Chapter

Pulsed Electrochemical Micromachining in Stainless Steel

Pablo Rodríguez, Daniel Hidalgo and Julio Eduardo Labarga

Abstract

This chapter presents research on pulsed electrochemical micromachining of stainless steel. Suitable equipment to study the process is described as well as a fitting procedure to machine and measure the variables involved. The pulse on-time must be maintained in the order of ns to achieve a good current confinement since the tool is active. Some experiments were carried out to assess the most important variables of the process: current confinement, surface roughness, material removal rate and efficiency. The current confinement has been observed to worsen when the pulse on-time increases, as well as the surface roughness. The material removal rate and efficiency increase with the voltage amplitude and the pulse on-time. The voltage amplitude must be higher than 12 V so that the phenomenon of passivation does not affect the process. There is a compromise in the choice of the variables, so a suitable combination of parameters is determined in order to achieve a good material removal rate with an acceptable result.

Keywords: pulsed electrochemical micromachining, current confinement, material removal rate, efficiency

1. Introduction

Microfabrication consists of obtaining products or parts with features at micro- or submicroscale, therefore requiring very narrowly controlled material removal. Microfabrication has been widely used for the manufacture of holes in injectors, fluidic microchemical reactors requiring microscale pumps, micromoulds and many more applications, as described by Brousseau et al. [1]. Microfabrication plays an increasingly important role in the miniaturisation of components from biomedical applications to manufacturing sensors. Surfaces to be obtained are slots, complex surfaces, microholes, etc. Combinations of those features must frequently be achieved in the industry of microelectronics. These parts are very often manufactured by conventional processes with all the limitations and problems involved, such as tool wear, inaccuracy due to low rigidity of the tool, heat generated by the process, etc. With the development of MEMS and multiple benefits of the microsystems, microproducts are widely accepted in various fields of applications like aerospace, automotive, biomedicine, etc. [2]. In this context, non-conventional processes, and especially electrochemical micromachining, acquire greater significance due to their specific characteristics to avoid the problems of conventional processes.

Since the first years of developments in electrochemistry, electrochemical methods have played an important role in precision technologies to machine structures and parts. In the 1950s, electrochemical machining arose as the most widely used

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technique to manufacture complex geometries, such as turbine blades, generally in dense materials. The ease of application of this technology along with the inherent advantages of the process, such as good surface roughness, promoted its application to more advanced processes in the field of micromechanics, microelectronics and micro-systems [3]. Electrochemical deposition techniques were used as standard technology to deposit copper to obtain connections in high performance circuits while lithographic techniques, LIGA, are used to manufacture micromoulds [4, 5].

Electrochemical micromachining is a highly specialised process used in the aerospace industry. Today, it is starting to be used in other industries, where difficult-to-manufacture parts, complex surfaces and components of a microscopic scale need to be obtained. Electrochemical micromachining is today widely used for manufacturing semiconductor elements and thin metallic films [6]. In addition, electrochemical micromachining can be easily hybridised with other processes to broaden the process capabilities and material processing window [7].

Analogous to conventional electrochemical machining (ECM), pulsed electrochemical micromachining (PECMM) is a controlled process of anodic dissolution to remove the material with current densities in the order of 10^5 A/m^2 between the tool (cathode) and the workpiece (anode) through the electrolyte [8]. PECMM uses a pulsed voltage signal and must be analysed per pulse according to the structure of the Helmholtz/Gouy-Chapman/Stern double layer [9], which can be modelled as a resistance in parallel with a capacitor. This model has provided good results in experiments and indicates that the current is used at first to charge the capacitor (capacitive current). When the charge is high enough, that is, when its voltage is high enough, some current will flow to be used in the anodic dissolution process (faradaic current) since the polarisation or overpotential will have a significant value. Therefore, two stages can be distinguished in each pulse. The first part of the pulse on-time is a transient period in which the current is used in the polarisation of the double layer, which has to be high to achieve fast polarisation. The second stage is a steady period in which the current is used mainly for the anodic dissolution. In this context, what seems most fitting is that the transient process (non-faradaic) should be very short and the steady process (faradaic) very long. In addition, the intermittent supply of voltage provides idle time to flush the hydrogen bubbles and sludge from the machining zone and also increases control over the dissolution process [10]. However, a long steady period decreases the accuracy of the process as the current confinement under the tool tip worsens when this period is lengthened. Therefore, a compromise in the time of the steady-state period is required. By solving the differential equation of the equivalent circuit, the expression of the current as a function of time is obtained. The resulting time constant is the product of the electrolyte resistivity, the capacity of the double layer and the distance between the interelectrode gap (IEG) [8].

$$\tau = \rho \cdot \mathrm{IEG} \cdot c_{DL} \tag{1}$$

A high value of the constant time will cause the current lines to spread over a broad area from the tool tip, thus reducing the accuracy of the process. Therefore, a low pulse-on time must be chosen to achieve accuracy.

