

ISEM XXI

Jet Application of Plasma Electrolyte Polishing

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Abstract

Plasma electrolytic polishing (PEP) has been gaining increased attention as a replacement for electrochemical polishing (EP) processes. It significantly enhances metal surface properties in terms of surface roughness and corrosion resistance. In contrast to EP, PEP does not require any preliminary workpiece cleaning or treatment with hazardous acids. However, PEP in bath applications yields inhomogeneous polishing results and, in particular, cannot polish more complex geometries such as cavities. To overcome these issues and to enhance the control of local polishing effects on metal workpieces, the use of a PEP jet is investigated. A jet configuration enables higher PEP control and increases PEP polishing rates by a factor of six compared to a polishing bath as a result of higher localized current.

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1. Introduction

For the general PEP process, the workpiece is anodically polarized and immersed in a bath containing an aqueous electrolyte solution [1]. The choice of aqueous electrolytes depends on the material to be polished [2] and in contrast to classical EP, the electrolytes used for PEP are typically less toxic and hazardous. For example, a suitable PEP electrolyte for stainless steel is aqueous sodium carbonate or aqueous ammonium sulphate [3].

The cathodic polarized tank has a significantly larger surface area [4] than the anodic workpiece. If a DC voltage is applied between the tank and workpiece, the current density is significantly higher on the workpiece than the rest of the circuit. Joule heating, concentrated in the electrolyte at the workpiece surface, leads to the formation of a vapor gaseous envelope (VGE). This VGE increases the electrical resistance dramatically [5]. By providing a sufficiently high supply voltage, the electrical current can be maintained by plasma ignition within the VGE, leading to PEP at the surface. The PEP process involves several physical and electrochemical processes. The relative importance of the different processes to the overall removal rate is still an active area of research. Electrochemical dissolution appears to play a significant role since the electrolyte touches the anodic surface locally, reducing the local PEP whenever the plasma and its VGE breaks down [5],[6]. Processes directly related to the plasma also participate in polishing the surface, such as anions that are accelerated within the plasma towards the metal surface, giving rise to a visible glow [3]. These anions, impacting the metal surface, can cause ejection of surface atoms from the workpiece, i.e. sputtering. The ejected material is transferred to the VGE and subsequently to the electrolyte [3]. The removed material is partially ionized and participates in maintaining the discharge current. The surface roughness of the workpiece in contact with the plasma sheath leads to a strongly inhomogeneous electric field distribution and consequently strong discharge current density variations. The electric field and current density are high at the peaks of the micro protrusions, which causes them to be eroded at a higher rate [7]. The more intense dissolution of the surface peaks leads to a decrease in surface roughness.

Although the classical bath PEP process shows various advantages [2], [3], there are some issues inherent to this configuration. For example, the total current is directly proportional to the overall surface area in contact with the electrolyte, increasing the power supply costs required to polish

large parts. In addition, the surface polishing effect is not homogeneous over the entire surface and, in particular, significantly reduced in cavities. Submerged samples indicate a more intense polishing rate in the deeper regions than in the upper regions closer to the electrolyte surface.

An electrolyte jet is proposed to overcome PEP action inhomogeneity and enhance the control of local polishing effects on metal workpieces. As with atmospheric plasma applications [8], the benefit of a jet is the freedom of localized sample treatment [9], e.g. with different process polishing rates and thus different resulting surface roughness or gloss effects on the same metal workpiece.

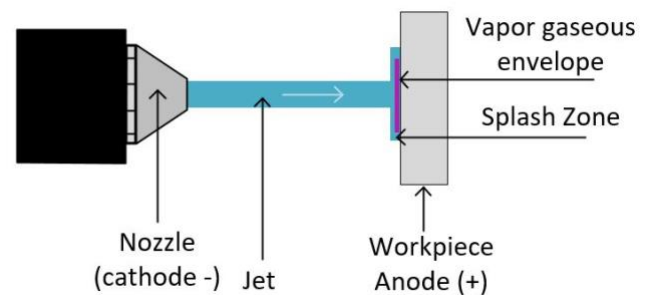


Fig. 1: Schematic of PEP in a jet process, where the negatively charged electrolyte impinges onto the anodic metal surface of the workpiece. The gaseous vapour layer establishes itself on the metal surface before the plasma is formed. Polishing occurs at the impinging zone's plasma-metal interface, enabling homogenous localized polishing.

