrons. Figure 2 shows a schematic flow of all the process and characterizations developed in this work.

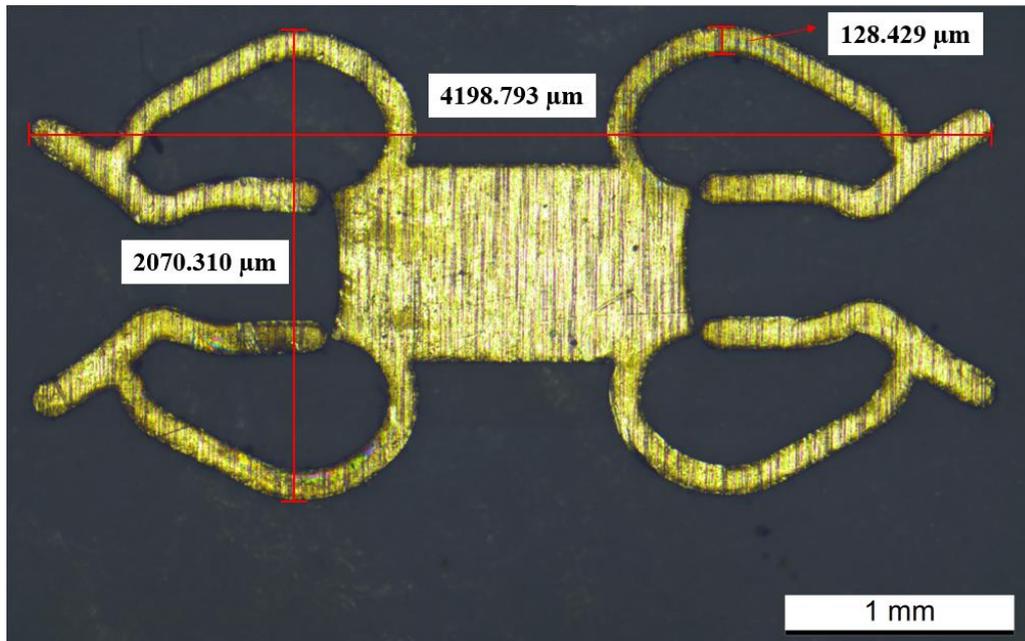


Figure 1. Clamp geometry after laser cutting.

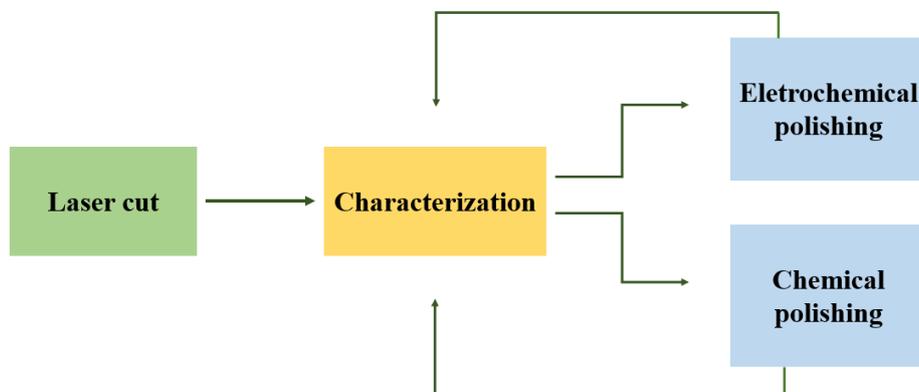


Figure 2. Scheme of the process performed in this work.

### 3. RESULTS

#### 3.1 Laser machined component characterization

In this section, the characterization of the laser machined titanium clamp, obtained from the analysis of the optical microscope and scanning electron microscopy are presented.

Figure 3 shows a picture of the top and bottom of the clamp. It is possible to observe some regions with defects arising from the laser cutting process such as oxidation, incomplete cutting regions and burrs. It can be seen which there are significant quality differences on both sides of the clamp (Bottom, Figure 3.a and Top Figure 3.b).

Figure 4.a and Figure 4.b show the bottom of the clamp surface with magnification of 100 x and 200 x, respectively. The presence of oxidation and some burrs are observable. Lasers with longer pulses (e.g., of the order of nano-milliseconds) remove material at a much higher rate when compared to ultrashort pulses (pico-seconds and femto-seconds), however the material vaporization is accompanied by an excessive amount of heat resulting in component oxidation, melting and micro-cracks.