By causing the tool to move towards the workpiece, the material is removed under its tip, since the current density is higher at a lower distance between the tool and workpiece, and thus, the geometry of the tool is copied as a cavity in the workpiece. As compared with other processes, PECMM is a high-precision technique to obtain holes of a small diameter or to obtain crack-free microcomponents without any residual stress. There are two methods of achieving accuracy with

electrochemical micromachining. One of them is to use a tool in which all the surface is isolated except for the tip. This method ensures that all the current flows from the tip and that the cavity obtained is equal to that tip, since this current is responsible for the anodic dissolution of the material. Another method is the use of ultrashort voltage pulses, usually shorter than 100 ns. This method achieves high accuracy by confining the faradaic current density under the tool due to the incomplete charge of the double layer in areas far from the part through which a very low current will flow.

An important phenomenon which affects the process is the formation of a passive oxide layer that hinders the anodic dissolution [11]. The characterisation of this phenomenon is very important, since some processes like electropolishing are performed more adequately in passivation conditions [12]. When this takes place, the voltage applied has to be above a threshold value to cause effective machining [13]. It can also be avoided by adding acid to the electrolyte, such as HCl or H_2SO_4, which dissolves the passive layer. This layer can be considered an additional electrical resistance in the equivalent circuit, which prevents the current from being confined under the tool tip [14]. According to this explanation, the current which flows from the sides of the tool finds a similar resistance to that which flows from the tool tip and therefore the current is spread over a broad surface.

Significant advances have been made in the research of this process on many materials such as aluminium, titanium, steel and copper [15, 16]. Stainless steel is a very important material to be used in any type of microcomponent, but dissolution is difficult since its chemical properties are not very suitable for this process. Some of the existing studies were performed specifically on stainless steel [17–20]. Nevertheless, the pulse on-time used in those cases is too high to obtain a good confinement of the current. Furthermore, there are few studies in which the size of the tool is as small as a few microns. Though some work has been done in order to control the process by varying the main parameters [21], there is a huge amount of work to be done to characterise this process correctly as regards the values of the parameters in order to obtain a good result in terms of current confinement, surface roughness, material removal rate (MRR) and experimental set-up. In this work, a broad study has been made of the results of PECMM in stainless steel with pulse on-time values in the order of ns as a function of the main variables.

2. Experimental set-up

The experiments performed for the study were made by means of equipment that allows accuracy and ease of handling of tools and parts to be achieved. **Figure 1** shows a sketch of this equipment.

The equipment for the experiments rests on an anti-vibrations Table TMC, which provides a floating bench that prevents the tool and the part from oscillating. The position of the recipient is controlled by a three-dimensional (3D) nanometric positioning system based on a piezoelectric technology and with a resolution of 1 nm. There is a system of recirculation for the electrolyte, which flows constantly through the cell to a tank from which it is pumped to the cell after passing through a filter. Thus, the particles that appear in the cell are constantly being removed from the electrolyte. Experiments were performed in a solution of $NaNO_3$ at 2% in weight as the electrolyte.

The material of the workpiece is AISI 304 stainless steel and the tool is made of Tungsten, 99.7% high purity. The tools are pins with a very small tip, measuring about 5 μm in diameter. The tool tip is sharpened by means of anodic dissolution in which the tungsten pin is used as the anode and the sheet of stainless steel as the

cathode. The electrolyte used for this process is a solution of KOH at 5% in weight. **Figure 2** shows a picture of the equipment used for the process. In **Figure 3**, a microtool used for the process is shown.

In order to apply the voltage pulses to the system, a Function Generator Agilent 33,250 A is used, which generates voltage signals of several types and a broad range of frequency, up to 100 MHz, which corresponds to a width of 10 ns in the

