

# PHOTOELECTROPOLISHING OF STAINLESS STEEL

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## INTRODUCTION

Herein we present a study on photoelectropolishing of stainless steel sheets. The advantage of this manufacturing process is that smooth surface finish is achieved where removal takes place. The areas being protected against removal by photoresist are not removed. Therefore, structuring takes place. This makes the process one of the electrostructuring processes.<sup>[1]</sup> Electrochemical removal processes are characterised by the combination of electropolishing and structuring by the use of any kind of resist that prevents metal from electrochemical removal. Different from photoetching, spray etching and PCM (photochemical machining),<sup>[2-10]</sup> the removal takes place without build-up of local cells.<sup>[11]</sup> The electrochemical removal process occurs when a current flow is applied to the system by use of an outer power supply. The metal components of the alloy are oxidised at the unprotected areas of the anodic workpiece. In contrast to PCM<sup>[12]</sup> it is quite easy to achieve shiny and smooth surfaces between the embossed structures.<sup>[13-14]</sup> By using different photomasks, different structures can be produced in a flexible and easy way. Using lithographical techniques, the etching stencil is made with the help of the photomask.<sup>[1-4]</sup> After exposure, development and rinsing, the workpiece, now partly coated with the etching stencil, is placed in a special electrolytic solution. Switching on the current starts the anodic removal process. The metal is dissolved at the uncovered areas of the sheet surface. A similar process is used for manufacturing embossing and moulding tools, e.g. for the furniture industry.<sup>[1]</sup> In this case, screen-printing ink is used instead of a photoresist. The disadvantage of screen-printing is the poor accuracy in comparison to photoelectropolishing. First experiments, as the ones

reported herein, confirm the technological feasibility of the manufacturing process. Disadvantageously, the photoresist used is stable no longer than 5 minutes, so that deeper structures cannot be made at the moment.

In a set of experiments we used a photoresist made by HTP HiTech Photopolymere AG. This resist showed its general suitability for this process. It has to be developed further to increase its stability.

Photoelectropolishing can be used to manufacture sheet metal parts and moulds. The workpieces can easily have canal widths of 50-500µm. We think moulds can be used to produce parts for microfluidic applications. With some further development, optical parts may be producible as well. Besides moulds, microfluidic and optical metallic parts will be possible to produce as well. Such sheet metal parts can be assembled like a sandwich as heat exchangers or micromixers. Advantageously, the electropolished canals have a smooth surface: they are not as rough as microfluidic parts produced by etching. These manufacturing methods are described by Crämer et al.<sup>[15, 16]</sup>

Since chemically-resistant stainless steel can be machined, this technique is an alternative to micro spark erosion, laser ablation and similar processes. Compared to electroforming, the manufacturing process described has the advantage that chemically-resistant material can be used, and not only a few metals like nickel, silver and so on, which are not resistant in some cases. Additionally, photopolishing can be applied for producing moulds for plastics like PMMA as well.

In respect to accuracy of the surface finish and the ratio of vertical to lateral removal, further work has to be done. The results we achieved convinced us that this work can be fruitful.

## EXPERIMENTAL SET-UP

### General Process Steps

Figure 1 pictures the production steps of the photoelectropolishing process. The process starts with the production of the phototools. As this is described elsewhere,<sup>[2-5]</sup> it is skipped here. The photoresist has to be applied carefully and the phototools have to be placed and adjusted carefully, because the resolution and the accuracy depend greatly on this step. The workpiece carrying the etching stencil can now be electropolished. The process parameters must be chosen depending on the material of the workpiece and the electrolyte used. In addition, the quality and the structure of the surface has to be taken into account. Important process parameters are, as in electropolishing, electrolyte temperature, polishing time, current density and metal load of the electrolyte.<sup>[1]</sup> If no parameters can be taken from the literature, some tests have to be done.

The photoelectropolished workpiece can be stripped and cleaned thereafter. A passivation step can be necessary in cases where the passivity of the workpiece itself is not high enough. After all this, the workpiece can be used, mounted or whatever.