In the top part of the clamp, represented by Figure 5.a., these characteristics are more accentuated. The top region presented has visually reduced dimensions when compared to the bottom region. The differences are inherent of the laser cutting process. The Gaussian energy distribution of the laser beam produces a V-shaped cavity on the surfaces, decreasing wall-to-wall distance along with cut depth. As the laser beam projects deeper, the total material removed per pass decreases because of the decrease of the substrate-laser interface. Also, defocusing of the laser beam and cut wall shadowing influence on the material removal, Figure 5.b, presents a fold region, this region facilitates the folding of the clamp.

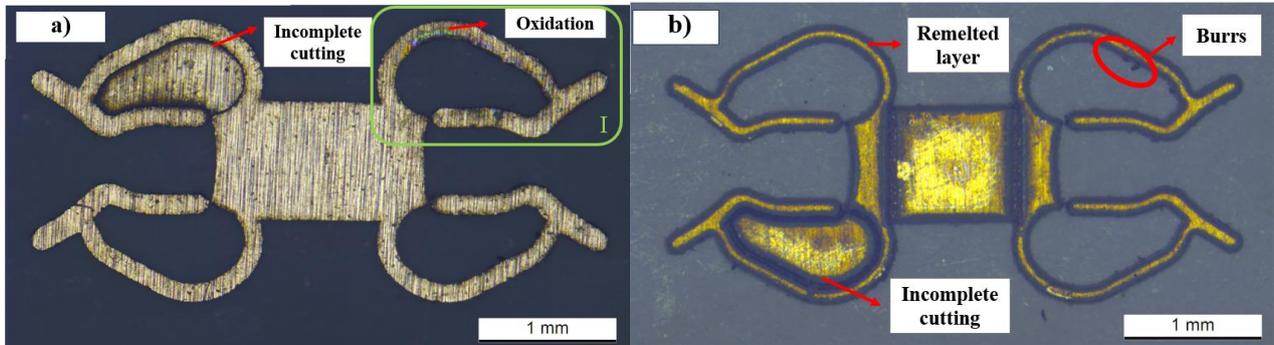


Figure 3. a) Bottom and b) top of the clamp obtained via optical microscope.

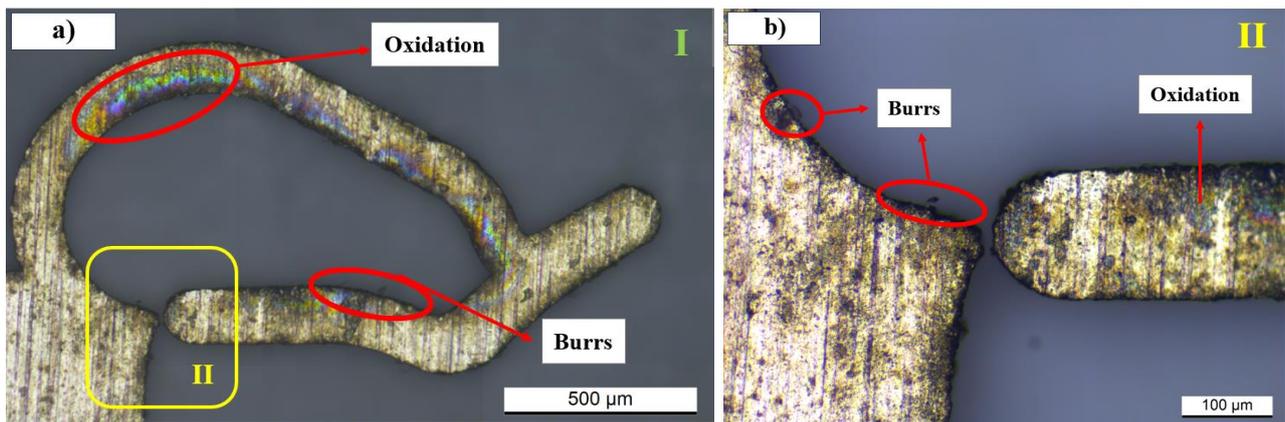


Figure 4. a) Magnification of 100 x of the region I (Figure 3.a) on the bottom of clamp, b) magnification of 200 x of the region II (Figure 4.a) both obtained via optical microscope.

To complement the analysis with the optical microscope, scanning electron microscope (SEM) images were performed. The aim of this procedure is to allow the analysis of some characteristics such as remelted layer, cracks and surface quality. The samples were studied on both sides to observe the variations seen in the optical microscope. Figure 6 shows a large formation of a remelted layer on the cut and fold region. This is the main difference seen in relation to the bottom surface of the clamp, Figure 7, where no remelted material was observed.

Performing a higher magnification of these surfaces, it is evident the great variation between the surfaces, as can be seen in Figure 8 and Figure 9. Figure 8.a shows the width difference between the top and bottom surfaces. The top edge to edge distance appears to be smaller than the bottom edge to edge distance, giving the appearance of a cone. Additionally, to the remelted layer, a small undulation can be perceived on the top view of the clamp near the cutting region, Figure 8b. Undulation could be related to the relative movement between the laser beam and the material surface during material processing.