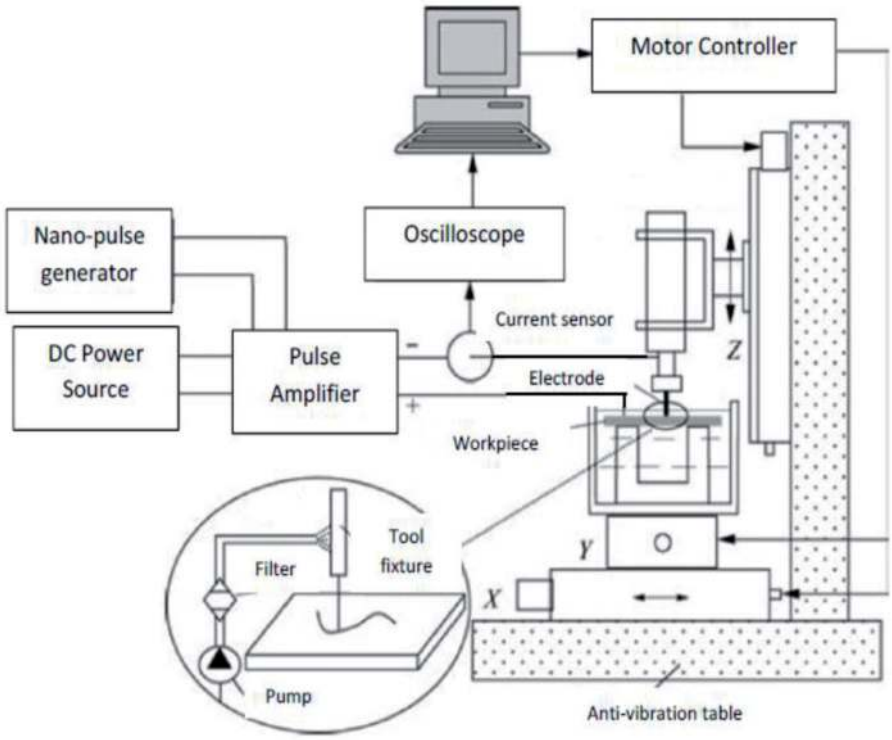

Figure 1.
Sketch of the equipment used for experiments.

Figure 2.
Equipment for electrochemical micromachining.

voltage pulses. The signal applied by the generator passes through a developed pulse amplifier that provides the necessary current for the process corresponding to the voltage amplitude. The amplifier is fed by a DC power source Keytheley 2220G-30-1 which provides a current limiting system, so that the amplifier is not overloaded. The graphs of voltage and current between electrodes for a machining process are shown in **Figure 4**.

In this graph, the current rises from zero to the stationary value going through a transient period of about 50 ns. Taking the criterion that the time constant is the time taken by the system to reach 63% of the total amount of change, a value of 25 ns for the time constant is deduced for a value of IEG = 1 μm and the other conditions described above. Therefore, values of on-time pulse above 50 ns are adequate for the process.

The electrochemical process is observed by means of a Supereyes USB Portable Digital Microscope B008 connected to a computer in which the amplified image

Figure 3.
Microtool used for the process.

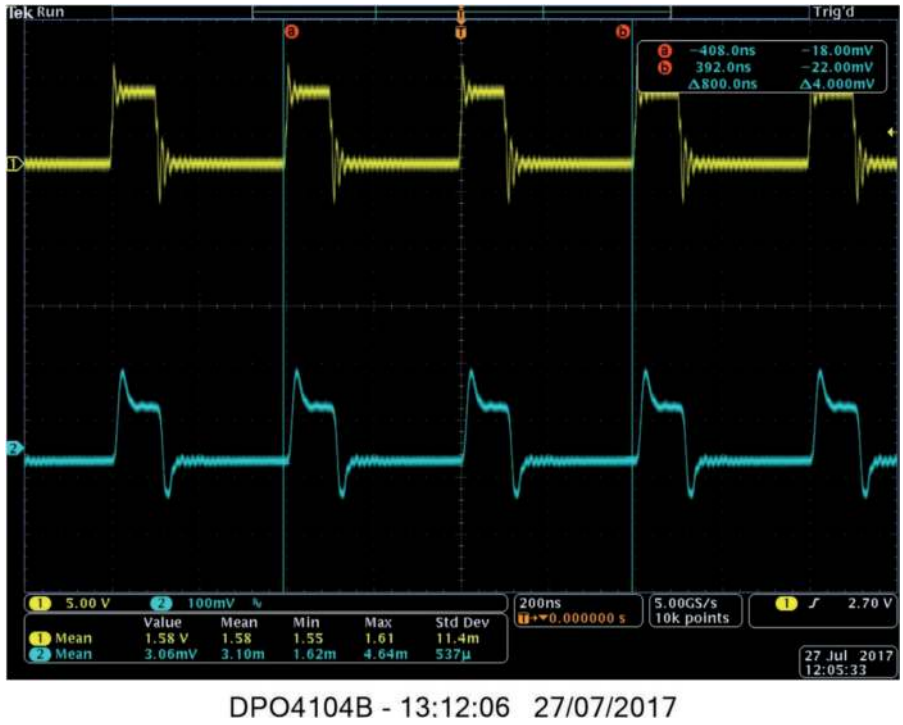

Figure 4.
Signals of voltage and current between the electrodes in the machining process. Signal 1: voltage (V) and signal 2: current (mA).

of the tool and the area of the part being machined can be seen. This microscope is also helpful to set the approach of the tool to the workpiece in order to establish the reference of distance.

