Some Contributions at the Technology of Electrochemical Micromachining with Ultra Short Voltage Pulses

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1. Introduction

The tendency to make progressively smaller and increasingly complex products is no longer an exclusive demand of the electronics industry. Many fields such as medicine, biomechanical technology, the automotive, and the aviation industries are searching for tools and methods to realize micro- and nanostructures in various materials. The micro-structuring of very hard materials, like carbides or brittle-hard materials, pose a particularly major challenge for manufacturing technology in the near future. For these reasons the Institute for Production Engineering and Laser Technology (IFT) of the Vienna University of Technology is working in the field of electrochemical micromachining with ultra short voltage pulses (µPECM) in nanosecond duration. With the theoretical resolution of 10 nm, this technology enables high precision manufacturing. [Kock M.] A question, which can illustrate the motivation to do this research work in this field, is: “Which parameters have to be set at a production machine and which framework conditions have to be managed to reach a desired result?” To answer this question for the materials nickel and steel (1.4301), the IFT has done experimental work.

2. Electrochemical micromachining

Basically, the term machining stands for the removal of material. Furthermore, micromachining is the production of very small scaled shapes and parts in the range of 100 µm - 0,1 µm. DIN 8580 is the classification of all manufacturing processes. Figure 1 illustrates DIN 8590 for ablation, which is a part of DIN 8580.

Ablation is a non-mechanical separation of material. It can be divided into chemical, thermal and electrochemical methods. For example water jet cutting is not yet assigned to either ablation methods or to cutting methods. Electrochemical micromachining (ECM) uses electrochemical reactions to treat a metal work piece. These reactions are for example processes in an electrolyser or a battery. In electrolysers the chemical reaction is driven by an externally applied voltage, whereas in a battery a voltage is created by a chemical reaction. As depicted in figure 1, the group of electrochemical processes are assigned to
ablation, which is a non-cutting technology. Cutting technologies for the realization of microstructures, like high speed cutting, induce mechanical stress, and thermal technologies, like laser ablation, induce thermal stress upon the work piece. Due to the fact that electrochemical technologies have none of these disadvantages, they are of interest to many industrial cases. No stress is induced in the work piece, therefore the structure of the work piece remains unchanged. Another advantage is that there is no machining force necessary and thus it is possible to machine areas which are difficult to reach. Pulsed electrochemical micromachining (PECM) as well as electrochemical micromachining with ultra short pulses (µPECM) belong to the electrochemical micromachining methods. Figure 2 shows the voltage-current curve of metal dissolution. This curve is segmented in active dissolution, passivity and trans-passive dissolution. PECM is positioned in the trans-passive section of the curve (2) whereas µPECM is positioned in the active metal dissolution area (1). Once a voltage of \( \varepsilon_P \) is reached, the current slopes down rapidly. The current remains low until the end of the passive section. At further increase of the voltage the current rises again to the trans-passive section. Machines, which are working with technologies in the range of active metal dissolution are more precise but obtain lower removal rates as others working in the trans-passive range.

Figure 3 shows the main differences of the electrochemical micromachining methods. The conventional ECM uses direct current as energy source. Whereas both PECM and µPECM, use pulsed energy sources, the major difference between these technologies is the pulse width. While the PECM uses pulse widths from milli- to microseconds, the electrochemical micromachining with ultra short pulses uses pulse widths from micro- to picoseconds. For PECM the removal rate is dependent on the current density distribution. µPECM directly controls the working gap by locally charging and discharging the so called electrochemical double layers. This leads to the advantage of µPECM, that the spatial confinement of electrochemical reactions and the thereby produced resolution is very high.
3. Electrochemical micromachining with ultra short voltage pulses (µPECM)

3.1 Method and procedure
Electrochemical micromachining with ultra short voltage pulses was developed at the Fritz-Haber-Institute of the Max-Planck-Corporation. Furthermore this innovative method for micromachining was published for the first time in the beginning of 2000. Other universities and companies working on similar topics can be found in Germany, Poland, Korea, and Austria. Since late 2010 the Institute for Production Engineering and Laser Technology (IFT) at the Vienna University of Technology has been working with this method as well. The IFT is striving to deliver machining strategies, new material–electrolyte combinations and production parameters for the industrial applicability. The machining technology of µPECM is based on the already well-established fundamentals of common electrochemical manufacturing technologies. The major advantage of the highest manufacturing precision is derived from the extremely small working gaps that are achievable through ultra short voltage pulses. This describes the main difference to common electrochemical technologies. As previously stated general advantage of electrochemical machining technologies is that the treatment of the work piece takes place without any mechanical forces or thermal influences. Therefore, no abrasive wear of the tool occurs and aspect ratios of >100 are possible which sets the basis for extremely sharp-edged geometries. There is no unintentional rounding of edges and no burring on the part.