In Figure 9 the bottom surfaces of the clamp are more homogeneous, without the formation of remelted layers, and with the presence of only a few burrs on the surface.

An image of the fold region is presented in Figure 10.a, Figure 10.b and Figure 10.c with magnifications of 500 x, 1500 x and 3000 x, respectively. This surface presents a non-homogeneous region, characterized by a valley caused by the laser material interaction. On the higher magnifications it can be seen some cracks and molten material over the surface in the forms of spheres. This molten material is caused by laser ablation of the material.

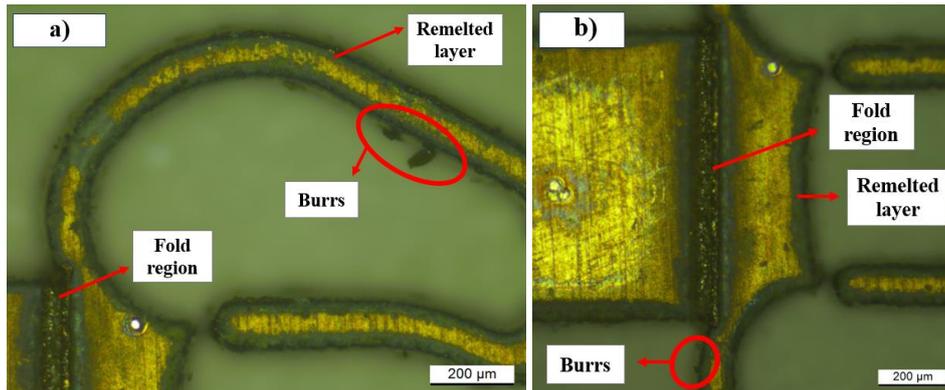


Figure 5. a) 100 x magnification on top region of the extremity of the clamp and b) fold region both obtained via optical microscope.

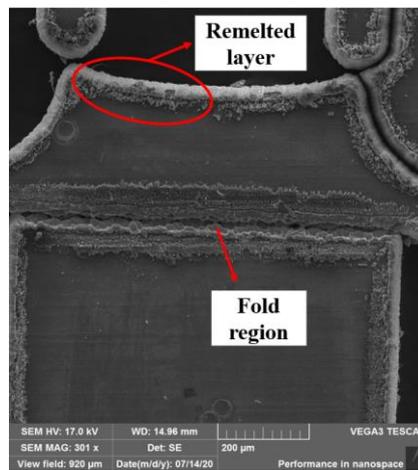


Figure 6. Clamp top in the fold region with 300 x magnification obtained via SEM.

One of the alternatives for improving the laser cutting process would be the adjustment of parameters for the specific situation of plate cutting with pulsed laser, such as pulse time duration, pulse repetition rate, among others. The authors Yu *et al.* (2019) evaluated some laser cutting parameters, such as laser fluence, number of pulses and laser intensity, in a process of drilling a surface formed by a titanium alloy. The results obtained revealed a surface similar to that shown in Figure 10. It is noteworthy that the laser used in this work consists of a nanosecond laser, while the authors Yu *et al.* (2019) use a picosecond laser. Still, the results obtained reveal a similar level of quality. The extremities of the fold region, indicated in the figure as remelted layer, probably occur due to the sample being fused and progressively moved away from the cutting region. Therefore, the removed material was quickly condensed and deposited around it.

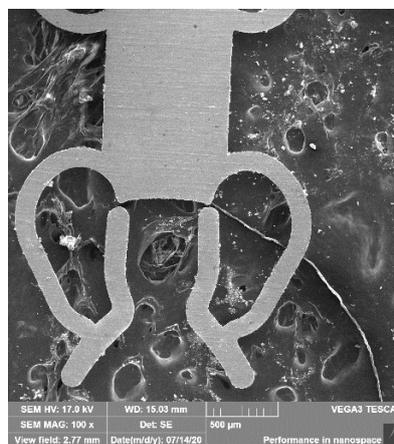


Figure 7. Bottom of clamp with 100 x magnification obtained via SEM.