### About This Set of Experiments

In the frame of this work, we produced channels on the surface of stainless steel sheets (1.4301, X 5 CrNi 18 10 according to German standard). We used an 800mL rectangular glass basin for photoelectropolishing. We filled it with electrolyte and heated the electrolyte basin in a water bath up to  $55\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ . The electrolyte was made of chemicals of analytical quality. It consisted of 45 wt./wt.-% phosphoric acid, 35 wt./wt.-% sulphuric acid, dissolved metal (>30 g/l) and water. This solution is capable of giving a smooth and bright surface finish.<sup>[1, 17]</sup> Similar electrolytes are sold commercially, at least in Germany. Their throwing power and permissible metal content is enhanced by surfactants and complexing agents. As far as we know, optimised electrolytes for photopolishing are not developed at the moment.

### I. Making of Etch Stencil

### II. Photo-Electropolishing

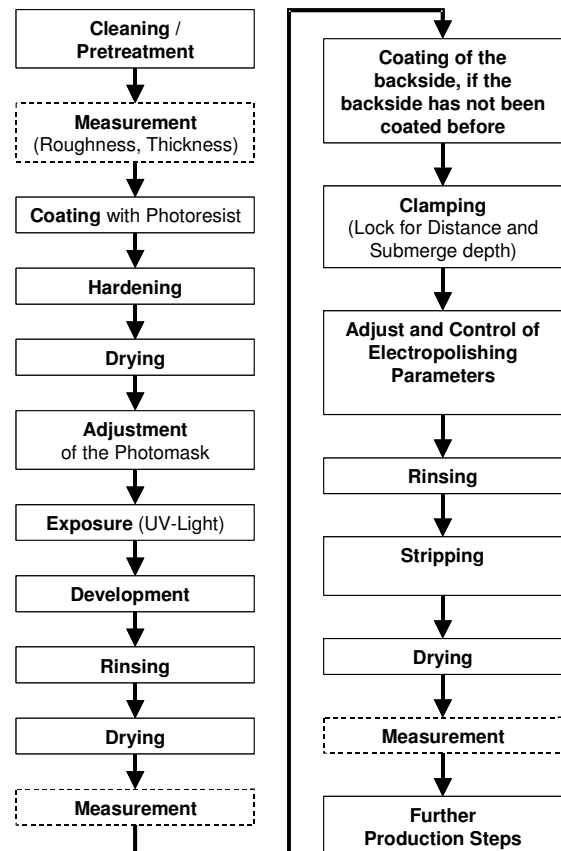


Figure 1: Manufacturing steps of the photoelectropolishing process.

The sheets we used were 4 cm wide. We put them perpendicular and 8 cm deep into the electrolyte solution. Then we connected them with the positive pole of the power supply (EA 4032). For this first set of experiments we photoelectropolished the workpieces for different times at current densities of 5 and 10 A/dm<sup>2</sup>. The uncovered surface area we took into account for all our calculation was about 30 cm<sup>2</sup>.

