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Electrochemical machining of pre-defined kinematic surface roughness on nickel 201 with continuous electrolyte jet

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Abstract
Nickel and its alloys are widely used in microsystems due to their high strength, ductility, and resistance to corrosion and fatigue. As performance demands grow, there is an increasing interest in developing methods for producing high-quality pure-nickel micro-components. However, nickel and its alloys are considered as difficult-to-machine materials due to their mechanical and chemical properties such as high tendency to strong work hardening. Thus, individual surface structures such as micro-fluidic channels or structured surfaces to increase the size of the active area for chemical reactions and to improve performance and efficiency is challenging. Alternative thermal ablation techniques cause microstructural changes of the base material and strong surface oxidation, which should usually be avoided. For these reasons, electrochemical machining (ECM) for surface shaping was investigated. Applying a continuous jet of electrolyte (Jet-ECM) offers the possibility for flexible shaping of metal surfaces with micrometre-scaled grooves that are characterized by a specific, kinematic surface roughness. However, the dissolution characteristics of pure nickel with Jet-ECM was not sufficiently investigated before. Hence, applicable combinations of electrolyte composition and machining strategy were investigated to prepare kinematic surface roughness values. Concentric arrangements of cylindrical groove sections with lateral infeed distance of 150 µm were manufactured on initially plane, mechanically turned surfaces of nickel 201 parts. The most efficient dissolution was realized with a sodium chloride electrolyte. From further experiments, nozzle motion speeds of 900 µm/s, 1800 µm/s and 3000 µm/s were derived to machine whole cylindrical sample surfaces. The measurements prove good accordance with the highest target roughness of *Rz* 27 µm, while the lower roughness values significantly differ from the targeted *Rz* of 20 µm and 15 µm. Hence, Jet-ECM represents a promising technique for structuring of pure nickel to increase surface area, but further optimizations are required to better meet the targeted roughness.

Keywords: electrochemical machining, Jet-ECM, kinematic surface roughness, nickel

1. Introduction

Nickel and nickel alloys exhibit good chemical resistance in many media [1]. Furthermore, they exhibit mechanical and chemical material properties that qualify them for applications in both high- and low-temperature ranges. Numerous applications in macro- and micro-apparatus construction and process engineering, such as fuel cells, are conceivable [2].

Non-alloyed, pure nickel 201, which is optimized for applications at temperatures above 300°C, is often used as electrode material for hydrogen electrolysis or in alkaline fuel cells [3]. To increase efficiency, an increase in the active reaction surface is sometimes sought [4]. This can be achieved by deliberately creating a kinematic roughness. Nickel, however, is difficult to machine with cutting processes, due to its high ductility, strong tendency to work hardening and chemical affinity to various cutting materials [5].

Finish machining processes, which are often used to reduce surface roughness, can be also used for targeted surface modification using micro-tools and sufficiently deep penetration depths. However, these often require very high machining forces, which can lead to excessive loads and initiate surface damage due to excessive compressive residual stresses [6].

Thermal machining processes such as laser beam machining and spark erosion cause surficial oxide formation, since nickel tends to form oxide layers under heat impact [7]. In addition, the heat impact might also cause changes of the microscopic grain structure, which can have negative influences on the material's mechanical properties.

Electrochemical machining (ECM) offers material dissolution without mechanical and thermal impacts on the material's microstructure. Using a continuous electrolyte jet (Jet-ECM) offers confined machining with high flexibility. With kinematics comparable to mechanical milling, micro-grooves can be arranged on the part's surface. While dissolution characteristics of pulsed-electrochemical sinking [8,9,10] and wire-electrochemical machining [11] of pure nickel were recently investigated, Jet-ECM of the same material is hardly reported and mainly known for machining holes and through-slots. Others focus on Jet-ECM of nickel alloys instead of pure nickel [12].

The lateral expansion of single removals machined with Jet-ECM is predefined by the nozzle's diameter and vertical expansions can be adjusted by processing parameters, which can be chosen individually [13]. However, the Jet-ECM technique to create kinematic roughness by overlapping grooves on surfaces of pure nickel workpieces is hardly investigated.

In the present study, initial experiments with three electrolyte compositions were carried out to choose the most efficient one. With the chosen electrolyte, the influence of nozzle motion speed on the surface topography was analysed. From a free non-linear interpolation, adequate values were chosen to realize a targeted *Rz* of 15 µm, 20 µm and 27 µm to increase the surface area.