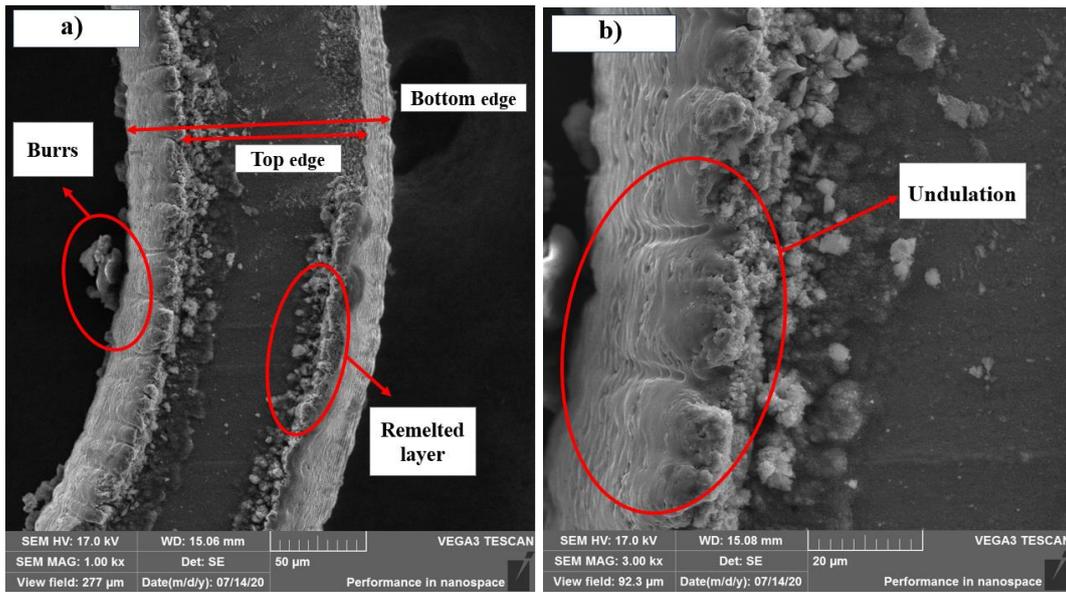


Figure 8. Clamp top with an increase of a) 1000 x and b) 3000 x both obtained via SEM.

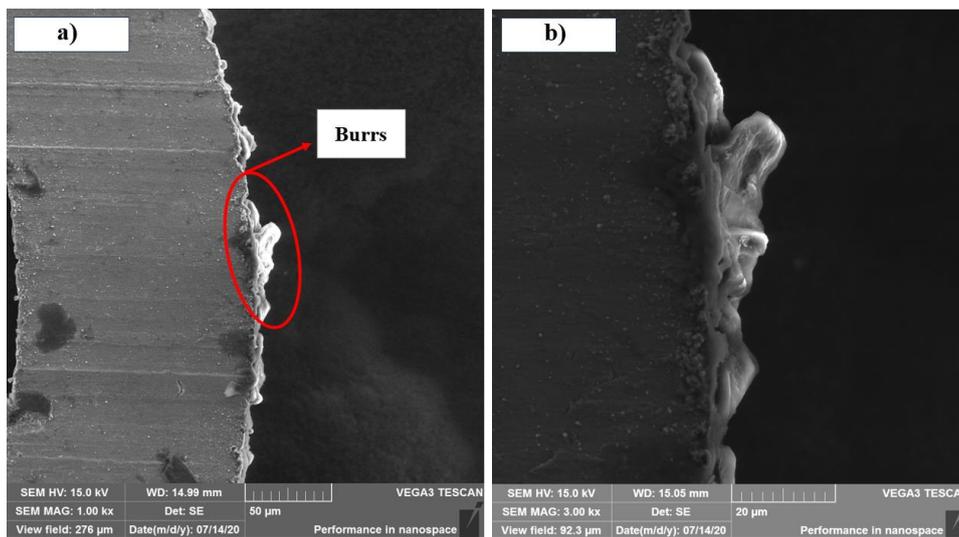


Figure 9. Bottom of clamp with a) 1000 x and b) 3000 x magnification both obtained via SEM.