These days appropriate electrolytes have already been found for several nonferrous metals such as nickel, tungsten, gold etc., as well as alloys like non-corroding steel 1.4301. Nevertheless, a main research focus for the Institute will be the search for new material-electrolyte combinations to expand the field of application for this technology and to enhance its manufacturing productivity. This needs to be accomplished in order to fulfil the requirements of industrial production because in industries such as the automotive sector the production rate is very important. At the Nano-/Micro-Machining-Center of the IFT, an assortment of high quality measuring devices is available. Based on the technology of µPECM and on the use of high end measuring devices, specimens and parts in the micrometer range are to be manufactured and analyzed in order to investigate material removal rates and the accuracy of resulting work piece geometries.
Due to the multidisciplinary nature of this technology, intensive cooperation with other institutes of the Vienna University of Technology in the fields of electro-technical engineering, high frequency technology and electrochemistry is established. The goal of this research will be to elevate this technology to an appropriate level of possible industrial usage by enhancing the manufacturing accuracy and the process efficiency for current components. Therefore a profound knowledge of material science, electrochemistry, and production technology for extremely small dimensions will be required. The necessary expertise in these fields will be provided by the cooperating institutes and interested companies.

To accomplish these improvements in the technology of electrochemical micromachining with ultra short pulses it will be necessary to merge several research projects which are currently dealing with the topics of piezo-driven nano-positioning devices and the development of high precision machine structures for different types of machines. Table 1 shows all the relevant adjustable parameters for µPECM. In addition to the proper choice of the electrical process parameters like the amplitude of the pulses, the pulse width, the voltages at the tool, and the work piece, the right choice of electrolyte is probably the most important aspect for this process.

| Adjustable parameters for the process                                      | abbreviations |
|--------------------------------------------------------------------------|---------------|
| amplitude of the pulses                                                  | A             |
| pulse width                                                              | P             |
| voltage at the tool                                                      | T             |
| current through the backing electrode                                    | I             |
| pulse–pause ratio                                                       | ppr           |
| diameter of the tool                                                      | D             |
| electrolyte solution                                                     | E             |

Table 1. Adjustable parameters which have an influence on the process

In figure 4, the relevant parameters of the applied voltage pulses are illustrated. The duty cycle is the sum of the pulse width and the pause time. A pulse width of 100 ns and a pause time of 800 ns conforms a pulse–pause ratio of 1/8.

![Fig. 4. Pulse-pause ratio of the applied voltage pulse, with pulse width p, length of pause, amplitude A, tool voltage T, applied pulsed voltage signal U(t)](image_url)

Fig. 4. Pulse-pause ratio of the applied voltage pulse, with pulse width p, length of pause, amplitude A, tool voltage T, applied pulsed voltage signal U(t)

Due to the fact that µPECM is one of the latest elaborated removal technologies, there are no fully developed machines available in the market. All the institutes and companies, which investigate these fields, work with machines in laboratory stage. The machine at the IFT is simple constructed and very easy to maintain, consequently it is adequate for industrial
usage. However, a more complex machine structure would give the possibility to reach the highest precision requirement. Figure 5 shows a view inside the IFT’s machine. The whole machining process takes place in a basin filled with an electrolyte solution that has to be adequately adapted to the work piece material used. At the bottom of this electrolyte basin a hole for the connection of work piece and machine can be found. It is important that the basin is well sealed, so that no leakage can occur. The basin is made of Teflon, which has resistance against the electrolytes used in the experiments. Even when filling the basin, caution is required due to the fact that once in contact with the electrolyte, the surface of the material could begin to react. To protect the work piece surface from the influence of the electrolyte-solution, a cathodic protection-current is applied by the backing electrode which is immersed in the electrolyte. At the IFT, a tungsten wire is the preferred tool for the electrochemical micromachining with ultra short voltage pulses. With the basin filled as needed, the process of work piece calibration can be performed.

Fig. 5. View inside the electrochemical machine with all important parts for the manufacturing process labelled

The measurement process for finding the work piece surface coordinate is executed automatically by the machine. Therefore a tool potential is necessary to detect the electrical short circuit thru a contact between work piece and tool. Another possible measurement process is to match the local coordinate systems of the work piece with the global coordinate system of the machine structure. With the result of this measurement process and three positioning screws on the plate, whereon the electrolyte basin is mounted, it is now possible to get the necessary congruence between these two coordinate systems. Then the manufacturing program, which conforms to a standard CNC-program, is started. The tool moves along the pre-programmed paths and selectively ablates material due to the principle, that is based on the finite time constant for double layer charging, which varies linearly with the local separation between the electrodes. During nanosecond pulses, the electrochemical reactions are confined to electrode regions in close proximity. [Schuster R.]

To view the manufacturing process and get optical magnification, a USB–camera is used. Similar to conventional electrochemical manufacturing methods the µPECM process uses an oppositional electric voltage for the work piece and the tool. At the phase boundaries between the tool and the electrolyte and also between the work piece and the electrolyte, an electrochemical double layer is formed. [Schuster R.]
Figure 6 shows the detailed structure of the double layer. The double layer consists of a rigid, outer Helmholtz layer (OHL) and a diffuse area. The inner Helmholtz layer (IHL) is a part of the OHL. In the diffuse area the hydrated metal ions are versatile. The functionality of the OHL can be understood basically as a kind of a plate capacitor, with a plate separation of half of the atom radius. [Hamann C.H.]