The voltage applied to the cell as well as the current passing through it is measured by means of a digital oscilloscope Tektronic DPO 4104, which allows several signals with up to 3 GHz to be visualised by using a maximum sample rate of 5 Gs/s. It also permits mean signal values to be measured, applying filters and making mathematical operations with signals, such us obtaining Fourier Transforms.

In order to observe and measure the dimensions of the features machined, as well as the tip of the tools, a scanning electron microscope and an optic microscope were used.

The reference for the position of the tool is taken as the point of value 0 for the IEG. That position was found by electrical contact between the tool and the workpiece. It is observed that, when using an active tool, the current does not change significantly as IEG decreases. However, when there is electrical contact, the current increases suddenly to a very high value. This phenomenon allows the reference to be found with a very slow movement of the tool and, therefore, a brusque impact is avoided, which could damage the tool tip.

3. Results and discussion

PECMM works on the principle of Faraday's laws of electrolysis. The process consists of applying a potential difference between the tool and the workpiece so that an electrochemical reaction arises, which removes material from the workpiece. The metal is detached atom by atom from the anode surface and appears in the electrolyte as ions (Fe^{2+}). These ions result in the precipitation of ferrous hydroxide $Fe(OH)_2$. Simultaneously, hydrolysis causes the water molecules to gain electrons from the cathode and they separate into free hydrogen gas and hydroxyl ion [22]. The reactions can be summarised in the following equations:

$$Fe \rightarrow Fe^{2+} + 2e^{-} \tag{2}$$

$$H_2O + 2e^{-} \rightarrow H_2 \uparrow + 2OH^{-} \tag{3}$$

$$Fe^{2+} + 2OH^{-} \rightarrow Fe(OH)_2 \tag{4}$$

3.1 Current confinement and surface roughness

In order to achieve precision in the machining the process must take place only under the tool tip, so that the cavity obtained in the workpiece is exactly the one determined by the profile of the tool. Therefore, current through the sides of the tool must be avoided, since it would remove material from other areas far from the tool tip. There are two methods of attaining this goal. The first one is isolating the side surface of the tool and using DC voltage as the process signal. The other one is using ultrashort voltage pulses and a very low IEG. The second method is used by several researchers [8, 11, 12] due to its ease of use if a function generator is available.

The confinement of the current can be assessed by observing the edge of the hole machined. If there is confinement, the contour of the hole will be sharp; otherwise the edge will be rounded. This phenomenon was studied by machining slots with

several values of pulse on-time and maintaining a constant voltage and period. By observing the size of the machined area, an assessment of the confinement of current can be achieved. The conditions for the experiments are shown in **Table 1**.

The average current is seen to decrease as the pulse on-time becomes lower, since the current only flows in the voltage pulse periods. A photograph of the holes machined in experiments R-01 to R-04 described in **Table 1** is presented in **Figure 5**. All the slots were machined with the same tool, which had a tip diameter of 10 μm. However, the width of the slot decreases with the pulse on-time from 150 to 70 μm approximately, as can be seen in the image. This is a consequence of the spreading of the current, which will be higher with an increased pulse on-time. In addition, the roundness of the edges can clearly be seen to be higher when the pulse on-time is augmented. A bright area can be observed around the slots, which suggests that the current also spread outside the hole and hence some material was removed from that area. It can therefore be deduced that when an active tool is used there is always a spreading of current outside the area under the tool tip, even if the edge of the hole is sharp. The holes machined with an isolated tool are shown in **Figure 6**. The current has clearly spread over a much smaller area since the diameter of the holes is much lower and there is no bright area around them.

Regarding surface roughness, the relationship between conditions and results are similar to those in mechanical machining, since a high MRR produces high surface roughness and vice versa. Therefore, a compromise must be achieved between surface roughness and process speed.

Experiment	IEG (μm)	Voltage (V)	Pulse width (ns)	Period (ns)	Average current (mA)
R-01	1	16	120	370	**26**
R-02	1	16	110	370	**22**
R-03	1	16	100	370	**16.1**
R-04	1	16	90	370	**10.3**

Table 1.
Conditions of the experiments for assessing current confinement and surface roughness.