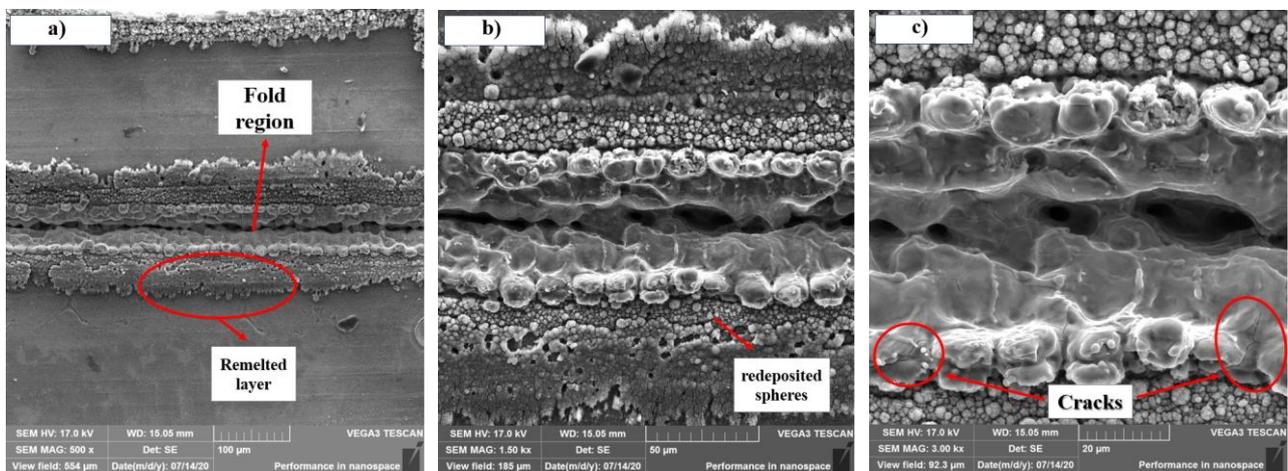


Figure 10. Top part of clamp, in the fold region, with an increase of a) 500 x, b) 1500 x and c) 3000 x all of them obtained via SEM.

### 3.2 Electrochemical polishing

In this section, the results referring to the electrochemical polishing process of the samples are presented. The samples were submitted to the electrolyte solution in different periods of time (5 min, 15 min and 23 min). Figure 11.b shows the polished surface at a time of 5 min along with an unpolished surface, Figure 11.a, for comparison. It can be seen that the oxidation present on the surface previously has been completely removed. However, burrs have not been fully removed.

Figure 12.a and Figure 12.b presents results for times of 15 and 23 min, respectively. Figure 12.a presents a high level of corrosion, represented by “trees format” on the surface image. The surface of Figure 12.b shows burns over the entire surface. Both surfaces effectively removed the burrs on the surface edges. One of the alternatives to improve the application of electropolishing would be the application of a voltage close to or greater than 30 V, as performed by the authors Dong *et al.* (2019) and Choungthong *et al.* (2019). With a higher tension the polish time would be reduced, possibly reducing the damage seen on surfaces with longer polish times.

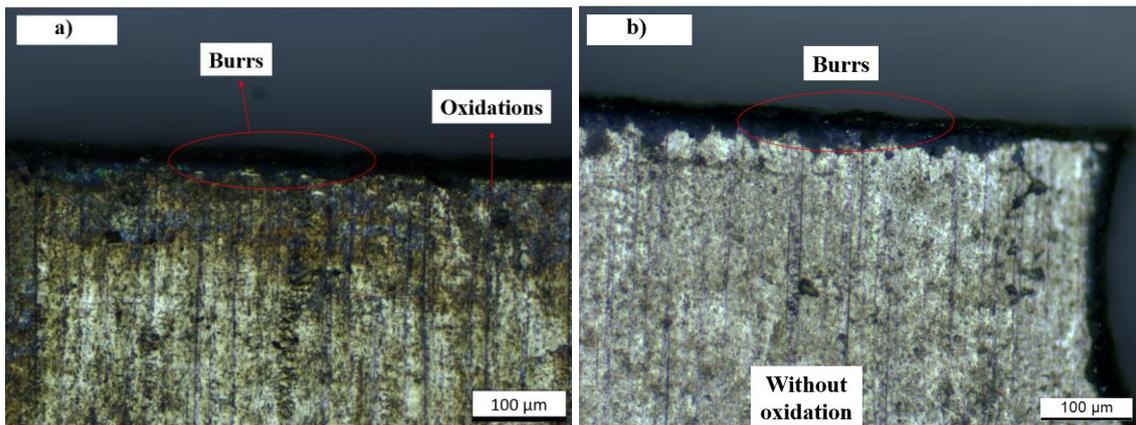


Figure 11. a) Unpolished surface and b) electrochemically polished surface in sulfuric acid solution for 5 min, both with 200 x magnification obtained via optical microscope.