![Fig. 6. Simplified Stern-Graham-Model of the electrochemical double layer [Hamann C.H.]](image)

The left section of figure 7 shows the schematic illustration of the tool, the work piece in the electrolyte basin, and the electrochemical double layers illustrated as plate capacitors. The electrolyte has comparable characteristics to a linear ohmic resistor with a value that is dependent on the length of the current path. The length of the current path is equal to the distance between the tool and the work piece. The right section of figure 7 shows the equivalent circuit diagram in a simplified version of the left illustration in figure 7. Through charging and discharging the electrochemical double layer, metal ions are solvated out of

![Fig. 7. Schematic illustration of the electrochemical double layers as capacitors and the electrolyte as electrical resistor between tool and work piece (left) and the equivalent circuit diagram (right) with U(t) as energy source, C_{DL} as capacitance of the double layers and R_{electrolyte} as the ohmic resistor of the electrolyte.](image)
the metal surface. If the voltage pulse width is very short, the erosion takes place very closely to the tool \( (R_{\text{short}}) \), since the ohmic resistance of the electrolyte prevents ablation at areas further away from the tool \( (R_{\text{long}}) \) due to the double layer capacitor not being able to be sufficiently recharged. [Zemann R.]

The right illustration in figure 8 shows schematically the two different charging curves of the double layers at the work piece for \( R_{\text{short}} \) and \( R_{\text{long}} \). At smaller distances between the tool and the work piece, the charging curve is steeper; this leads to the formulas (1) and (2).

\[
\tau = R_{\text{electrolyte}} \cdot C_{\text{DL}} \tag{1}
\]

\( \tau \) time constant for double capacitor charging

\( R_{\text{electrolyte}} \) resistance of the electrolyte

\( C_{\text{DL}} \) capacitance of the electrochemical double layer

\[
U_{\text{DL}} = U(t) \cdot \left(1 - e^{-t/\tau}\right) \tag{2}
\]

\( U_{\text{DL}} \) charging voltage of the electrochemical double layer

\( U(t) \) applied voltage with dependence on time

\( \tau \) time constant for double capacitor charging

Another important influence on the charge of the double layers has the pulse width and the choice of the electrolyte. Small working gaps between the tool and the work piece of less than 1 µm are produced with pulse widths of less than 100 nanoseconds and lead to a very high resolution of the machined structure. Even more accurate machining can be achieved with pulse widths of less than 1 nanosecond and by separating the processing pulse into a pre-pulse and a main pulse, which is a future research topic for the IFT. In order to elaborate on the research work concerning the technology of using ultra short voltage pulses, the relevant demands of industry, basically increasing the material removal rate, has to be considered as a main goal. Subsequently, an increase in the already high machining accuracy is regarded as a principal target.

Another major advantage of this technology is the possibility to reverse the process electrically. This means that not only the work piece can be machined, but also the tool itself can be defined as the work piece and be machined to its ideal geometry without any further set-up. Regarding all these functionalities, the requirements for precise micromachining are
Possible tasks that can be performed with this machining centre include: tooling, milling, turning, sinking, and measuring.

Characteristics of the µPECM process with ultra short voltage pulses:
- High precision (theoretical resolution of 10 nm)
- No thermal load
- No mechanical process forces
- High aspect-ratio >100 (only limited thru the young’s modulus of the material)
- No tool wear
- Small working gaps between tool and work piece (< 1 µm)
- Manufacturing of hard materials
- Very small edge-rounding
- No burring
- Adjustable roughness of the work piece surface
- High quality measuring function

Table 2 shows that electrochemical micromachining with ultra short voltage pulses has several advantages compared to other nano- and micromachining technologies. For example the theoretical dissolution range and the aspect ratio are outstanding, whereas in case of the removal rate, µPECM is not competitive against technologies like high speed cutting. For material removal, µPECM is mainly used for post-processing and for producing surfaces with hydrophobic and hydrophilic characteristics at the moment.

| Method          | theoretical dissolution range | aspect ratio | treatable materials                      | category                      | removal rate |
|-----------------|--------------------------------|--------------|-------------------------------------------|-------------------------------|--------------|
| µPECM           | limit: 10 nm                   | > 100        | electrochem. active materials             | electrochem. micromachining   | *            |
| Lithography     | >10 nm                         | ~ 1          | etch-able, evaporable materials           | chemical method               | **           |
| LIGA            | ~ 100 nm                       | ~100         | galvanic removable materials              | mechanical/thermal method     | **           |
| Laser ablation  | ~ µm                           | ~ 1          | metals and dielectrics                    | thermal method                | **           |
| high speed      | ~ µm                           | ~1           | metals and polymers                       | cutting method                | ***          |
| cutting         |                                |              |                                           |                               |              |
| FIB             | ~ 30 nm                        | ~10          | conducting materials                      | thermal method                | **           |
| EDM             | ~ µm                           | ~10          | metals                                    | thermal method                | **           |

LIGA is the acronym for lithography (LI), electroforming (G) and molding (A)
FIB focussed ion beam milling
EDM electric discharge machining