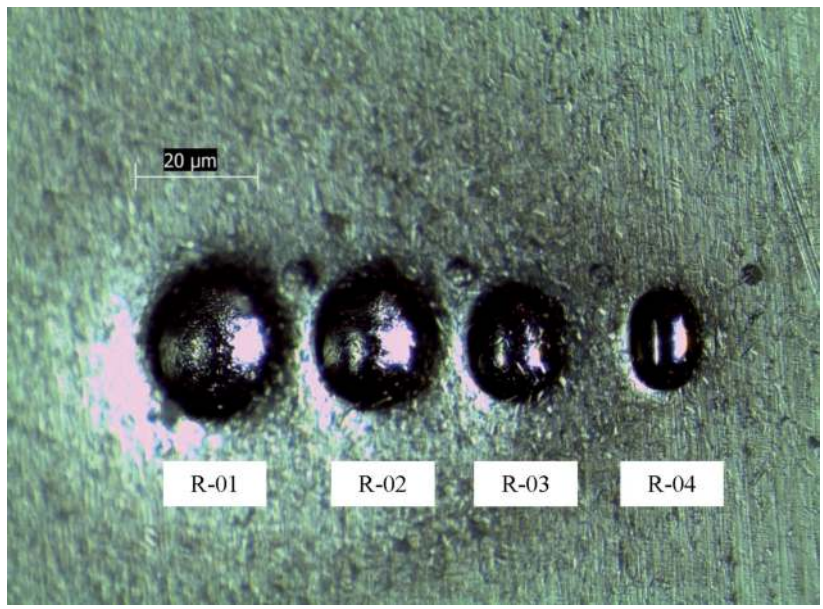

Figure 5.
Slots machined in experiments R-01 to R-04.

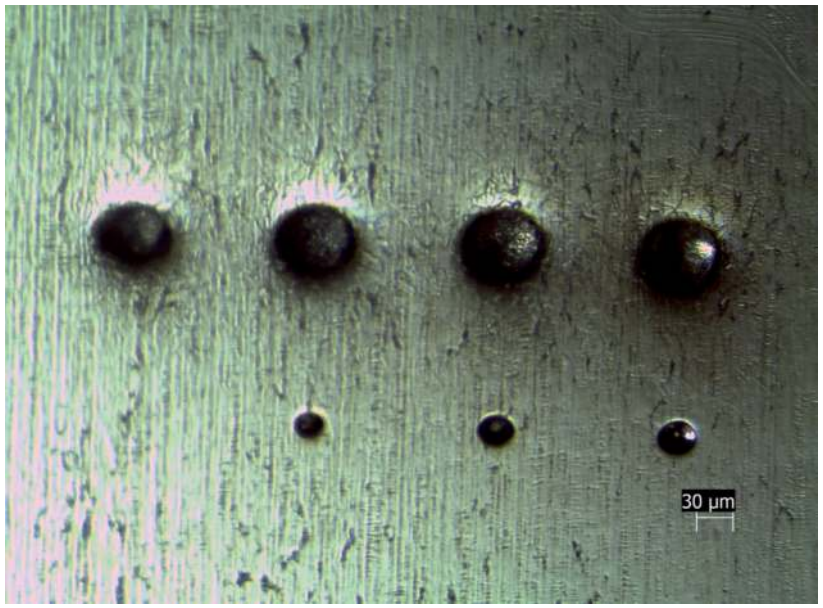

Figure 6.
Holes machined with an isolated tool.

Electrochemical machining has been observed to cause tiny craters in the workpiece surface, as a result of the localised current flowing through the electrolyte at the points of least electrical resistance. Therefore, if the current intensity is lower, the craters will be less deep and the resultant surface will be smoother. This can be seen clearly in **Figure 4**, which shows that the roughness is increasingly higher in the holes corresponding to R-04, R-03, R-02 and R-01, that is, when the pulse on-time grows. Therefore, it can be concluded that a good result is achieved by applying a voltage of 16 V and a pulse on-time of 80 ns and both confinement and surface roughness worsen when more aggressive values are used.

3.2 Material removal rate (MRR)

Material removal rate is a crucial variable in machining, since it determines the productivity of the process. This variable depends on the overpotential η, according to the principles of electrochemistry [9]. Therefore, the amplitude of the voltage signal determines the current intensity. Nevertheless, as the voltage signal applied to the cell consists of pulses, what determines MRR is the mean value of the current, according to Faraday's law of electrolysis:

$$MRR = \dot{m} = \frac{A \cdot I}{Z \cdot F} \tag{5}$$

where A is the gram atomic weight, Z is the valence of dissolution, F is Faraday's constant and I is the average current. In turn, the average current depends on the ratio between the period and the pulse on-time of the signal. Therefore, the main parameters which determine MRR are the pulse amplitude and the ratio between pulse on-time and period.

In order to determine the value of the parameters to attain a maximum of MRR several experiments were performed, setting the combination of parameters by means of an experiment design in which the voltage varied between 7 and 16 V and the pulse on-time from 50 to 80 ns, keeping the period constant at 370 ns. The output variable

considered was the current density, which provides more information regarding the performance of the process than the current intensity, as it takes the tool tip size into account. The results can be observed in **Figure 7**, which shows the variation of the average current density as a function of the voltage for every value of the pulse on-time.

