
Localized surface functionalization of steel by jet-plasma electrolytic polishing

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Abstract

Plasma electrolytic Polishing (PeP) is an innovative technique for finish machining of metallic parts even with complicated shapes or with micro surface features. Since the applicability of the immersion-based PeP process is limited by the maximal available energy of the applied process energy source and localized machining of confined areas or features was not feasible up to now, the application of an electrolyte jet was developed. Jet-Plasma electrolytic Polishing (Jet-PeP) not only offers the possibility for controlling the required process energy by an adjustment of the nozzle diameter but also for localized treatments.

The present investigations are focused on the localized surface functionalization of stainless steel AISI 316 L, which is a typical material for medical applications. Here, usually specific requirements on the surface parameters such as a roughness value of R_a less than $0.2 \mu\text{m}$ are necessary, e.g. to avoid biofilm formation. Thus, it will be shown that localized polishing by Jet-PeP is realized at voltages in the range of 300 V to 400 V. The influence of the flow velocity of the electrolyte jet on the resulting total electric current was investigated by varying the volume flow rate of the electrolyte pump between 150 ml/min and 400 ml/min. The analyses of the surface parameters revealed that roughness values of R_z between $0.10 \mu\text{m}$ and $1.03 \mu\text{m}$ were achieved. From the comparison of the cross-sectional profiles of the initial surface and the surface after Jet-PeP, it will be shown that the surface characteristics were changed from a sharp-edged to a rounded surface with less significant peaks. In addition, the wetting characteristics on the polished surface areas were investigated. The results indicate that the surface energy was not increased.

Plasma electrolytic Polishing, PeP, Jet-PeP, localized functionalization, surface energy

1. Introduction

Surfaces, which define functionality, application and life time of parts are characterized through a multitude of distinct properties to suit the requirements in terms of tribological, optical, thermal and other properties. Biocompatibility, bacteria growth inhibition and freedom from adsorbates or particles are often requested in medical applications [1, 2]. High fatigue strength is one main requirement for aerospace and other topology-optimized components. Products can be enhanced by selectively modifying their surface properties.

Known conventional technologies often fail to operate on complex and micro geometries, which are especially generated in precision engineering and in additive manufacturing. Precision parts require the right surface in the right place, meaning that surface functionalities need to be locally set without interfering e.g. with neighboring sharp edges. Many surface treatment processes such as tumbling and electropolishing are applied over the complete part. Local processing is often manual work and very time-consuming. Mechanical polishing or blasting may lead to residual stresses on surface layers. Hence, these processes are not applicable for fragile and micro parts. Furthermore, many existing processes are not resource efficient, which leads to long machining times, the necessity of dangerous electrolytes or the need for post-processing [3]. In addition, small features such as grooves are difficult to access. Jet-Plasma electrolytic Polishing (Jet-PeP) using a material-specific, low concentrated electrolyte thus offers a potential alternative.

2. State of the Art of Jet-PeP

In Plasma electrolytic Polishing (PeP) a setup comparable to electropolishing is used. Material-specific, low concentrated electrolytes with conductivities usually in the range from 80 mS/cm to 120 mS/cm and with temperatures in the range from $75 \text{ }^\circ\text{C}$ to $85 \text{ }^\circ\text{C}$ are applied [3]. High voltages of several hundreds of volts are required for the initialization of the gaseous layer surrounding the workpiece surface. Material removal rates less than $5 \mu\text{m}/\text{min}$ are characteristic for PeP [3, 4]. The PeP process is based on a combination of anodic dissolution, oxide and hydrogen formation, and plasma-chemical reactions [5]. Due to the concentration of the electric flux lines, the removal of near-surface micro peaks is preferred thus offering adequate polishing results [6]. The main restriction of PeP is the polishable component size, which depends on the capability of the process energy source. In addition, complex internal features with high aspect ratio can hardly be addressed due to the concentration of the process on the part's surface according to the distribution of the electric flux lines.

To overcome these limitations, the immersion based PeP process was enhanced by the development of the jet-based process Jet-PeP. Here, the cathode is a nozzle, which is moved over the workpiece in the three-dimensional space, especially for adjusting the distance between the nozzle and the workpiece surface to assure a constant, specific working gap. Figure 1 presents the principle of Jet-PeP.