2. Experimental Setup

2.1. Material and Pretreatment

The workpiece material used in this work is stainless steel 316 (X5CrNiMo17-12-2). This steel is known for its wide range of applications due to superior corrosion resistance, including in medical and chemical industries. The stainless austenitic chromium-nickel-molybdenum alloy consists of 17% chromium, 12% nickel and 2% molybdenum. The steel sheet is laser cut into 30 x 30 mm² coupons. In order to achieve a uniform initial roughness for the experiments, all specimens are sandblasted. The initial roughness is measured with a confocal microscope yielding a Sa of 2.1 μm. Each sample is cleaned in an ultrasonic isopropanol bath for 5 minutes prior to polishing to ensure sample consistency. The samples are weighed before and after the PEP treatment to determine the removed mass by dividing the removed mass over the processing time.

2.2. PEP-Bath Application

PEP was carried out in a classical immersion configuration with the same electrolyte composed of 10% ammonium sulphate and deionized water to compare with the PEP jet results. The liquid in the bath was heated to a temperature of 80°C. The applied voltage between the anodic workpiece and the cathode is 350V, where the plasma ignites easily, and the polishing action is stable within the electrolyte.

2.3. PEP-Jet Application

The PEP jet system used in this work is illustrated in Fig. 1, where a metallic nozzle acts as the grounded cathode, and the workpiece acts as the anode. In order to complete the circuit, the DC must travel through the electrolytic jet. The kinetic nature of the jet produces a higher electrical resistance than a static electrolyte bath, necessitating an increase in the electrolyte conductivity, which is easily achieved by increasing the amount of ammonium-sulphate in the electrolyte. For this reason, a 10 wt.% ammonium sulphate solution was used for the PEP jet experiments, which is slightly higher than in most of the reported PEP bath applications for stainless steel 316 [3].

The setup contains a pump to create a constant volumetric flow of about $1.7 \cdot 10^{-6} \text{ m}^3/\text{s}$. The flow is measured with a magnetic-inductive flowmeter and smoothed by a pulsation dampener. The electrolyte enters the cycle preheated and is observed by a pH- and a conductivity- sensor. The pH – and conductivity levels are at 3.6 and 240 mS/cm, respectively. The electrolyte temperature at the nozzle outlet, which matters for the PEP-process, is kept constant at 70° C via the heating system. The diameter of the nozzle used throughout the experiments has an inner diameter of 2 mm. The electrolyte jet impinges the workpiece horizontally, thereby closing the electric circuit. Electrolyte jets are typically positioned vertically in the literature, resulting in a large splash zone [3]. The splash zone size can be reduced by orienting the jet horizontally, resulting in more concentrated polishing with higher local removal rates. After establishing a constant laminar electrolytic flow, the plasma is ignited by applying 350V DC to the workpiece, as shown in Fig. 2. The total current in the electrolytic jet is about 300 – 500 mA, depending on the fluctuating size of the splash zone, which is polished as well.

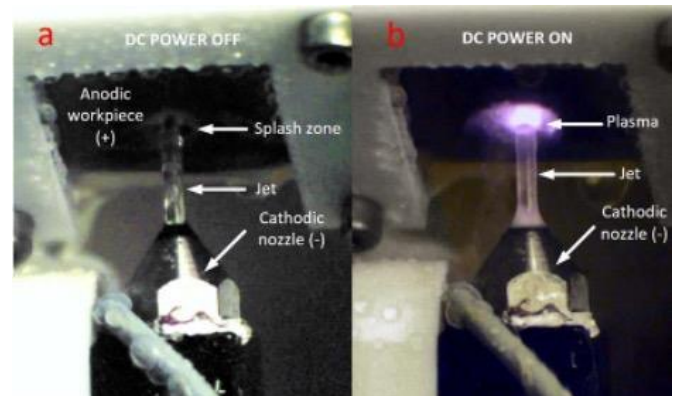


Fig. 2: a) Electrolyte jet impinging on a workpiece without applying voltage. A U-shaped PTFE holder maintains the 316 stainless steel sample in place. b.) PEP-Jet setup with 350V applied between the cathodic nozzle and the anodic workpiece, where the plasma ignites. Polishing occurs at the plasma-workpiece interface.