As can be seen in the graph, from 7 to 11 V, there is a decrease in current density as the voltage increases. This is due to the passivation phenomenon which occurs on the stainless steel surface. At a value of 12 V, the current density increases dramatically and then remains approximately constant. The range beyond 12 V is therefore the transpassive area, in which the voltage of the tool is enough to dissolve the passive layer under the tool tip and to remove material locally. As the current density was calculated by dividing the total current by the area of the tool tip, the sudden increase in the current density in that area does not involve a significant increase in the current as whole. Therefore, the average current grows in a constant manner as the voltage increases, as can be seen in **Figure 8**.

This effect determines that, in order to achieve good machining without dispersion of the current, the voltage value must be high beyond the passive area of the stainless steel so that the MRR is maximum.

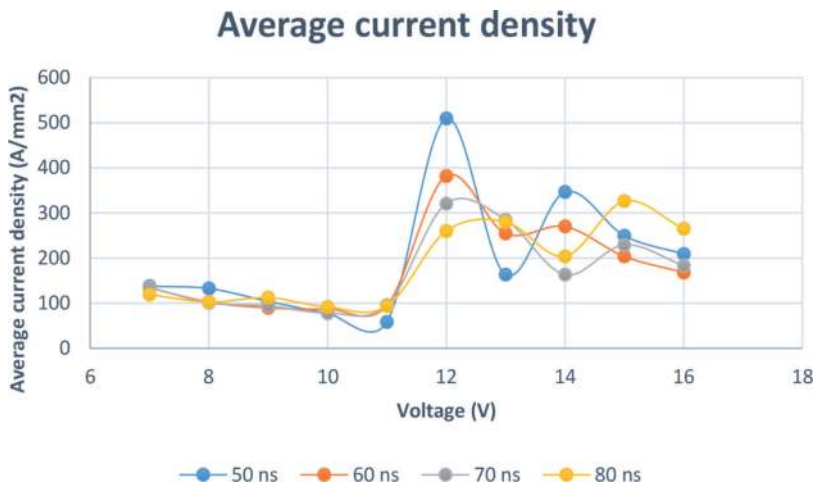

Figure 7.
Average current density as a function of voltage amplitude and pulse on-time.

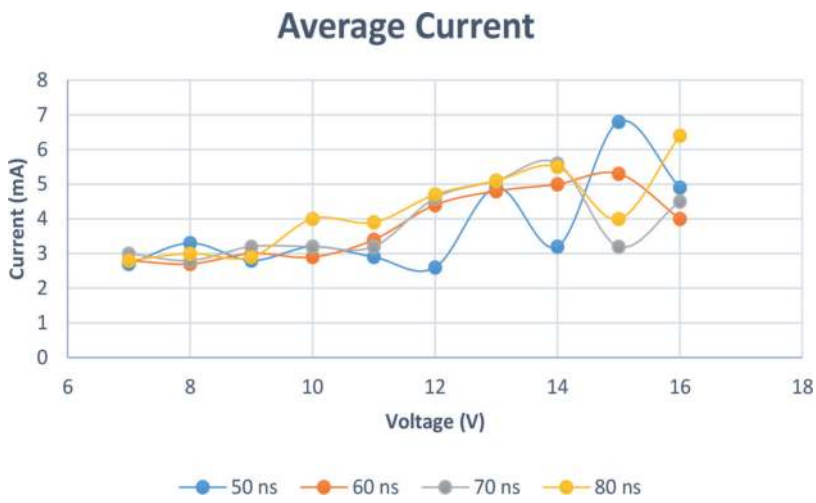

Figure 8.
Average current as a function of voltage amplitude and pulse on-time.

The method of assessing the real MRR is to observe the volume of material removed, which is determined by the geometry of the hole made in every experiment. The volume removed can be represented as a function of the voltage applied and the pulse on-time. These graphs are shown in **Figure 9**.

The graph shows that the volume removed increased with the voltage applied for every value of the pulse on-time. According to this tendency, the best value of the voltage to achieve a good MRR is the highest possible one. On the other hand, the increase is observed to be faster when the pulse on-time grows, so the value of this parameter should be as high as possible while maintaining the conditions of confinement. This graph can be presented in a 3D format in order to show the join effect of voltage and pulse on-time, as it can be seen in **Figure 10**.

3.3 Efficiency

The efficiency of the electrochemical machining can be obtained by comparing the theoretical value of material removed with the real one. The theoretical value is given by Faraday's law (5) and can be calculated from the current in the process.