Table 2. Comparison of nano- and micromachining methods [Kock M.]
3.2 Tooling
The favoured material used for the tool is tungsten. Tungsten can be easily treated with NaOH as electrolyte and has preferable mechanical properties like a Mohs hardness of 7.5 and a Young’s modulus of 410 GPa. For the experimental work wires with a diameter of 75 and 150 µm were used. The first tooling step is, to cut the tungsten wire manually to a length of 15 – 20 mm. The wire is fixed with a collet in the toolholder and should protrude far enough to produce the necessary geometries, mostly that is about 4 – 5 mm. The toolholder has to be protected from the acid to prevent corrosion, which is performed by a layer of Lacomit. It is a dark red fluid, once hardened it isolates the toolholder against the electrolyte. This red fluid functions as a barrier between the electrolyte and the toolholder. Only the top of the upper part of the tungsten wire is free of Lacomit to treat the work piece. Figure 9 shows two toolholders with the different diameters of tool wire.

![Fig. 9. Tools ready for manufacturing. The left tool has a diameter of 75 µm and the right tool a diameter of 150 µm, both with Lacomit layer.](image)

As mentioned before the tool/wire is cut off manually. Due to the mechanical characteristics of tungsten it is possible that the cut end splits. If that happens the split section and the usual cut end of the tool (figure 10, left) has to be removed.

![Fig. 10. Tungsten wire with a diameter of 150 µm, untreated with the end after manual cutting (left) and the finished end after electrochemical flattening (right).](image)
The flattening process is performed directly in the µPECM machine. Due to the fact that the spatial resolution and pulse width are linearly related: the higher the pulse width, the higher the spatial resolution [Kock M.], the flattening process is split into two parts to produce a tool with high quality. Another advantage of this sequential machining is that the machining time is reduced. At first a large pulse width (i.e. 400 ns) is used to increase the removal speed of the cut end. Afterwards a smaller pulse width (i.e. 80 ns) is used to create a sharp edged tool with a glossy surface. Only with such tools it is possible to produce geometries with sharp edges on the work piece. Figure 11 illustrates the difference of the radius on the tool’s top for small and large pulse widths.

![Fig. 11. Influence of the pulse width on the radius on the top of the tool](image)

3.3 Manufacturing of nickel

Nickel is a hard (Mohs hardness: 3,8) and ductile metal with a silvery-white and slightly golden shine. Nickel is apart from chrome and molybdenum an important element for the refinement of steel. The ferromagnetic metal is corrosion-resistant. Nickels protective oxide surface resists most acids and alkalis. The corrosion-resistance is one of the most important characteristics of parts in laboratory environments or health care, therefore nickel is the common material in those branches. For the electrochemical manufacturing of nickel the electrolyte hydrochloride acid (HCl) is used. HCl deactivates the passive surface of nickel and renders the material processable. The following experiments were done to find the optimal processing parameters for the manufacturing of products and special surfaces made of nickel. To evaluate the outcome of the experiments, the produced structures were measured with a high-end optical measuring device. Also optical considerations through a light microscope helped to evaluate the following characteristics of the produced surfaces:

- shape / geometry
- topology (smoothness of the bottom surface)
- shine of the surface
- edge rounding

3.3.1 Pulse width (p) and amplitude (A)

In the first experiment the pulse width and the amplitude of the pulse were varied in order to see which effects the adjustment of these parameters cause. The experimental setup is a block with five parallel grooves. Every groove is made with different pulse widths from 400 ns to 80 ns. A sketch of the groove geometry is illustrated in figure 12. Overall four of these blocks with different amplitudes were manufactured. The range of the amplitudes was from
3000 mV to 2100 mV in 300 mV steps. After measuring the width of every groove, the working gap can be calculated via formula (3).

\[
D + 2a = B
\]

(3)

\[D\] tool diameter in µm
\[a\] working gap in µm
\[B\] measured width of the groove in µm

Fig. 12. Sketch of the produced groove

The diagram in figure 13 shows that a smaller pulse width reduces the working gap. The optical estimation shows that grooves made with lower pulse widths have much better optical qualities (figure 13, left). This outcome can be explained by the localization of the manufacturing reactions. Smaller voltage pulses lead to a spatial confinement of the electrochemical reactions so that the working gap shrinks and the geometry gets more precise which is confirmed in figure 13, right. As a consequence, the pulse width is the most important parameter for the machining precision. Dependent on the machine, the minimal pulse width of \(p = 80\) ns is further used in the experiments to produce grooves in high quality. The adjusted electrochemical parameters for this experiment are indicated in table 3.

Fig. 13. Illustration of grooves (left) - from top downwards different pulse widths were used. Diagram of the appurtenant working gaps over pulse widths (right).
Table 3. Adjustments for the experiment of figure 13

Figure 14 shows that similar to the pulse width the reduction of the amplitude causes a reduction of the working gap. At a pulse width of $p = 80$ ns an amplitude of less than 3000 mV does not lead to a removal of material, due to the fact that the double layers cannot be sufficiently charged with the provided energy. Equally the provided energy of 2400 mV amplitude and 100 ns pulse width is not sufficiently for production. The overview of the production parameters for these experiments is mentioned in table 4.