2. Methodology

The Jet-ECM setup recently reported to machine additively manufactured steel [14] was used in this study. The following table 1 gives an overview of the experimental parameters.

Table 1 Experimental parameters of preliminary investigations

Parameter	**Value / symbol**	**Unit / description**
Sample material	Ni 201	pure nickel
Nozzle diameter	100	µm
Nozzle motion speed	500 - 3000	µm/s
Voltage	60	V
Electrolyte type and concentration	NaCl, 15 $NaNO_3$, 30 NaOH, 4	wt.-%
Electrolyte supply rate	10	mL/min
Working distance	200	µm

A nozzle made of stainless steel was used to supply a free jet of electrolyte. The working distance between the nozzle's front surface and the workpiece surface was measured by electrostatic probing and adjusted while machining by a normal vector-based compensation of the surface inclination.

3. Analysis of the most efficient electrolyte

Concentric circles with diameters ranging from 15 mm to 37 mm and a radial distance of 150 µm were arranged as nozzle motions. To reduce machining time, 10° angles were machined as preliminary sectional experiments. Three electrolyte compositions based on previous studies and own experiences from Jet-ECM experiments of metal alloys were chosen to be analysed. The electrolyte solutions were prepared with comparable electric conductivities between 150 mS/cm and 170 mS/cm. The conductivities were chosen as compromise between sufficiently high values for useful dissolution rates and sufficiently low to avoid supersaturation. In addition, the three compositions with a certain variation of the pH with slightly acidic (NaCl, pH 5), neutral (NaNO3, pH 6) and alkaline (NaOH, pH 10) character.

The image in figure 1 c) shows a photograph captured during the Jet-ECM process. The overview image in figure 1 d) shows the arrangement of the experiments. The single electrolyte compositions are indicated by different colours, and the respective nozzle speeds are indicated by numbers, e.g., the blue-coloured "NaCl1000" was machined with NaCl electrolyte at a nozzle speed of 1000 µm/s.

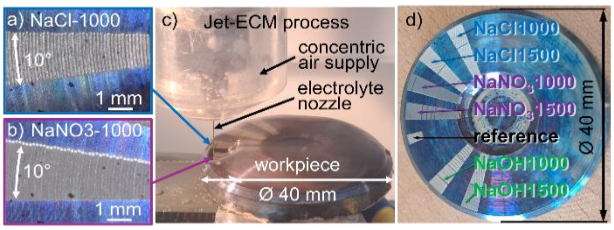

Figure 1. Detailed microscope image of machining results with NaCl (a) and $NaNO_3$ electrolyte (b), photograph of Jet-ECM (c), and arrangement of 10° sectional experiments with electrolyte variation (d)

Before and after carrying out the experiments with each of the different electrolytes, the sample was flushed with deionized water. A blue coloration can be seen on the initial surface, which is expected to be caused by spontaneous oxides. After Jet-ECM, no blue colours are cognizable, hence, the resulting oxide thickness is expected to differ from the initial one. The two exemplary top view microscope images show details of the machining results realized with NaCl (figure 1 a) and $NaNO_3$ (figure 1 b) electrolytes. The single circular grooves are clearly visible. However, certain deviations regarding localization of the electrochemical dissolution that result in differences of the kinematic roughness can be expected.

The point diagrams in figure 2 show the electric current over time of the three electrolytes detected at consistent nozzle motion speed of 1000 µm/s. Each machining operation is cognizable from current values higher than 10 mA, while recurring drops to approx. 0 mA represent infeed movements.

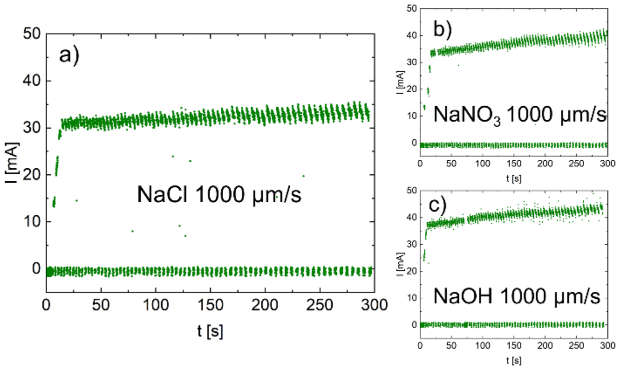

Figure 2. Electric current over time, electrolyte: NaCl (a), $NaNO_3$ (b) and NaOH (c), nozzle motion speed 1000 µm/s