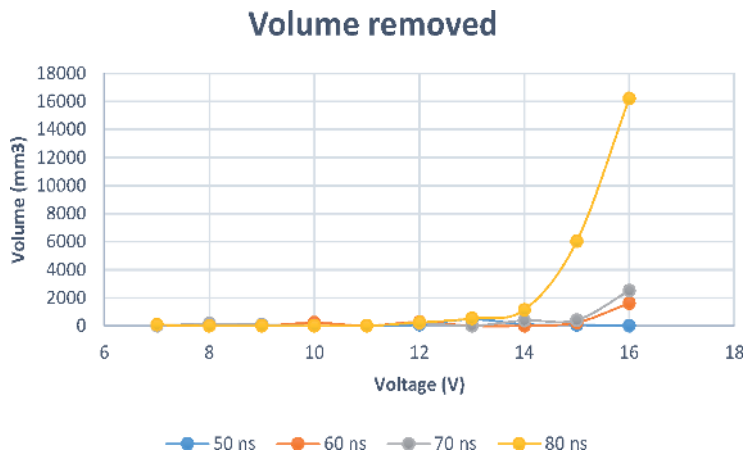

Figure 9.
Volume of material removed as a function of voltage amplitude and pulse on-time.

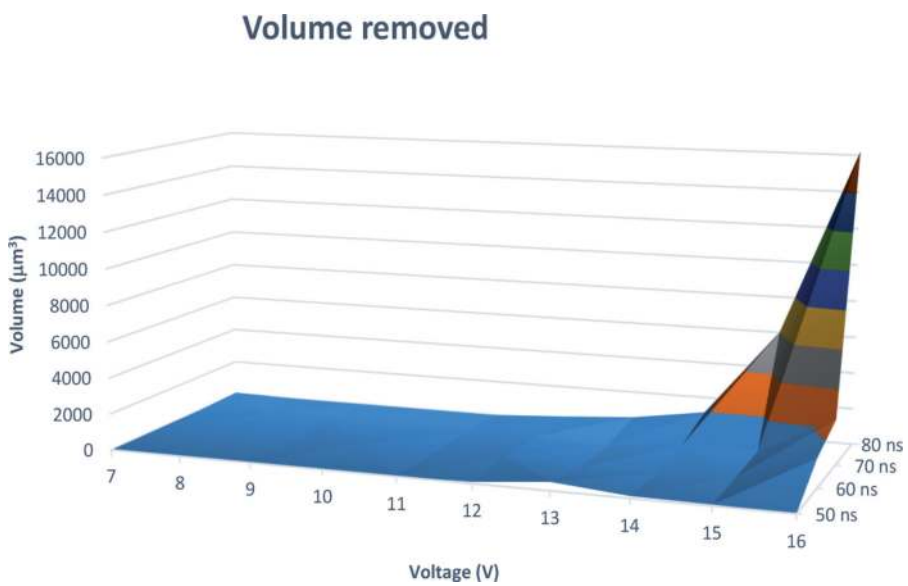

Figure 10.
3D representation of the volume removed as a function of voltage amplitude and pulse on-time.

The real value can be calculated from the geometry of the machined feature, as explained in Section 3.2.

This characteristic of the process is of great significance to the cost of the process, most of all, at an industrial level, and should be optimised by choosing the appropriate parameters.

In order to assess the efficiency of the process the results of the experiments made for observing the MRR were used. The ratio between the volume removed and the theoretical volume corresponding to the current was obtained and represented in **Figure 11**.

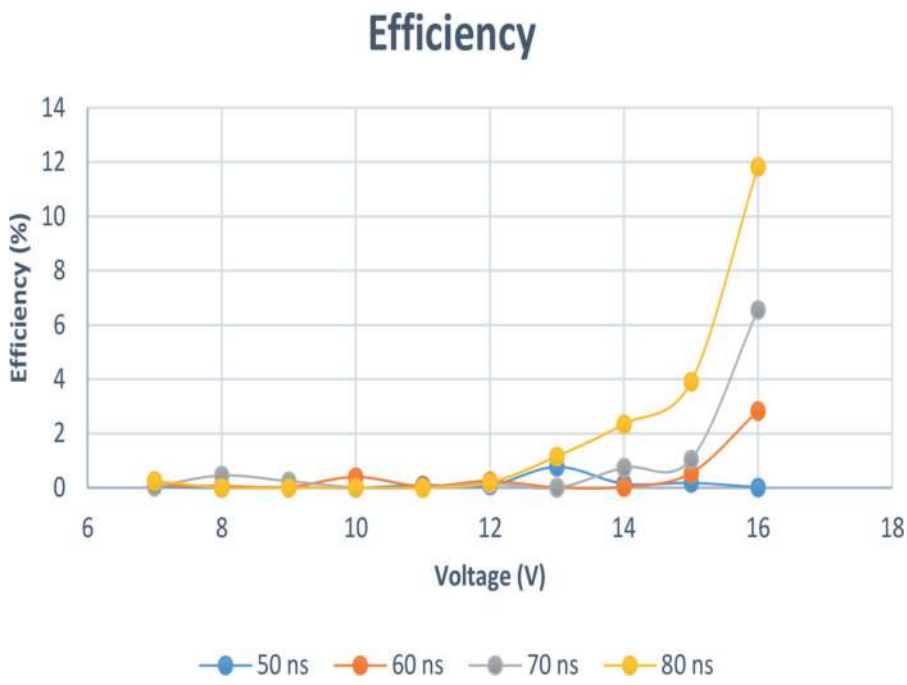

Figure 11.
Efficiency of the process as a function of voltage amplitude and pulse on-time.