Table 4. Adjustments for the experiment of figure 14

3.3.2 Electrolyte-concentration

The concentration of the electrolyte is a very important parameter for the electrochemical processing. In the equivalent circuit diagram of the electrochemical cell, the electrolyte is equal to an ohmic resistor. For this experiment hydrochloric acid (HCl) in three different concentrations was used to explore the correlation between the electrolyte-concentration and the working gap. The diagram in figure 15 shows that the reduction of the electrolyte concentration leads to smaller working gaps. This outcome can be explained by the reduced conductivity of the electrolyte and the following localization of the reactions. A reduction of the concentration increases the resistance because of the lack of ions in the aqueous solution. In such solutions ions are the charge carriers and therefore responsible for the electric conductivity. The illustration in figure 15 shows the optical differences of changed electrolyte concentrations. The processing parameters for this experiment are indicated in table 5.

Fig. 14. Working gaps over amplitude at different pulse widths.
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Fig. 15. Image of grooves made with 0.5M HCl and 1M HCl for $A = 2400 \text{ mV}$ (left). Working gap over pulse width at different electrolyte concentrations for $A = 3000 \text{ mV}$ (right)

| $A$ = varied | $p$ = varied | $T = 200 \text{ mV}$ | $E$ = varied |
|--------------|--------------|----------------------|-------------|
| $I = 1000 \mu\text{A}$ | $\text{ppr} = 1/8$ | $D = 75 \mu\text{m}$ |             |

Table 5. Adjustments for the experiment of figure 15

3.3.3 Current through the backing electrode ($I$)
To investigate the influence of the current through the backing electrode, the current was varied between 500 $\mu\text{A}$ and 4000 $\mu\text{A}$. The results in figure 16 (left) show an increased processing time at higher currents. The minimal working gaps are in the range of 2000 to 3000 $\mu\text{A}$, as illustrated in figure 16 (right). Because of the optical criteria and the working gap a current of $I = 2000 \mu\text{A}$ was used for further experiments. The illustration in figure 17 shows the difference between a high-quality and a low-quality groove. The electrochemical parameters for this experiment are shown in table 6.

Fig. 16. Processing time at different currents (left) and working gap at different currents (right)
3.3.4 Tool voltage (T)

For successful application of ultra short voltage pulses for electrochemical machining, the electrochemical conditions, e.g. the average electric potentials of the tool (T) and the work piece have to be precisely controlled. These potentials are independently adjusted by a low-frequency bipotentiostat and a platinium backing electrode. [Kock M.]

To investigate the influence of T, seven grooves with different tool voltages were produced. The production parameters for this manufacturing are indicated in table 7. After the measurement and evaluation of the working gap via formula (3), the results show that between -100 mV and + 100 mV the working gap reaches a minimum (figure 18, left). The optical appearance of these grooves has also the highest quality (figure 18, right). Another advantage is that the processing time decreases with lower tool voltages. For the further experimental work a tool voltage of +100 mV was used.
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| A      | p       | T       | E       |
|--------|---------|---------|---------|
| 3000 mV | 80 ns   | varied  | 0.5 M HCl |
| 2000 µA | 1/8     | 75 µm   |         |

Table 7. Adjustments for the experiment of figure 18

### 3.3.5 Pulse-pause ratio

The pulse-pause ratio is an important parameter that influences the electrochemical reactions. To ensure a precise and fast dissolution of the material, the ratio of pulse time to pause time should be correctly chosen. Every single pulse that charges the electrochemical double layer dissolves a monolayer of atoms from the material into the electrolyte solution. Due to the fact, that one monolayer of atoms is a very small amount of material the pulses must be applied with very high frequency to solvate the material in a reasonable rate. If the ratio is too high, the process time is unnecessarily lengthened as these rates obey an exponential law (Butler-Volmer equation). To find an appropriate pulse-pause ratio, five grooves with a different ppr-parameter were produced. Figure 19 shows a decreased removal rate at higher pulse-pause ratios for the drilling and milling processes. All of these grooves have the same working gap with negligible deviations in the range of maximal 5 µm. There is great potential to speed up the process by reducing the pulse-pause ratio without losing much precision. The used parameters for the experiment are specified in table 8. Considering the optical estimations, a pulse-pause ratio between 1/6 and 1/8 is recommended.

![Fig. 19. Removal rate over pulse-pause ratio](image)

### 3.3.6 Drilling with µPECM

In this experiment the maximum possible drilling depth should be found. The drilling process works without any problems to a depth of 140 µm. All over the removal speed slows down slightly. At a depth of 140 µm the drilling speed slows down rapidly and the experiment has to be stopped. An explanation is that in this depth the exchange of
electrolyte is not sufficient, so the dissolved metal ions saturate the electrolyte in the drilled hole and prevent any further metal dissolution. This can be disabled by an alternately up and down movement of the tool to realize a kind of flushing (pulsed mechanical movement). In figure 20 the removal speed over drilling depth is shown. Table 9 indicates the drilling parameters for the process.