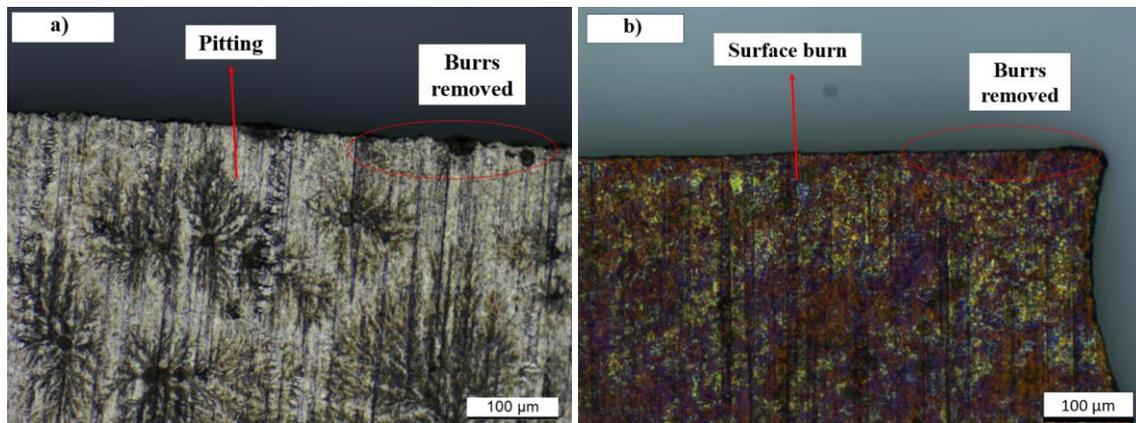


Figure 12. Electrochemically polished surface in sulfuric acid solution for a) 15 min and b) for 23 min, both with 200 x magnification obtained via optical microscope.

### 3.3 Chemical polishing

In this section, the results related to the chemical polishing process of the samples are presented. From the images obtained, it can be seen that the burrs and oxidation reduced considerably after polishing. However, polished samples showed a porous surface that may be the result of the formation of bubbles during the process or a characteristic of the surface that was exposed after polishing. The diluted solution showed a bigger concentration of these pores and less removal of burrs.

The sample submitted to 100 % solution was initially analyzed on both sides. The result indicated to a reduction in the dimensions of the clamp at the bottom and top, in which the latter showed a greater reduction. Figure 13.a and Figure 13.b show the bottom region of the clamp for unpolished and chemically polished cases, respectively. There is a reduction in the dimensions of the edges, reduction of burrs and disappearance of the oxidized region. The Figure 14 show the clamp top region, for unpolished and chemically polished cases, respectively. The same factors occurred, highlighting a

considerable reduction in the remelted layer. In the top part of the clamp, greater reductions in dimensions were observed, as indicated by the measurements shown in the Figure 13 and Figure 14. We can observe that in the edges of the top part reduces about 25 % in relation to the anterior face, while in the bottom part of the clamp, the biggest reduction was about 10 %.

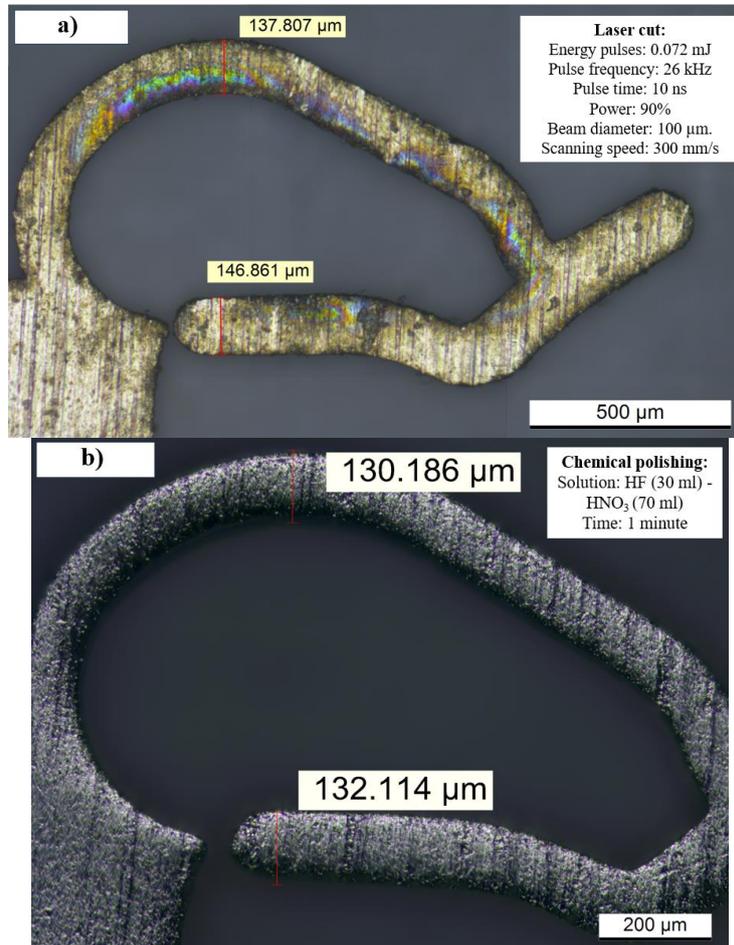


Figure 13. Clamp surface characteristics a) before and b) after chemical polishing HF-HNO<sub>3</sub> (100 %), bottom region with 100 x magnification both obtained via optical microscope.