As in immersion-based PeP, the tool nozzle is connected to the cathodic pole of the process energy source, while the workpiece is connected to the anode. By continuously ejecting electrolyte from the nozzle to the workpiece surface, a continuous free jet

is formed, which is used as electrical connection to the workpiece. Hence, the electrical circuit is closed and electric charge is exchanged between the nozzle and the workpiece.

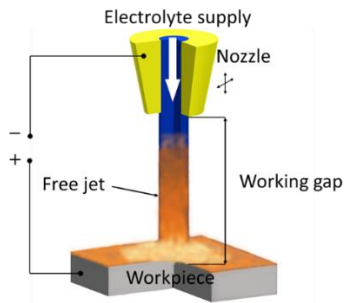


Figure 1. Principle of Jet-PeP [7]

It was shown that voltages higher than 200 V are required for the formation of a gaseous layer, which is necessary for the initiation of the Jet-PeP process [8]. Lower voltages lead to an increasing amount of electrochemical processes, which is not aspired since its results in too high removal rates, which is not applicable for polishing. At voltages higher than 320 V a strong increase of the system resistance due to the expansion of the gaseous layer was observed. An intense glow at the anode was observed, which was caused by electrical discharges and the polishing results were downgraded.

Ablyaz et al. [9] investigated the Jet-PeP process of complex components, which were produced by selective laser melting (SLM). The components were polished with 330 V with an ammonium sulfate based, aqueous electrolyte at electrolyte temperatures between 85 °C and 90 °C. The surface roughness R_a was decreased from 5.6 μm to 1.4 μm . It was proven that polishing of internal surfaces was possible.

Own preliminary experiments in Jet-PeP showed that a minimal voltage of 300 V, a working distance larger than 5 mm and an electrolyte conductivity of 120 mS/cm as well as an electrolyte temperature of 75 °C are applicable for the initialization of the process [7]. It was found that temperatures less than 70 °C lead to a significant increase of the electrochemical removal process.

3. Experimental setup

The design and setup of the components for the experimental investigations are described in [10]. Keysight 34465 A digital multimeters are used to detect the voltage and the current values. A custom control software based on National Instruments LabVIEW was developed to control the peripheral devices and extended to measure the electrolyte temperature via thermocouples.

By using a Keysight N8762 A process energy source, voltages in a range from 300 V to 400 V with an increment of 20 V were applied. The remaining process parameters are charted in Table 1.

Table 1 Process parameters for Jet-PeP experiments

Parameter	Value
Electrolyte temperature	(79 ± 1) °C
pH value	2.12
Electrolyte conductivity	101.7 mS/cm
Nozzle diameter	5 mm
Working gap	6 mm
Volumetric flow rate	(150, 200, 300, 400) ml/min
Polishing Time	1 min

An aqueous solution of ammonium sulfate was used as electrolyte. The electrolyte was delivered from Beckmann-Institut für Technologieentwicklung e.V.

The experiments were carried out on a planar surface of a sample made of AISI 316 L stainless steel. The sample had a size of 48 mm x 48 mm and a height of 9 mm. Before carrying out the experiments, the sample was cleaned with acetone and dried afterwards.

4. Characterization of the surfaces

After the Jet-PeP-Experiments and before carrying out the surface characterization, the sample was cleaned with isopropanol and dried with compressed air. The material composition of the initial surface and the composition after Jet-PeP were measured with a scanning electron microscope (SEM) Zeiss EVO 25 by energy-dispersive X-ray spectroscopy (EDS) for comparative analyses. Likewise, the initial surface roughnesses and the roughnesses resulting from the Jet-PeP were characterized using a Keyence VK-9700 confocal microscope equipped with an objective with a magnification of 50 and used for comparison. For these investigations, each roughness value was measured three times according to DIN EN ISO 4287 and ISO 13565-2.

Contact angle measurements were carried out on the polished surfaces and analyzed with a Dataphysics OCA 200 from DataPhysics Instruments GmbH. From the resulting contact angles, the surface energy σ was calculated according to DIN 55660-2:2011-12 using water, diiodmethan and ethyleneglycol as reference solutions.