3. Results and Discussion

3.1. PEP-Bath

In the regular PEP application, where workpieces under high voltage are immersed in an electrolyte bath, the VGE is disturbed by steam created at lower locations on the workpiece, pushing the envelope away from the metal surface. This phenomenon can be seen in Figs. 3 and 4, where a half-immersed stainless steel 316 workpiece is shown.

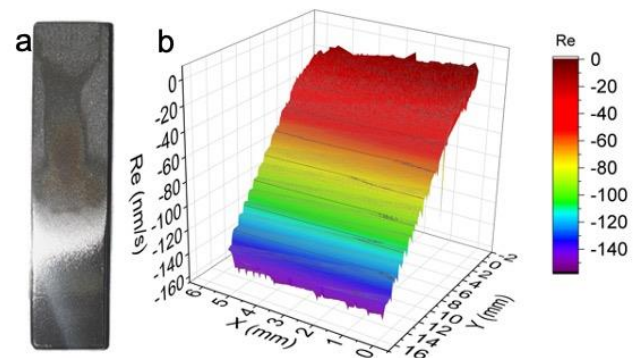


Fig. 3: a) Image of a PEP polished stainless steel 316 sample that was partially submerged in an ammonium-sulphate salt electrolyte bath under 350V for 15 min. The sample is sized 5.5 x 30 mm². The different polishing zones along the immersion depth can be seen. b) Confocal microscope analysis of the partially submerged stainless steel 316 sample showing the depth dependence of the polishing rate, with the highest polishing rates occurring at the bottom of the bath.

The rising steam disturbs the VGE and perturbs the plasma, resulting in a decreased surface polishing effect. The steam bubbles introduce further irregularities in the surface smoothening process, leading to higher surface variations and inhomogeneities.

The limitations of PEP bath-polishing lay in processing big workpieces [10] and in zones where high rates of steam formation have a detrimental effect on the process. Furthermore, deep cavities and the inner surfaces of tubes cannot be polished. Limitations of size for workpieces are due to power consumption. The average current density measured in the performed PEP bath experiments is around 3 mA/mm^2 , agreeing with the literature on stainless steel 316 [3]. The power supply used in our experiments has a power limit of 1.2 kW and, together with the 350 VDC process voltage, limits the surface size that can be polished.

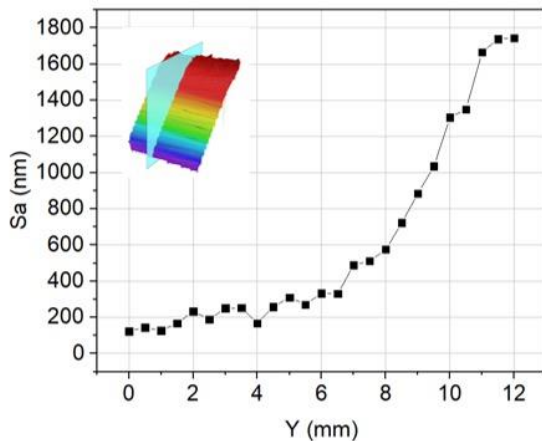


Fig. 4: Cross-section of Fig. 3, highlighting the inhomogeneous roughness of the stainless steel 316 sample along the immersion direction.