![removal speed over drilling depth](image)

**Fig. 20.** Removal speed over drilling depth

| A = 3000 mV | p = 80 ns | T = 100 mV | E = 0,5M HCl |
|-------------|----------|------------|--------------|
| I = 2000 µA | ppr = 1/8 | D = 75 µm  |              |

Table 9. Adjustments for the experiment of figure 20

### 3.3.7 Dwelling time

For this experiment the tool was positioned 4 µm above the nickel surface and remained at this position for different time periods. At the first position the dwelling time was 0 seconds. On each position the dwelling time was doubled to finally 640 seconds. The longer the pulses are applied, the more material is removed (figure 21). At 0 seconds only a scratch was produced. At higher dwelling times the holes are deeper. Finally, the removal rate decreases and a maximum gap will be developed. The electrical resistance between tool and work piece grows with the distance of them, until finally no more reaction/dissolution is possible. A referential groove was produced for the measurement. It is very important to adjust an optimized machine feed rate, because longer dwelling times lead to enlarged working gaps. The Adjustments for this experiment are illustrated in the table 10.

| A = 3000 mV | p = 80 ns | T = 100 mV | E = 0,5M HCl |
|-------------|----------|------------|--------------|
| I = 2000 µA | ppr = 1/8 | D = 75 µm  |              |

Table 10. Adjustments for the experiment of figure 21
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Fig. 21. Averaged groove depth over dwelling time

Fig. 22. Images of the microstructure, photographed with a scanning electron microscope (SEM) at different resolutions
3.3.8 Part production (micro injection mould)
The manufactured microstructure in figure 22 has an overall diameter of less than 50 µm, is 15 µm deep, and approximately shaped like a gearwheel. This microstructure was manufactured in 4 hours, with an electrolyte concentration of 0.2M HCl. The tool for this experiment (figure 23) was made out of a tungsten wire with diameter $D = 150$ µm by successively reducing the diameter in the tooling basin to $< 5$ µm. The magnification of 45 in a light microscope was not sufficient to examine the structure; therefore, a scanning electron microscope has to be used. The experiment shows that the production of a micro injection mould in a range $< 100$ µm is possible with the IFT’s machine.

![Image of the tool to produce the micro injection mould](image)

Fig. 23. Image of the tool to produce the micro injection mould with a top of $D < 5\mu m$.

| $A = 3000$ mV | $p = 80$ ns | $T = 100$ mV | $E = 0.2$M HCl |
|------------|-----------|-------------|---------------|
| $I = 2000$ µA | $ppr = 1/8$ | $D < 5$ µm |               |

Table 11. Adjustments for the experiment to produce a micro injection mould

3.4 Manufacturing of steel (1.4301)
1.4301 steel is the most widely used non corroding steel and it has a very broad scope of application. The need of micro-structuring of such a standard material is continually growing. A solution of hydrofluoric acid and hydrochloric acid was used as electrolyte. The exact designation of this electrolyte solution is 3% HF/3M HCl. As previously mentioned, four criteria were used for the optical consideration of the grooves. These are:
- shape/ geometry
- topology/ smoothness of the bottom surface
- shine of the surface
- edge rounding
The experiments on 1.4301 were the same as on nickel with the difference that the electrolyte was not changed.

3.4.1 Pulse width (p) and amplitude (A)
Grooves with a length of 200 µm and a depth of 20 µm were manufactured. Thereon the amplitudes and the pulse widths were varied and the optical consideration of the grooves was performed to classify the results. The spatial resolution is almost linearly related to the pulse width. [Kock M.]. Figure 24 confirms this as the working gap shrinks with the reduction of the pulse width. The combination with the highest manufacturing precision
was $A = 2800 \text{ mV}$ and $p = 100 \text{ ns}$. The production with shorter pulse widths with the tool diameter of 150 µm was not possible. The energy applied by shorter pulse widths or lower amplitudes was not sufficient to recharge the double layer in order to realize material removal. By increasing the amplitude it was possible to finish grooves made with a pulse width of 80 ns, but the overall result was not favorable. The overview of the used parameters for the experiment shown in figure 24 is illustrated in table 12.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{working_gap_over_pulse_width.png}
\caption{Working gap over pulse width for $A = 2800 \text{ mV}$}
\end{figure}

\begin{table}[h]
\centering
\begin{tabular}{|c|c|c|c|}
\hline
$A$ & $p$ & $T$ & $E$ \\
\hline
varied & varied & 100 mV & 3% HF/3M HCl \\
$I$ & $ppr$ & $D$ & \\
1500 µA & 1/8 & 150 µm & \\
\hline
\end{tabular}
\caption{Adjustments for the experiment of figure 24}
\end{table}

### 3.4.2 Current through the backing electrode ($I$)

This experiment was performed to show the influence of the cathodic protection-current on the process. The applied current protects the work piece in the electrolyte from corrosion or any other reactions. Eight grooves with the same dimensions as in the experiment before were made with $I$ from 4000 to 500 µA. An obvious trend of how the cathodic protection-current influences the process could not be observed from the series of grooves. The results show that $I$ from 3000 to 4000 µA achieves the smallest working gap and the best surface condition. Figure 25 shows two grooves with an obvious optical difference. Topology of the ground, sharpness of the edges, and form of the groove is much better with $I = 3000 \text{ µA}$. Therefore, $I$ has to be fixed at 3000 µA for the next attempts. All other electrochemical parameters for this experiment are indicated in table 13. During this phase of the experiments, the choice of which of the parameters to fix was dedicated by the optical assessment and the working gap measurement and not yet by the removal rate.