5. Results and Discussion

Figure 2 shows SEM images of the initial surface and the polished surface at different positions of the sample, to highlight the modification of micro-scratches resulting from preliminary mechanical preparation methods.

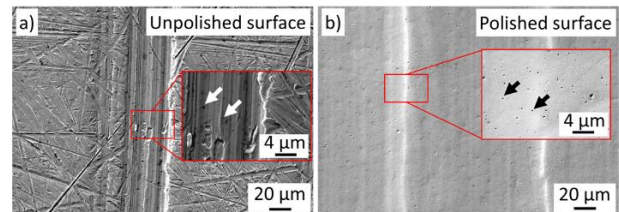


Figure 2. SEM images of the initial surface (a) and the surface polished at a voltage of 320 V and an electrolyte flow rate of 300 ml/min (b)

In comparison to the initial surface shown in Figure 2 a), the polished surface shown in Figure 2 b) appears smoother and micro-scratches are obviously removed. Holes in sub-micrometer scale, which are marked by the white arrows in Figure 2 a), were detected on the initial surface. On the polished surface, the holes are even better cognizable as highlighted by the black arrows in Figure 2 b). According to [5] the PeP process is based on anodic dissolution. It is expected that the holes are caused by higher removal rates of ignoble intermediate constituents of the steel due to locally higher current densities compared to the surrounding, more noble matrix [11].

The results of the EDS measurements of the initial and the polished surface are presented in Table 2. The measurement results of the initial surface show the expected composition for this material according to online available data sheets of material manufacturers. The relative change of single element concentrations are illustrated by the arrows. The comparative values indicate, that polishing of AISI 316 L with Jet-PeP leads to a reduction of the weight content of Cr of -0.09 % and to an increase of the weight content of Fe of +2.1 %, which corresponds to the results in [12].

Table 2 EDS results of the composition for polished surface and change of the content of the element (\uparrow increasing, \downarrow decreasing)

Element in wt.%					
Si	Cr	Mn	Fe	Ni	Mo
Initial surface					
0.55	17.48	1.80	66.57	10.62	2.98
Polished surface					
0.48	17.39	1.41	68.67	9.79	2.26
Change of the content of element					
\downarrow	\downarrow	\downarrow	\uparrow	\downarrow	\downarrow

The remaining elements Si, Mn, Ni and Mo are reduced by less than 1%. Thus, the surface composition of AISI 316 L is slightly influenced by the Jet-PeP process, but chemical residues of the electrolyte were not detected.

Figure 3 presents the average values of the measured total electric currents as function of the applied voltages and electrolyte flow rates.

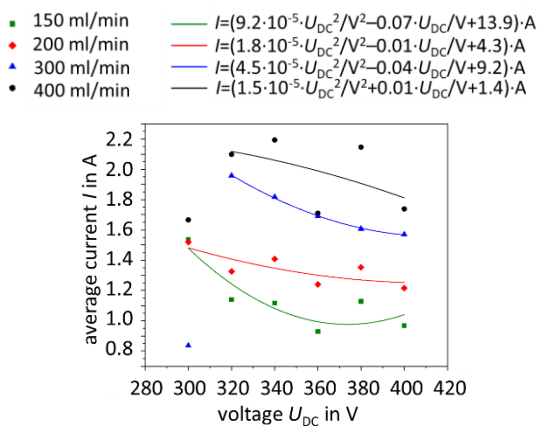


Figure 3. Average electric current as function of the voltage at flow rates increasing from 150 ml/min to 400 ml/min

The points represent the averaged current values over the whole duration of each experiment. Polynomial fits were calculated to highlight the current development at increasing voltages. For the flow rates of 300 ml/min (blue triangles) and 400 ml/min (black circles) the polynomial fits were calculated without considering the values measured at a voltage of 300 V, since they are situated in the region predominated by electrochemical removal. In general, the increase in voltage leads to significantly decreasing trends of the averaged currents as highlighted by the polynomial fits. At a flow rate of 400 ml/min the relatively wide variation indicates a low process stability. An increasing trend of the averaged electrical currents at increasing flow rate was determined as can be seen from the comparative values measured at 150 ml/min (green squares), 200 ml/min (red diamonds) and the higher flow rates. As a reason, it is expected that a higher flow rate causes a higher pressure reducing the thickness of the gaseous layer, which leads to a higher averaged current due to a decrease in ohmic resistance. According to comparative results of the current characteristics in PeP, it can be stated that the Jet-PeP process can reliably be initiated at voltages higher than 300 V and flow rates less than 400 ml/min [13].