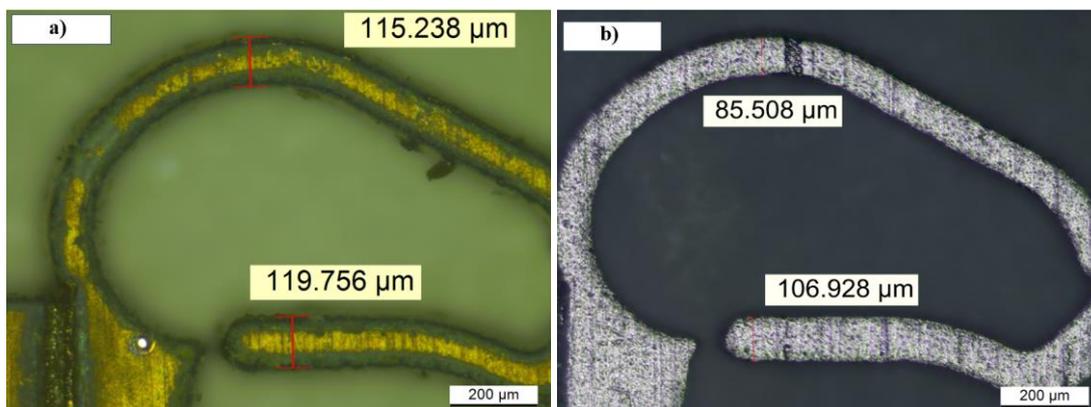


Figure 14. Clamp surface characteristics a) before and b) after chemical polishing HF-HNO<sub>3</sub> (100 %), top region with 100 x magnification both obtained via optical microscope.

The sample submitted to the 50 % solution was also analyzed. As well as for the sample of 100 % reductions of burr, remelted layer, oxidized region and dimensions of the clamp also occurred. Figures 15 and 16 show, respectively, the bottom and top of the clamp chemically polished by 50 % solution. Differently from what happened in the previous case,

the reductions in the dimensions of the edges occurred in close percentages for both regions, with limit values of 19 % reduction in relation to the unpolished surface. Chemical polishing also resulted, for both solutions, in a surface characterized by an excess of porosity, as shown in Figure 17. This result is similar to the study developed by Vlcek *et al.* (2020) and Wysocki *et al.* (2019), as presented above. These authors used different proportions of HF/HNO<sub>3</sub> that could be tested on the surfaces of the clamps to improve the result obtained here.

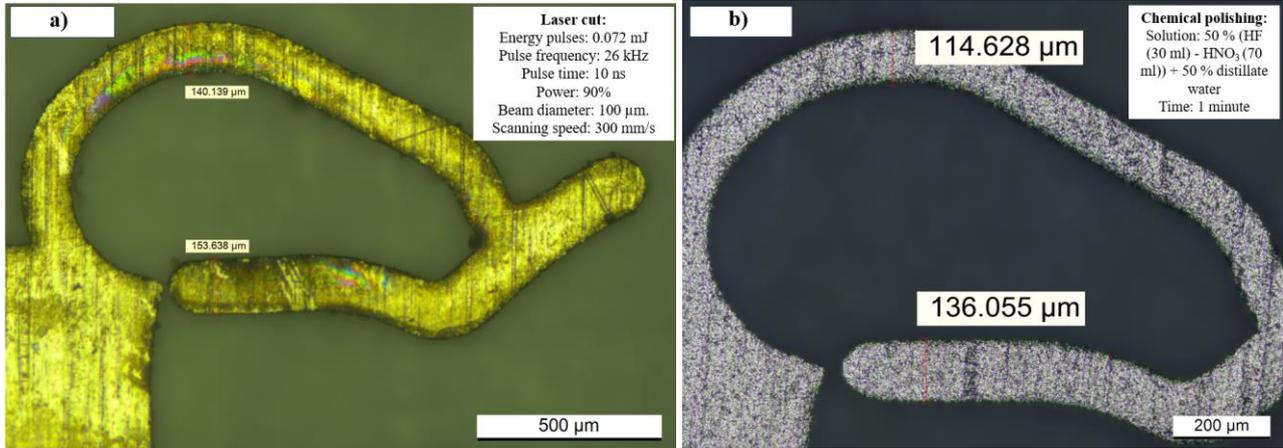


Figure 15. a) Before and b) after chemical polishing HF-HNO<sub>3</sub> (50 %), clamp bottom region with 100 x magnification both obtained via optical microscope.