According to the similar electrolyte conductivities, the measured electric currents are comparable, too. However, slightly lower average values of (30…35) mA were measured when machining the planar surface section with the NaCl electrolyte, compared to (32…42) mA with $NaNO_3$ and (36…45) mA with NaOH. In every experiment, the lowest values were detected in the beginning. The slight increase over the 300 s machining time in every experiment indicates a slight conical shape of the initial sample surface that was not compensated with the chosen strategy, which only provides a compensation of the inclination. A cognizable increase in electric current was expected at higher nozzle speed in case of a significantly reduced removal depth. However almost similar characteristics were determined at 1500µm/s, which indicates only a slight change in relation to the initial working distance. Hence, these results are not explicitly presented.

The quantification of realized surface roughness was carried out by tactile measurements with a Mahr LD 120. Three positions named P1, P2 and P3 were analysed, which were arranged in a radial distance of approximately 20 mm, 25 mm and 30 mm from the centre of the cylindrical sample. The average (AV) and standard deviation (SD) were calculated from the three single values. The results charted in Table 2 indicate the highest roughness with NaCl, even if the lowest electric currents were measured in this case. It is expected that this is caused by the highest current efficiency of this electrolyte.

Table 2 Surface roughness *Rz* of 10° sections with electrolyte variation

electro-lyte	***v* in µm/s**	***Rz* in µm**				
		P1	**P2**	**P3**	**AV**	**SD**
NaCl	1000	13.1	13.1	13.7	**13.3**	0.3
NaCl	1500	12.1	11.3	10.8	**11.4**	0.5
$NaNO_3$	1000	10.7	10.1	10.1	**10.3**	0.3
$NaNO_3$	1500	10.6	10.8	10.3	**10.6**	0.2
NaOH	1000	8.5	8.3	8.3	**8.4**	0.1
NaOH	1500	6.8	6.9	6.8	**6.8**	0.1

The roughness realized with $NaNO_3$ is slightly lower with approximately 80-90 % of the values realized with NaCl. With NaOH significantly lower values, partially only 50% compared to NaCl, were realized. Even if these electrolytes provided higher electric charge exchange, a significantly larger amount of it was consumed by side reactions rather than for electrochemical dissolution resulting in lower charge efficiency. Thus, the NaCl solution was chosen as most adequate electrolyte and used for further investigations.

4. Investigation of surface roughness as function of the nozzle motion speed with the selected electrolyte

Further experiments with NaCl electrolyte were carried out to analyse the influence of the nozzle motion speedon the machining results focusing on the surface roughness value *Rz*. Since the nozzle speeddetermines machining time and thus the depth of the single grooves, the kinematic roughness is mainly influenced by the nozzle speed. As a basis for an interpolation, six velocities ranging from 500 µm/s to 3000 µm/s with a step size of 500 µm/s were investigated. The remaining parameters were kept according to the previous investigations explained before and charted in table 1. The image in figure 3 a) shows an overview of all experiments and figure 3 b) gives a detailed view on the section machined with 1500 µm/s.

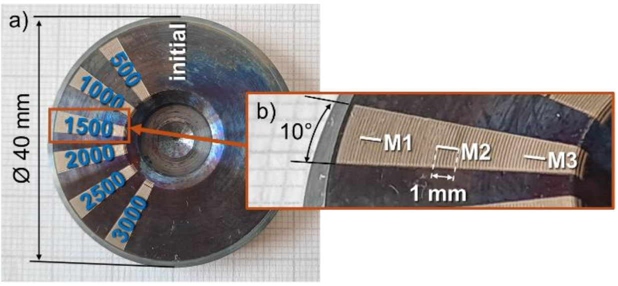

Figure 3. Overview of the 10° sectional experiments with nozzle speedvariation (left), detailed view on the section machined with 1500 µm/s (right), NaCl electrolyte

As indicated in the detailed image, three measurement lines were arranged on each section, which are situated near the sample's outer edge (M1), in the centre of the section (M2) and near the sample's inner edge (M3). Almost constant measuring lengths of approximately 1 mm were arranged perpendicularly to the circular removals after Jet-ECM to detect the kinematic roughness. For comparison, a similar arrangement was measured on the initial sample surface as indicated in the upper part of the left-handed image. In this case, the measuring lines were situated perpendicularly to the turning marks of the mechanically prepared, initial surface.