In Figure 4 the cross-sectional profile of the initial surface and a comparative profile of the surface polished at a voltage of 360 V and an electrolyte flow rate of 150 ml/min are shown. Sharp-edged peaks and valleys are characteristics of the initial surface, while the polished surface comparatively smooth with rounded edges. The measurements also shows that the kinematic roughness with an amplitude of approximately 2 μm ,

which is a result from the previous mechanical preparation, is retained. The waviness of the kinematic roughness was filtered to exclude influences on the roughness measurements according to DIN EN ISO 4287.

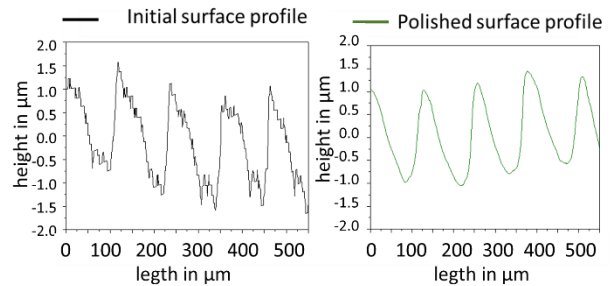


Figure 4. Cross-sectional profiles of the initial surface (black) and the surface polished at 360 V and 150 ml/min (green)

The analysis of the maximum profile peak height R_p and the maximum height of the profile R_z after polishing with varying voltages and electrolyte flow rates are shown in Figure 5.

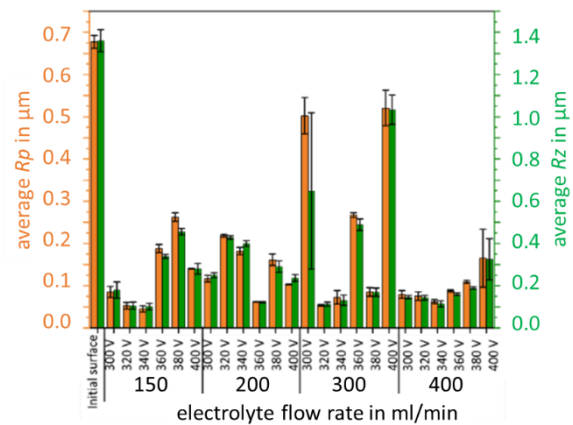


Figure 5. Average value of R_p and R_z depending on the voltage and flow velocity

The green columns show the averaged values of the three measurements and the error bars highlight the respective standard deviations. The results marked by the orange-colored columns indicate that R_p is reduced from an initial value of 0.68 μm to values between 0.05 μm and 0.50 μm , where the minimum was realized at an electrolyte flow rate of 150 ml/min and a voltage of 340 V. Also R_z indicated by the green columns is reduced for all investigated voltages and flow rates from an initial value of 1.36 μm to values between 0.10 μm and 1.03 μm . The lowest R_z was achieved at a voltage of 340 V and a flow rate of 150 ml/min, too. In general, it can be stated that voltages between 320 V and 360 V result in the lowest roughness values for both analyzed parameters R_p and R_z . In contrast, the maximal R_p and R_z were measured at a voltage of 400 V and 300 V each at a flow rate of 300 ml/min. These values can be caused by more instable process at 400 V and electrochemical removal at 300 V.

In addition to R_p and R_z , the core roughness depth R_k , the reduced peak height R_{pk} and the reduced valley depth R_{vk} were analyzed to describe the Abbott-Firestone curve of the surface profiles according to EN ISO 13565-2. Figure 6 a) shows the result for the initial surface and Figure 6 b) the exemplary result for the surface polished at a voltage of 360 V and a flow rate of 200 ml/min. The comparison of the Abbott-Firestone curves indicates a significant reduction of the distribution of the measured values. Hence, the core roughness depth as well as both the peaks and the valleys are significantly reduced.