3.2. PEP-Jet

The effect of the PEP-jet polishing on the stainless steel 316 sample is shown in Fig. 5. A clear and high polishing effect is seen in the centre of the coupon. The lowest resistance drop from cathode to anode lies in this central area, and thus the plasma polishing is most active here. The polishing rates reach around 200 nm/s , roughly an order of magnitude higher than the polishing effects seen in PEP bath applications as illustrated in Fig. 4 with around 30 nm/s . Fig. 5 a) shows the actual sample, with an area of $30 \times 30 \text{ mm}^2$, after a treatment of 5 minutes. The middle spot is polished to a high gloss level, and the achieved surface roughness S_a is $< 0.2 \mu\text{m}$. This middle spot is roughly the diameter of the 2 mm electrolyte jet. A splash zone approximately 6 mm in diameter is apparent around the high gloss polished centre, where PEP action still occurs. However, this anodic surface area is polished at a lower rate, as seen in the cross-section of the polished surface in Fig. 5 b). The PEP action diminishes rapidly towards the outer edge of the polishing circle due to a rapidly increasing electric resistance caused by the thinning electrolyte film. After this enlarged circular 6 mm PEP-active splashing zone, other processes are present in this extension that need further investigation. The polishing rate and the smoothening rate are falling quickly into a single-digit nm/s polishing rate and are thus neglectable from a polishing point of view. The high polishing zone in the centre and the quickly decreasing

polishing rate in the splash zone enable a PEP-jet that can polish large areas with locally controllable polishing rates.

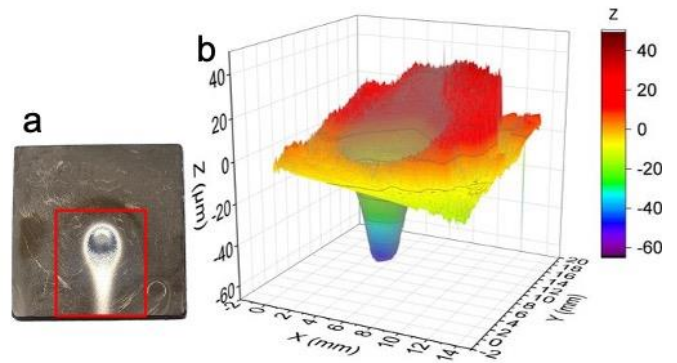


Fig. 5: a) A stainless steel 316 anodic workpiece after being processed by a stationary PEP-jet at 350V for 5 min and 0.1 L/min constant electrolytic flow. b) A confocal microscope analysis illustrates the measured surface profile that reaches depths up to about $60 \mu\text{m}$ at the centre of the electrolytic jet.

3.3. PEP-Jet versus PEP-Bath

Besides the homogenous polishing of the electrolyte jet, the plasma polishing action is considerably higher in the jet than in the bath. The elevated plasma polishing effect is reflected by the higher polishing rate of the PEP-jet than in the PEP-bath of 200 nm/s compared to 33 nm/s , respectively. Each sample is treated either in a PEP-bath or locally by a PEP-jet, while the surface roughness is measured where the highest polishing rate for different processing times appears.

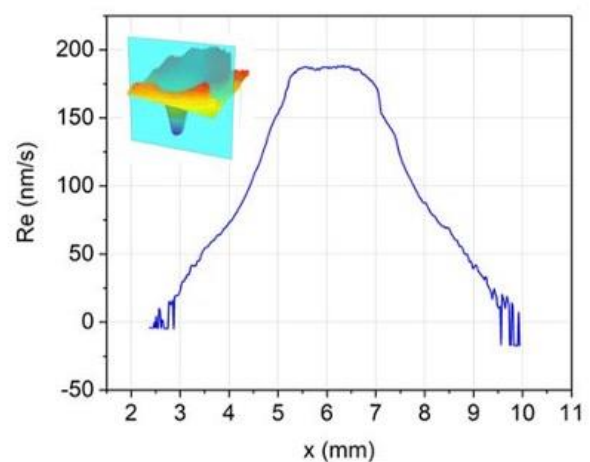


Fig. 6: Illustration of the polishing rate from a cross-section of the polished sample from Fig. 5. The polishing rate is the highest in the middle with almost 200 nm/s , where the 2 mm electrolytic jet hits the anodic workpiece and falls rapidly off in the splash zone of the electrolytic water. This splash zone is a circle around the impact spot of roughly three times the diameter of the electrolyte jet.