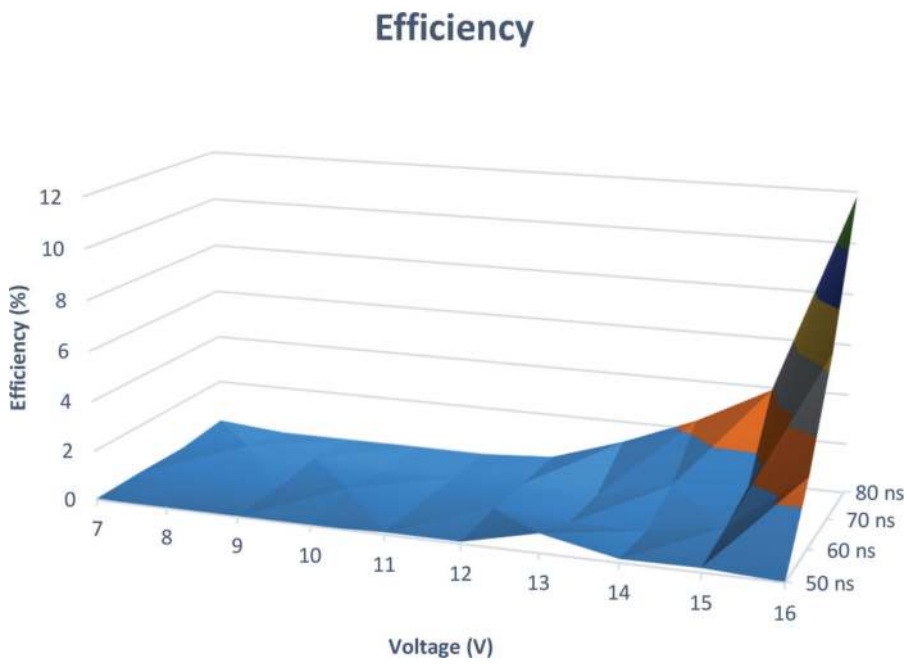

Figure 12.
3D representation of the efficiency of the process as a function of voltage amplitude and pulse on-time.

In these graphs, very low values of efficiency can be seen, because the maximum efficiency is lower than 12%. This is a consequence of the dispersion of the current, as the results presented in Section 3.1 show. According to the graphs, the higher the voltage and pulse on-time, the better the confinement of the current in the area under the tool tip and hence the higher the efficiency. The join effect of voltage and pulse on-time in efficiency is shown in the 3D of **Figure 12**.

These results can be analysed along with those presented in Section 3.2. Observing those graphs and **Figure 5** it can be deduced that the reason why the volume removed increases so drastically for voltage values higher than 14 V is not the increased current, but the clear increase in the efficiency of the process for those values. So, in order to achieve the best efficiency along with a good value of material removed, the highest possible value of voltage amplitude must be used along with the widest pulse maintaining the confinement and surface roughness within acceptable values.

4. Conclusions

A study of the optimum conditions for pulsed electrochemical micromachining of stainless steel has been presented. The equipment and the conditions for the process have been described. In order to find the optimum parameters for the process, the most important variables for the performance of the process have been taken into account. These variables were confinement, surface roughness, material removal rate and efficiency. Observing the results of the experiments, it can be stated that surface roughness increases with the pulse on-time of the voltage signal, whereas the confinement is better when the pulse on-time is lower. The passivation phenomenon takes place at voltage amplitude values lower than 12 V and disappears at higher voltages. The material removal rate is higher when both voltage amplitude and pulse on-time grow. The efficiency of the process is an important variable which increases with voltage amplitude and pulse on-time. Nevertheless, these variables must not be chosen beyond the limits of acceptable surface roughness and confinement. These limits have been set at 16 V and 80 ns, respectively, in this study.

Author details

Pablo Rodríguez*, Daniel Hidalgo and Julio Eduardo Labarga
Department of Manufacturing Engineering, Industrial Engineering Faculty, University of Leon, León, Spain

*Address all correspondence to: pablo.rodriguez@unileon.es

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