The measuring lines were captured with a Keyence VK-9700 confocal laser-scanning microscope after Jet-ECM. The VK-Analyzer software provided by the manufacturer of the microscope was used for the evaluation. To exclude strong deviations, e.g. by dust particles, the measured profiles were corrected by smoothing the surface height via the option 'simple average' of '+/-2' values 'over all areas'. The measurement of the initial surface roughness showed a value of *Rz* = 17 µm.

The point diagram in figure 4 shows the comparative results after Jet-ECM along with the initial roughness indicated by the dashed, horizontal line. The points represent the average values of three measurements M1 – M3, and the error bars highlight their standard deviations. The highest roughness of *Rz* = 32 µm was realized at the slowest nozzle speed of 500 µm/s. Here, the deepest grooves were realized by the highest electric charge exchange, which exhibited approximately 20.0 C according to an integration of the measured current over time. The lowest roughness *Rz* = 15 µm was realized at the highest nozzle speed of 3000 µm/s, where the least charge exchange of approximately 2.8 C was measured. When comparing with the initial value, a slight machining effect is also cognizable from the slight roughness reduction by approximately 2 µm.

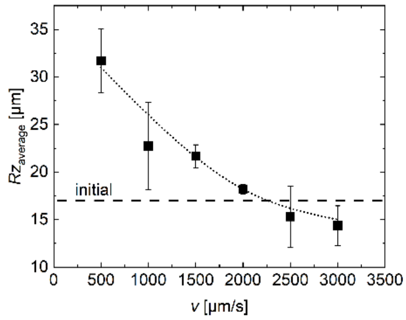

Figure 4. Average surface roughness as function of the applied nozzle motion speed with NaCl electrolyte, the dotted line indicates a non-linear spline interpolation

A declining decrease of *Rz* is cognizable with increasing nozzle speed, which is highlighted by the dotted line from a free spline interpolation. The interpolated trend was used to derive adequate nozzle motion speeds for three target surfaces characterised by surface roughness values. Grinding techniques typically result in *Rz* ≤ 10 µm, which was defined as too low for a significant increase of the surface area. High values that typically result from rough milling are at *Rz* ≥ 30 µm, which would result in an excessive machining time in Jet-ECM. Hence, three values in between were chosen considering the concentric arrangement with a radial groove distance of 150 µm resulting in a similar wavelength. Thus, *Rz* 27 µm, 20 µm and 15 µm, which represent aspect ratios slightly less than 1/5, similar to 1/7.5 and 1/10, respectively, were identified as useful target values. From the interpolated trend in figure 4, nozzle speeds of 900 µm/s, 1800 µm/s and 3000 µm/s were derived applicable.

5. Jet-ECM shaping of full sample surfaces

For prospective applications where the full surface is required as functional area, a circular arrangement with a concentric layout similar to the preliminary investigations was used and expanded to 360° circles. As shown in the top view microscope image of figure 3 a), the complete sample surfaces were machined by Jet-ECM.

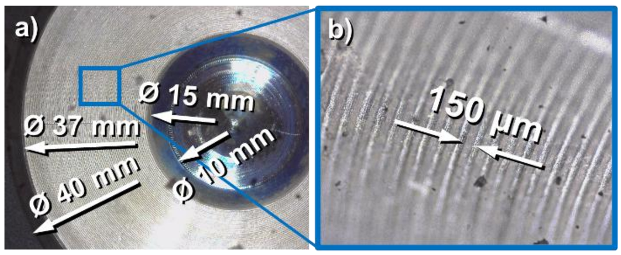

Figure 5. Top view microscope image (a) and inclined detailed view on a section (b) of a surface machined with 1800 µm/s, NaCl electrolyte, 360° circular nozzle movements

The overview image shows the machined sample surface with an outer diameter of 40 mm and an inner diameter of 10 mm. Considering the chamfers on the inner and outer edge, a machining area ranging from an inner diameter of 15 mm to an outer diameter of 37 mm remains as plane surface. The detailed image in figure 3 b) shows an inclined view on 20 out of a total of 74 circular grooves machined with a radial infeed distance of 150 µm.

The point diagram in figure 6 shows the electric current measured over time of the machining experiment with a nozzle speed of 3000 µm/s.