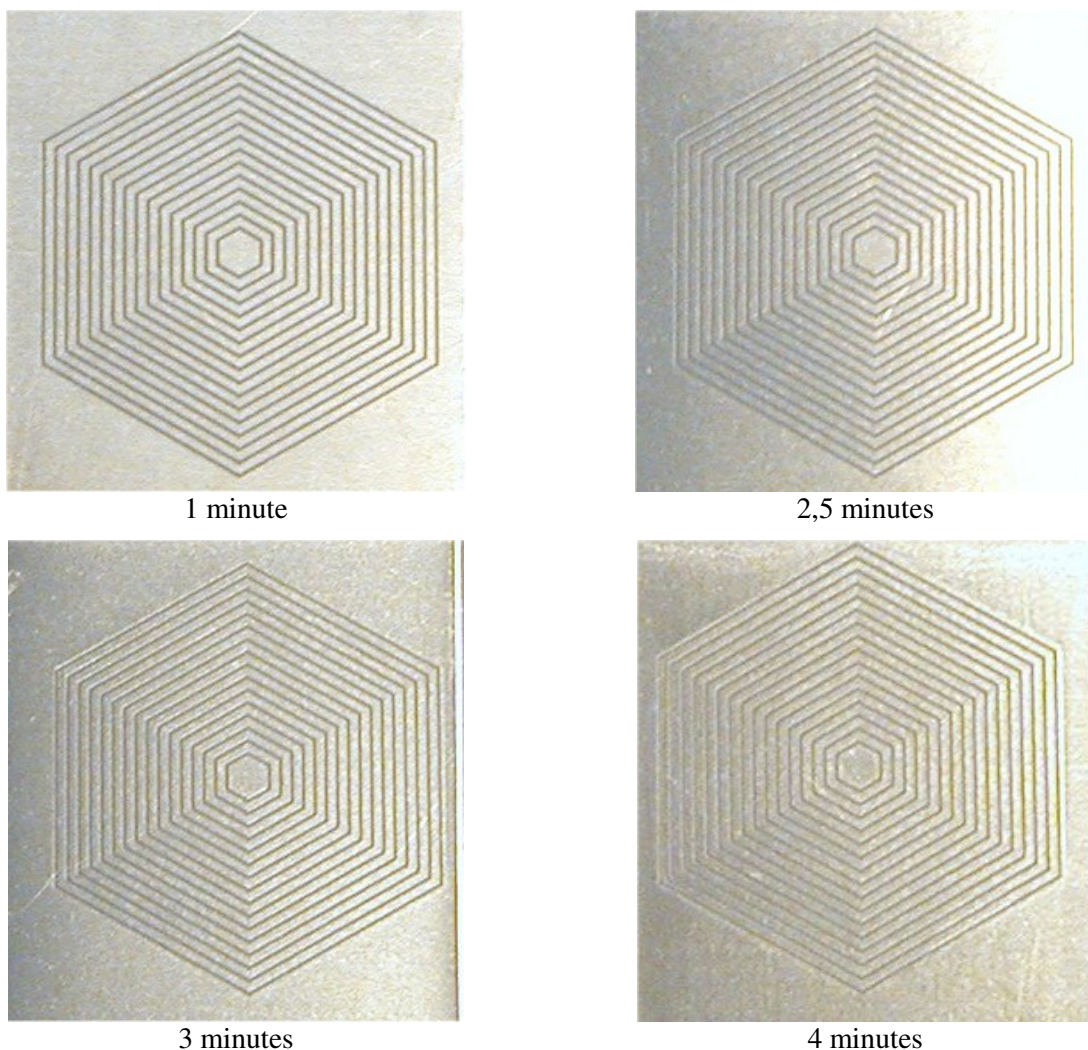
For these experiments we used a negative photoresist from HTP based on epoxy-acrylate. The resist was dip-coated and the resist stencil was made by UV-lithography. The depth of etch was measured at the hexagonal structure. The roughness was measured at the sides of the structure.

After the metal removal, the photoresist was stripped using a 5 wt./wt.-% sodium hydroxide (NaOH) solution. Then the workpieces were stripped in deionised water. We measured roughness and depth of removal with the help of an UBM-3D-Oberflächenprofilometer.

## RESULTS

Figure 2 shows hexagonal structures made by photoelectropolishing. They have been treated for different times (from 1 up to 4 minutes). The width of the structure is 3 cm. Easily, the structure made by photoelectropolishing can be seen. A minimum of the surface roughness

could not be achieved using this short electro-polishing times. Therefore, removal times of more than 15 minutes are needed.<sup>[18, 19]</sup> Up to now, no optimisation of the manufacturing parameters for photoelectropolishing has been made. The electropolished areas are semi-bright. A typical patterning has taken place at these areas. Such patterning can be easily avoided in electropolishing [20, 21]. We assume that the same can be done in photoelectropolishing. Compared to the blank stainless sheets the electropolished areas have been smoothed. In general, electropolished surfaces are more corrosion resistant than non-electropolished ones.<sup>[1]</sup>