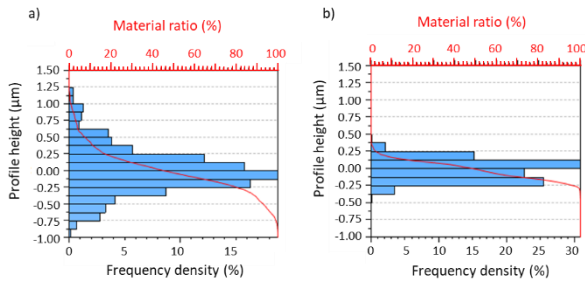


Figure 6. Abbott-Firestone curve of the initial surface (a) and the surface polished at a voltage of 360 V and a flow rate of 200 ml/min (b)

The single values measured after polishing at increasing voltages at a flow rate of 400 ml/min are presented in Figure 7.

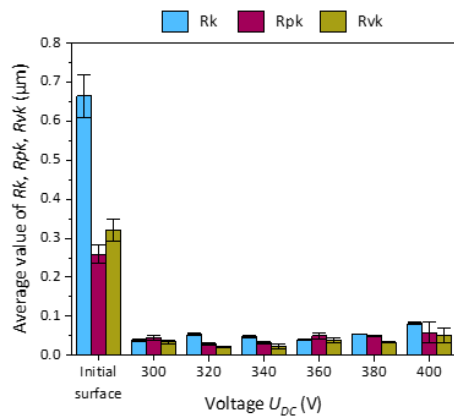


Figure 7. R_k , R_{vk} and R_{pk} of the polished surfaces machined at flow rate of 400 ml/min

The columns represent the average values of the three measurements and the error bars indicate the respective standard deviations. It can be seen that the surface roughness was significantly reduced after all analyzed polishing processes, partially down to less than a tenth of the initial values. The least values were achieved at voltages in the range from 300 V to 380 V. For R_k the minimal realized value is 0.04 µm and for R_{vk} the minimum amounts to 0.02 µm.

For both parameters, certain deviations without clearly cognizable trends were observed in the voltage range from 300 V to 380 V. But for the reduced peak height R_{pk} a decreasing trend from the initial value of 0.26 µm to the lowest value of 0.03 µm at a voltage of 320 V can be recognized. At voltages higher than 320 V, an increasing trend can be derived. At the highest analyzed voltage of 400 V the averages as well as the standard deviations are significantly increased.

Figure 8 shows the surface energy σ of initial surface and after Jet-PeP at 300 V and 400 V as function of the electrolyte flow rate.

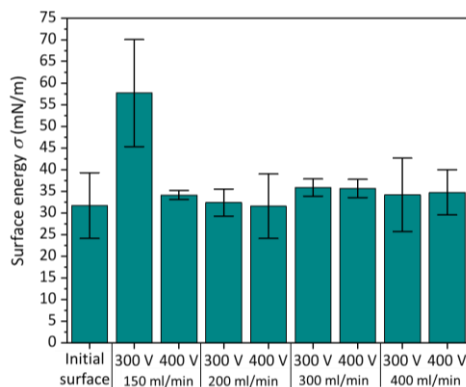


Figure 8. Surface energy σ of initial surface and after Jet-PeP at 300 V and 400 V as function of the electrolyte flow rate

The measured value of the initial surface energy was 31.67 mN/m. As can be seen by the green columns, most of the values measured after Jet-PeP are within the standard deviation, which is represented by the value measured after machining at a voltage of 300 V and an electrolyte flow rate of 150 ml/min. In summary, the surface energy was not systematically increased by the Jet-PeP process but almost kept constant.

6. Conclusion

It was shown that local surface treatment of AISI 316 L stainless steel is possible with Jet-PeP. Comparative SEM images indicated the qualitative smoothing of the workpiece surface. In contrast to the sharp-edged initial profile, the cross-sectional profile after Jet-PeP was characterized by a rounded topography, which was highlighted by the significant reduction of peaks and valleys indicated by the parameters R_{pk} and R_{vk} . Also for the remaining roughness parameters such as R_p , R_z and R_k reductions partially down to less than a tenth of the initial values were determined. The roughness value R_z was reduced from an initial value of 1.36 µm down to 0.10 µm, but the surface energy was not systematically increased by the local treatments. Hence, Jet-PeP offers a potential alternative to conventional processes for localized polishing of micro-structured surfaces without mechanical impact.

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