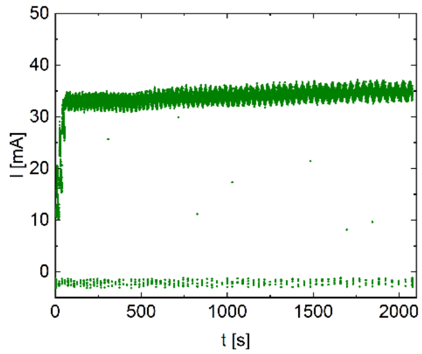

Figure 6. Electric current over time of a 360° full surface machining experiment with a nozzle speedof 3000 µm/s, NaCl electrolyte

Almost similar current values were measured compared to the preliminary experiments with the same electrolyte shown in Figure 2. A certain non-compensated deviation of the sample surface can be retraced from the regular current fluctuations of approximately ± 2 mA within the 74 machining steps that last between 20 s and 40 s according to the size of the single radii. Similar to the preliminary experiments, a slight deviation of plane surface towards a concave shape can be retraced from the current increase over time. Hence, the average current rises from 32 mA in the beginning to 35 mA at the ends of the machining process.

The integral of the electric current calculated over time resulted in an electric charge exchange of 68.7 C at the fastest nozzle speed of 3000 µm/s. The machining time in this case was 35 min. Reducing the nozzle speed to the medium value of 1800 µm/s resulted in an almost linear increase of the electric charge to 118.0 C. Due to the slower nozzle speed, the total machining time was raised to 57 min. The further reduction of the nozzle speed to the minimum value of 900 µm/s also led to an almost linear increase of the charge exchange. Here, the highest value of 226.4 C was calculated after a total machining time of 113 min. The linear correlations indicate that the average electric currents remained almost constant, regardless of the nozzle speed and machining time.

Figure 5 shows top view microscope images with colour-scaled representation of the surface topographies.

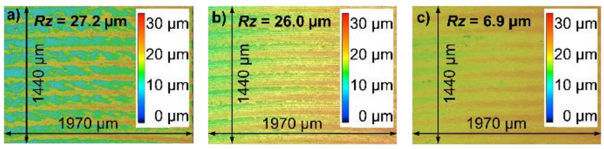

Figure 5. Colour-scaled top view microscope images of the surfaces machined by 360° circular motions with NaCl-electrolyte at a nozzle motion speedof 900 µm/s (a), 1800 µm/s (b) and 3000 µm/s (c)

The measured sections of the machined surfaces contain approximately ten single grooves cognizable from their horizontal developments. Turning marks of the preliminary mechanical preparation process are still cognizable on the original surfaces between the Jet-ECM grooves in figure 5 b) and in figure 5 c), which were machined at medium and highest speed resulting in lower removal depths compared to the result in figure 5 a). For the latter one, the turning marks were almost completely removed by the deeper grooves. The roughness measurement in this case resulted in an *Rz* of 27.2 µm, which represents very good accordance with the targeted *Rz* of 27.0 µm. However, the result shown in the central image with an *Rz* of 26.0 µm differs from the targeted value of 20.0 µm. Most significantly, the result in the right-handed image with an *Rz* of 6.9 µm represents a deviation to less than half of the targeted value of 15.0 µm.

6. Summary and conclusions

Jet-ECM was successfully carried out on sample surfaces made of nickel 201. Concentric circular grooves were machined with three electrolytes. A solution of 15 wt-% NaCl represented the most efficient machining results. Further experiments revealed a non-linear correlation between the roughness *Rz* and the applied nozzle motion speed. Discrete nozzle speeds were derived to machine the complete surfaces of initially plane samples with targeted surface roughness. While the largest target roughness of 27 µm corresponds well with the result, the medium roughness was exceeded by 30 % and the lowest value was underrun by 54 %.

A linear correlation between electric charge exchange and nozzle speed, and hence machining time, was determined. Due to the constant nozzle diameter, a constant current density distribution, and thus, consistent groove width was expected. Hence, a linear influence on the removal depth, and thus on the roughness, was expected. But the deviations indicate non-linear correlations, which indicates significant influence on the groove width that should be analysed in future. In addition, influences of the initial turning marks, which were not completely removed, on the resulting roughness need to be considered.

Obvious blue colorations of the initial surfaces were not cognizable anymore after Jet-ECM. Considering subsequent applications, where surface chemistry might be a crucial property, future analyses should be carried to determine the chemical composition of the surface and to measure changes in oxide thickness before and after Jet-ECM.

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