The surface roughness is reduced by roughly one order of magnitude from $S_a = 2.1 \mu\text{m}$ to $S_a < 0.2 \mu\text{m}$ after either 15min in the PEP-bath or after 3min with the PEP-jet. The consequent surface smoothening by the jet, with the same voltage of 350 V applied between the cathode and the anodic workpiece, is about five times faster than in the bath.

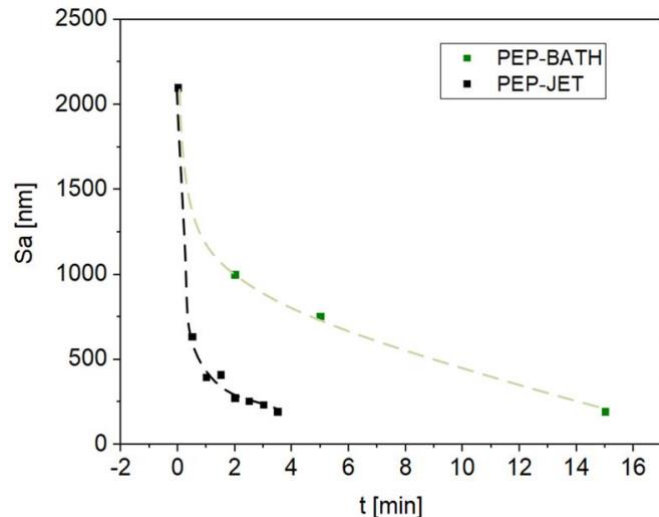


Fig. 7: Roughness results on two identical stainless steel 316 samples with a homogenous starting roughness of $S_a = 2.1 \mu\text{m}$. The two fitted lines are only to aid visualization. The upper (grey) line represents the surface roughness evolution of the sample submerged in an electrolytic PEP-bath at 80°C and 350V, while the lower (black) line represents the roughness evolution of the same sample in the centre of an electrolytic PEP-Jet at 70°C and 350V. One order of magnitude smoother surface roughness $S_a < 0.2 \mu\text{m}$ is reached after 15min in the PEP-bath, while the PEP-jet requires a bit more than 3min, reflecting the higher PEP action in the jet. The surface roughness of the PEP jet centre spot is compared to the lowest surface roughness found on the PEP bath processed sample.

4. Conclusions & Outlook

The physical electrolytic flow enhances the ionic ammonium salt flow. Hence it directs the current towards the plasma zone, such that the plasma electrolytic polishing is more active in the anodic surface area. In the impact zone of the 2 mm diameter wide electrolyte jet with a laminar flow of $1.7 \times 10^{-6} \text{ m}^3/\text{s}$, the electrolytic flow leads to about six times higher polishing rates at 350V. Thus, a quicker smoothening rate in the jet's impact zone results of about five times at 350V in terms of surface roughness. A surface roughness below $S_a < 0.2 \mu\text{m}$ has been reached after only 3 min with the PEP-jet, which is an order of magnitude lower than the initial surface roughness of $S_a = 2.1 \mu\text{m}$ of the stainless steel 316 samples. High polishing rates are generated within the impact zone where the electrolyte jet meets the anode. A quickly diminishing polishing rate is established when the water drains off the stainless steel sample away from the jet impact zone. In this splashing zone of the jet, plasma polishing is still apparent but with diminishing polishing action because of the decreasing current due to the higher electric resistance of the splashing zone.

The localized polishing area results in constant power consumption and thus enables scanning of large surfaces with the given circle-like polishing pattern. Different final surface roughness of the same sample and thus well-controlled polishing can be achieved. This lends itself to industrial application of the PEP jet where varying local polishing or surface roughness is required.

The splash zone around the main polished area is divided into two parts, one where plasma still contributes to the polishing process and one where no plasma is present. The zone with no plasma is considered to be dominated by soft electrochemical polishing, but this requires further investigation.

Acknowledgements

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