**Figure 2: Hexagonal structures made by photoelectropolishing. The stainless steel sheets (1.4301 according to German Standard) have been electropolished using a current density of 10 A/dm<sup>2</sup> with an electrolyte temperature of 55 °C. The polishing times (1 to 4 minutes) are written below the photographs.**

## Removal

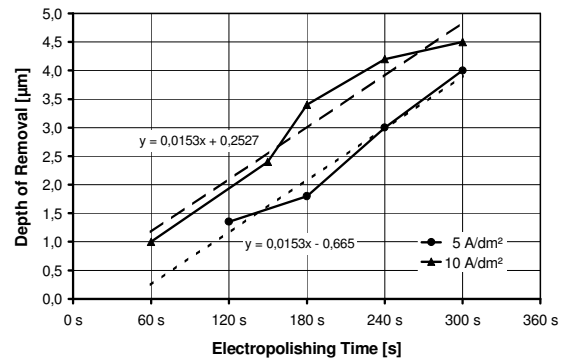
The sheets have been electropolished at two different average current densities (5 and 10 A/dm<sup>2</sup>). We applied different removal times between 60 and 300 s. The removal depth depends linearly on the removal time, at least for the investigated parameters. This dependency is illustrated in Figure 3. After 5 minutes of photoelectropolishing, a removal depth of 4.5 μm in the channels was achieved. The current density was 10 A/dm<sup>2</sup> in this case. As the resist was not formulated for this application, longer removal times and higher current densities could not be realised. In our opinion, an optimisation of the resist for this application is possible. For the photoelectropolishing process, the resist should be stable for a longer time at 50-60°C and average current densities of 30 A/dm<sup>2</sup>. The higher current density is necessary to achieve smoother surfaces.<sup>[19]</sup> At current densities higher than 20 A/dm<sup>2</sup>, the lowest roughness can be expected.<sup>[1]</sup> In addition, the highest brightness can be realised at such current densities. A high gloss value indicates small roughness values at scales below 0,4 μm. This is the lower end of the wavelength of visible light. A high gloss value and a low roughness in the micrometer-scale do normally not happen at the same manufacturing parameters. There is no general correlation between them. On the other hand, they are length-scale dependent.<sup>[19, 22]</sup> However, both effects make it necessary to electropolish for at least 10 to 30 minutes.<sup>[1, 19]</sup>

As the removal was low, we did not examine the etch factor of this set of experiments. The removal speed was 0,7 μm/min using an average current density of 5 A/dm<sup>2</sup> and 1 μm/min at an average current density of 10 A/dm<sup>2</sup>, see Figure 4

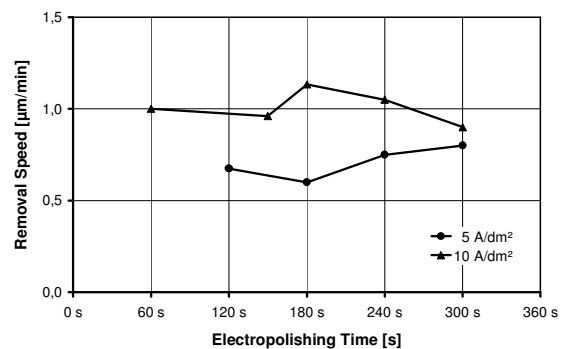
The decrease in the current efficiency is the reason for not doubling the depth of removal by doubling the average current density. With higher current densities, more oxygen is formed and less metal is removed.