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3.4.3 Pulse-pause ratio

The idea of this experiment was a variation of the pulse–pause ratio from 1/5 to 1/11. Figure 26 shows the manufactured grooves of the ppr experiment. The manufacturing parameters of this process are illustrated in Table 14. For this experiment the voltage at the tool was zero. An experiment with the potential at the tool has shown that a very low voltage leads to the best results in case of the optical considerations. But these low tool voltage could bring up some problems.

When the drilling depth is higher, it can happen that the positive ions from the work piece treatment deposit at the tool. This deposition starts with a slight change of the tool geometry and can lead to a kind of ion based short circuit bridge between tool and work piece. Such a short circuit disrupt the manufacturing process. For the further experimental work the tool voltage was set at 100 mV to avoid any unwanted occurrences.

Table 13. Adjustments for the experiment of figure 25

| $A = 2800 \text{ mV}$ | $p = 100 \text{ ns}$ | $T = 100 \text{ mV}$ | $E = 3\% \text{ HF/3M HCl}$ |
|----------------------|------------------|------------------|-------------------|
| $I = \text{varied}$ | $\text{ppr} = 1/8$ | $D = 150 \text{ mUm}$ |

Fig. 26. Grooves produced for the pulse–pause ratio experiment

Figure 27 shows that the higher the pulse–pause ratio, the lower the removal rate. If within a period of time fewer pulses are applied, the charging and discharging of the electrochemical double layer also occurs less frequently. This is the obvious explanation for the low manufacturing speed of the groove made with a ppr of 1/11. For this ratio the manufacturing process was stopped because economic material removal could not be realized.
The best combination of the optical quality of the surface and the removal rate was detected from a pulse–pause ratio of 1/7. The consequence was to fix this parameter for the next experiments. Based on the optical result, the pulse–pause ratio of 1/5 was not viewed in the evaluation.

Fig. 27. Removal rate over pulse–pause ratio

![Graph showing removal rate over pulse–pause ratio]

Table 14. Adjustments for the experiment of figure 26 and 27

| A = 2800 mV | p = 100 ns | T = 0 mV | E = 3% HF/3M HCl |
|-------------|------------|----------|------------------|
| I = 3000 µA | ppr = varied | D = 150 µm |                   |

3.4.4 Drilling with µPECM

To this point in the series of experiments all grooves were manufactured with an adjusted depth of 20 µm. This experiment was done to show how the manufacturing depth influences the process. Figure 28 shows that at a depth between 125 – 175 µm the speed of removal rapidly reduces from above 35 to less than 10 µm per minute. A possible explanation is that the electrolyte is not sufficiently available in the drilled hole. The electrolyte is sated in such depth, so the transport of new solved ions out of the bore slows down and the removal speed reduces. After the depth of around 425 µm was reached, the process was stopped, because it was no longer possible to manufacture the work piece. To prepare sufficient electrolyte solution in such depth and thus realize better transport of the solved ions out of the bore, the mechanical movement of the tool inside the drilled hole could be pulsed to get a kind of flushing and reach higher depths. The manufacturing parameters of this experiment are illustrated in table 15.
Fig. 28. Removal speed over drilling depth

| A = 2800 mV | p = 100 ns | T = 100 mV | E = 3% HF/3M HCl |
|-------------|------------|------------|------------------|
| I = 3000 μA | ppr = 1/7  | D = 75 μm  |                  |

Table 15. Adjustments for the experiment of figure 28

3.4.5 Dwelling time

Figure 29 shows the effect of the dwelling time during the process. In this experiment a tool with a diameter of 150 μm was positioned 4 μm above the work piece’s surface. The parameters of the experiment are shown in table 16. The tool was stopped at eight different positions. On the first position the dwelling time was about 0 s, and afterwards it was doubled on each position from 5 s to 640 s. With the maximum depth of around -10 μm at the longest dwelling time this experiment confirmed the relevance of the dwelling time for the manufactured geometry. If the manufacturing feed rate is chosen too low, the precision of the manufactured geometry shrinks - caused by the time-dependent development of the working gap. This is one of the effects, which has to be controlled in industrial usage of the µPECM technology. Table 16 gives a overview of the process parameters for the dwelling time experiment.

Fig. 29. Averaged groove depth over dwelling time
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A = 2800 mV  p = 100 ns  T = 100 mV  E = 3% HF/3M HCl
I = 3000 µA  ppr = 1/7  D = 150 µm

Table 16. Adjustments for the experiment of figure 29

3.4.6 Manufacturing of the Institute’s logo with µPECM

The goal of the last experiment was to produce a micro structure with the knowledge of the described experimental work. So, the emblem of the Institute for Production Engineering and Laser Technology was chosen to be machined in a small steel plate. The first step, as in all other experiments, was to provide an appropriate tool to produce a high quality result. To manufacture grooves with a maximum width of 30 µm a tool diameter of about 20 µm is necessary. In a special tooling basin the diameter reduction from 150 µm to 20 µm was realised. Figure 30 shows the result of the tooling process.