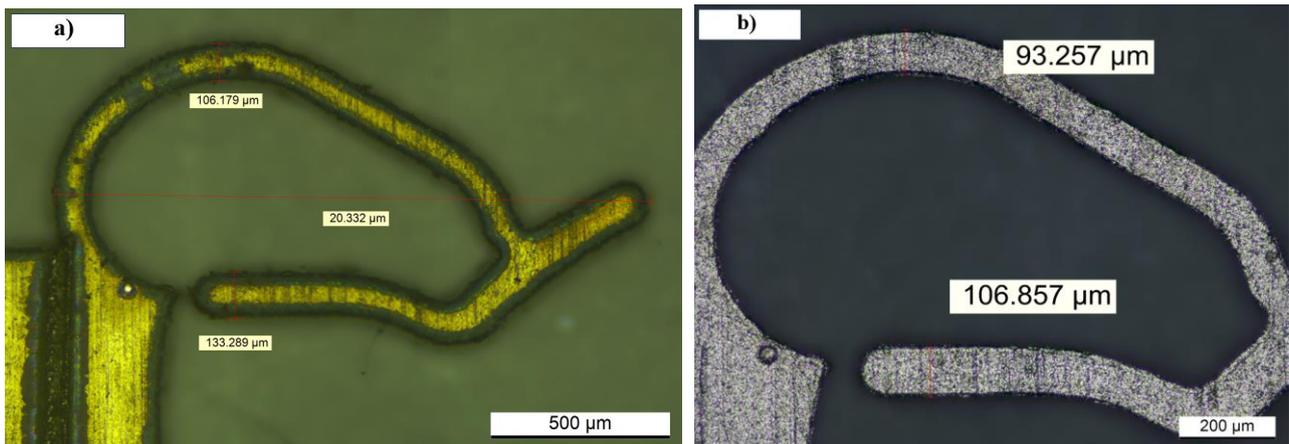


Figure 16. a) Before and b) after chemical polishing HF-HNO<sub>3</sub> (50 %), clamp top region with 100 x magnification both obtained via optical microscope.

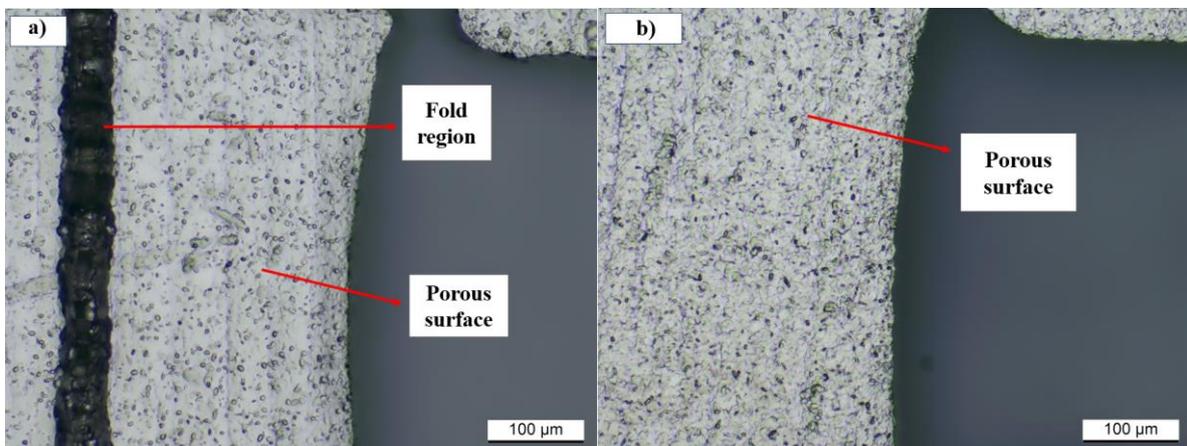


Figure 17. Optical microscope of the porous surface for a) top region with 100 % solution and b) bottom region 50 % solution, respectively, with 200 x magnification.

#### 4. CONCLUSIONS

In this work, surface quality of laser machined titanium clamps were characterized before and after electrochemical and chemical polishing. The characterization revealed burrs on the clamp edges, remelted layers and some regions where the cut did not effectively occur after the laser cutting process. Both polishing processes were performed to remove burrs and remelted layers caused by the laser cutting process. Electrochemical polishing was able to eliminate burrs and remelted layers from the surface, however, the surfaces suffered considerable damage due to the process, such as excessive corrosion and surface burning. Chemical polishing reduces burrs and remelted layers, but results in a porous surface and considerably dimension reduction of the samples. Both polishings were able to improve the surface quality of the clamp. The manufacturing route is promising for fully implantable hearing devices fabrication.

#### 5. ACKNOWLEDGEMENTS

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