## Roughness

Using a current density of 10 A/dm<sup>2</sup>, low roughness can be realised at removal times of



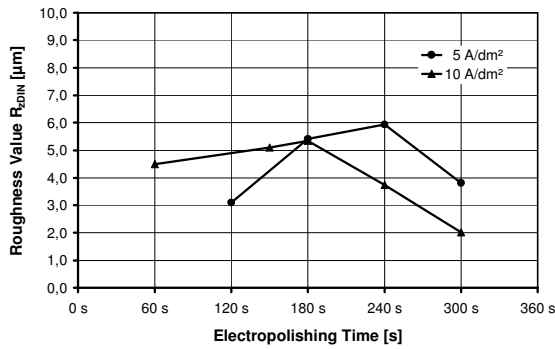
**Figure 3: Linear dependency between electropolishing time and removal depth.**



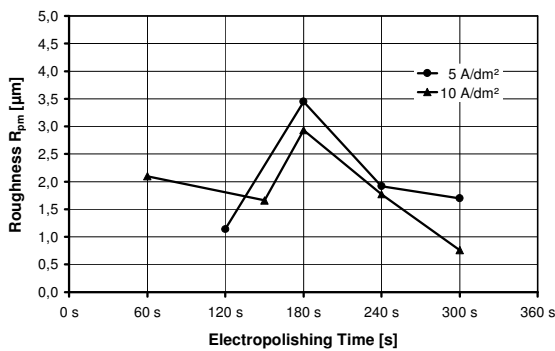
**Figure 4: The removal speed is 0,7 μm/min at an average current density of 5 A/dm<sup>2</sup> and 1 μm/min at 10 A/dm<sup>2</sup>.**

more than 3 minutes (see Figure 5). For both applied current densities in this set of experiments, the highest roughness ( $R_{pm}$ ) was found on the workpieces which had been photoelectropolished for 3 minutes. This is illustrated in Figure 6. In cases where smoother surfaces are needed, longer polishing times are needed.

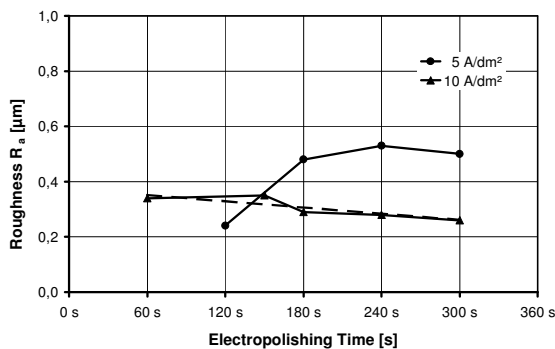
The roughness value ( $R_a$ ) exhibits the same behaviour as the  $R_{zDIN}$  roughness value (see Figure 7). The smallest roughness value  $R_a$  within this set of experiments was achieved after 5 minutes of photoelectropolishing at an average current density of 10 A/dm<sup>2</sup>. The  $R_a$ -value was less than 0,3 μm. As mentioned above, lower roughness values can be achieved at longer electropolishing times with higher current densities. We assume that a further reduction of roughness may be possible if pulse-electropolishing is applied.<sup>[1, 17, 23]</sup>



**Figure 5:** The roughness ( $R_{zDIN}$ ) decreases with electropolishing times longer than 4 minutes in this set of experiments.



**Figure 6:** The lowest roughness value in this set of experiments is achieved after 5 minutes of photoelectropolishing.



**Figure 7:** The roughness value  $R_a$  behaves similar to the  $R_{zDIN}$  roughness value.

## SUMMARY AND CONCLUSION

The advantage of the presented process is that the surface of the removed areas is electropolished. This means that the surfaces are smooth, germs adhere badly and the electro-

polished surface area is more corrosion-resistant. In general, nearly all metals and alloys can be electropolished. Depending on the brightening results, this photoelectropolishing process can be applied on other metals and alloys. Usable electropolishing techniques are known for several types of stainless steel, aluminium, copper, brass<sup>[1]</sup> and titanium.<sup>[24]</sup> The investigations presented show that the photoelectropolishing process works in general. Making the resist more stable will offer the possibility to electropolish longer and at higher current densities. Both are necessary to achieve deeper and smoother structures, such as those needed for microreactors or moulds, for instance. We assume that the HTP-Photoresist will be stable after it is optimised.

In cases where the depth of removal of the photoelectropolishing process is not enough, a combination of spray etching and photoelectropolishing is possible. The high removal will then be achieved by etching, and the smoothness realised by electropolishing thereafter.<sup>[25, 26]</sup>

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