![Fig. 30. Tool before (diameter 150 µm - left) and after the tooling process (diameter ≈20 µm - right)](image)

Figure 31 shows the result seen through a light microscope with forty-five-fold magnification and table 17 illustrates the used processing parameters. To get an idea of the dimensions of the emblem, a human hair was attached for comparison. The total removal time to produce this logo was 03:04:44 (hh:mm:ss). The groove 0-1 has an adjusted length of 322,5 µm and an adjusted depth of 30 µm. The manufacturing time was 11,02 minutes and the width is 26,3 µm. This leads to a removal rate of 0,027 10^6 µm³/min.

A = 2300 mV  p = 80 ns  T = 100 mV  E = 3% HF/3M HCl
I = 3000 µA  ppr = 1/7  D ≈ 20 µm

Table 17. Adjustments for the manufacturing of the Institute’s logo

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4. Conclusion

The technology of electrochemical micromachining with ultra short voltage pulses has successfully displayed the many applications especially for prototype building or for the manufacturing of special products where there is no other technology which can combine a very high manufacturing precision for special materials without any mechanical forces or thermal influences. [Zemann R.] In principal, it can be applied to all electrochemically active materials, including semiconductors. [Schuster R.] Also, the use of applicable effects on process accuracy and material removal rate of difficult to machine materials offers a wide range of possible applications for μPECM technologies in the future. The occurring electrochemical problems are tradable and topics at the IFT, as well as the micromachining of many different materials like nickel, tungsten, titanium, non-corroding steels, or hard metals. As already mentioned, the machine at the IFT is simple constructed and very easy to maintain, so it is adequate for industrial use. However, a more complex machine structure would enable to reach highest precision requirements, but needs more maintenance and a higher financial investment. The experiments on the IFT’s machine proved that electrochemical micromachining is achievable for SME’s. With the parameter sets in table 18 and 19 appropriate results were manufactured. Appropriate results means, that with these parameters, the grooves deliver adequate working gaps and optical results – geometry, topology, sharpness of the edges, and shine of the ground. Other parameters would perhaps reach higher removal rates, but on the other side lose quality with regard to precision.
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| A = 3000 mV | p = 80 ns | T = 100 mV | E = 0,2M HCl |
|-------------|-----------|------------|-------------|
| I = 2000 µA | ppr = 1/8 | D = 75 µm  |             |

Table 18. Adjustments to achieve appropriate results working on nickel

Caused by the complexity of this technology, the variation of one of the adjustable parameters could significantly affect the result. Therefore at this point of research it is not definitely possible to give tangible instructions on how to reach requested results. It is very much experience necessary to interpret the proceedings at the machine correctly and to enhance the manufacturing process. Due to the multidisciplinary nature of this technology, intensified cooperation with other experts and an extensive research study has to be done; before a reasonable forecast for the processing parameters of a specific manufacturing process can be done.

| A = 2800 mV | p = 100 ns | T = 100 mV | E = 3% HF/3M HCl |
|-------------|-----------|------------|------------------|
| I = 3000 µA | ppr = 1/7 | D = 150 µm  |                  |

Table 19. Adjustments to achieve appropriate results working on steel (1.4301)

5. Prospects

In the course of the experiments, it was also tried to treat carbide metal by electrochemical micromachining with ultra short pulses. The work piece used for experimental work was a K40FF. This carbide metal consists of a 12% cobalt matrix with 88% tungsten-carbide as strengthener. The electrolytes used were 3% HF/3M HCl and 2M NaOH. Both electrolytes were found to be unsuitable in combination with this carbide metal. A major challenge is to find new material-electrolyte combinations to apply electrochemical micromachining with ultra short pulses. The IFT has some tangible visions to realize treatment of carbide metal. A prospectively area for application of this technology could be protection of plagiarism. Technical devices and parts could be branded with the µPECM technology so, that only the producer can find the printed serial number, due to the small size of it.

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The book "Cutting Edge Research in New Technologies" presents the contributions of some researchers in modern fields of technology, serving as a valuable tool for scientists, researchers, graduate students and professionals. The focus is on several aspects of designing and manufacturing, examining complex technical products and some aspects of the development and use of industrial and service automation. The book covered some topics as it follows: manufacturing, machining, textile industry, CAD/CAM/CAE systems, electronic circuits, control and automation, electric drives, artificial intelligence, fuzzy logic, vision systems, neural networks, intelligent systems, wireless sensor networks, environmental technology, logistic services, transportation, intelligent security, multimedia, modeling, simulation, video techniques, water plant technology, globalization and technology. This collection of articles offers information which responds to the general goal of technology - how to develop manufacturing systems, methods, algorithms, how to use devices, equipments, machines or tools in order to increase the quality of the products, the human comfort or security.

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