Article

Optimum parameters for Electrochemical Micromachining of Stainless Steel

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Abstract: In this work a research on pulsed electrochemical micromachining of Stainless Steel is presented. A suitable equipment to study the process is described as well as a fitting procedure to machine and measure the variables involved. A tool of Tungsten with a tip of about 5 µm diameter sunk in an electrolyte of $NaNO_3$ is used for the process. 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 made to assess the most important variables of the process, as current confinement, surface roughness, material removal rate and efficiency. It is observed that the current confinement get worse when the pulse on-time increases, as well as surface roughness. The material removal rate and the 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 disappears. There is a compromise in the choice of the variables, so a suitable combination of parameters is determined so that a good material removal rate with an acceptable result is achieved.

Keywords: Pulsed electrochemical micromachining; current confinement; material removal rate; efficiency

1. Introduction

Microfabrication consists in obtaining products or parts with features at micro or submicroscale, which therefore requires very narrowly controlled material removal. Microfabrication has been widely used for the manufacturing of holes in injectors, fluidic microchemical reactors requiring microscale pumps, micromoulds, and many more applications, as (1) describes. Microfabrication plays an increasingly important role in miniaturization of components which expand from biomedical applications to manufacturing of sensors. Surfaces to be obtained are slots, complex surfaces, microholes, etc. Frequently combinations of those features must be obtained in the industry of microelectronics. These parts are manufactured very often by conventional processes, with all the limitations and problems that these processes bring about, such as wear in the tool, inaccuracy due to low rigidity of the tool, heat generated by the process, etc. Even more problems may arise when manufacturing 3D microstructures, as some authors state (2). In this context, non-conventional processes, and especially electrochemical micromachining, acquire more importance due to their specific characteristics to avoid the problems of conventional processes.

From the early years of development of Electrochemistry, electrochemical methods have played an important role in precision technologies to machine structures and parts. In the 1950's Electrochemical Machining arose as a usual technique to manufacture complex geometries as blades of turbines, 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 microsystems (3). Electrochemical deposition techniques were used as a standard technology to deposit copper to obtain connections in high performance circuits and lithographic techniques LIGA are used to manufacture micromoulds (4,5).

Electrochemical micromachining has been a process with high specialization to be used in aerospace industry. Today it is starting to be used in other industries, where difficult to manufacture parts, complex surfaces and components in the microscopic scale is necessary to obtain. Electrochemical micromachining is today widely used for manufacturing semiconductor elements and thin metallic films (6).

In an analogous way to conventional Electrochemical Machining (ECM), Pulsed Electrochemical Micromachining (PECMM) is a controlled process of anodic dissolution to remove material which takes place at high current densities, typically in the order of 105 A/m² between the tool (cathode) and the workpiece (anode) through the electrolyte (7). By causing the tool to move towards the workpiece, the material is removed under its tip, since the current density is higher at lower distance between 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 small diameter or to obtain crack-free microcomponents and without residual stress. The use of ultrashort voltage pulses, usually shorter than 100 ns, allows achieving accuracy by confining the faradaic current density under the tool, since this current is the responsible for the anodic dissolution of the material. Confinement is 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 (8). When this phenomenon takes place, the voltage applied has to be over a threshold value to cause effective machining (9). It can also be avoided by adding acid to the electrolyte, as HCl or H_2SO_4, which are substances that dissolve the passive layer. This layer can be considered as an additional electrical resistance in the equivalent circuit which prevents the current to be confined under the tool tip (10). 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.

A very important advance has been made in the research of this process on many materials such as Aluminum, Titanium, Steel and Copper (11,12). Stainless Steel is a very important material to be used in any type of microcomponents, but its dissolution is difficult since its chemical properties are not very suitable for this process. Some of the existent studies were performed specifically on Stainless Steel (13–15). Nevertheless, the pulse on-time used in those works is too high to obtain a good confinement of the current. Furthermore, there are no studies in which the size of the tool is as small as a few microns. Therefore, there is a huge amount of work to do to characterize correctly this process regarding the values of the parameters in order to obtain a good result in terms of current confinement, surface roughness, material removal rate and experimental setup. In this work, a broad study 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, has been made.

2. Materials and Methods

The experiments performed for the study were made by means of an equipment that allows achieving accuracy and ease of handling tools and parts. 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 keeps the tool and the part from oscillations. The position of the recipient is controlled by a three-dimensional nanometric positioning system PI-Micos 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 electrolyte.

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The material of the workpiece is AISI 304 Stainless Steel and the tool is made of pure Tungsten at 99.7%. The tools are pins with a very small tip, of about 5 m in diameter. Figure 2 shows a picture of the cell with the tool and the workpiece. 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.

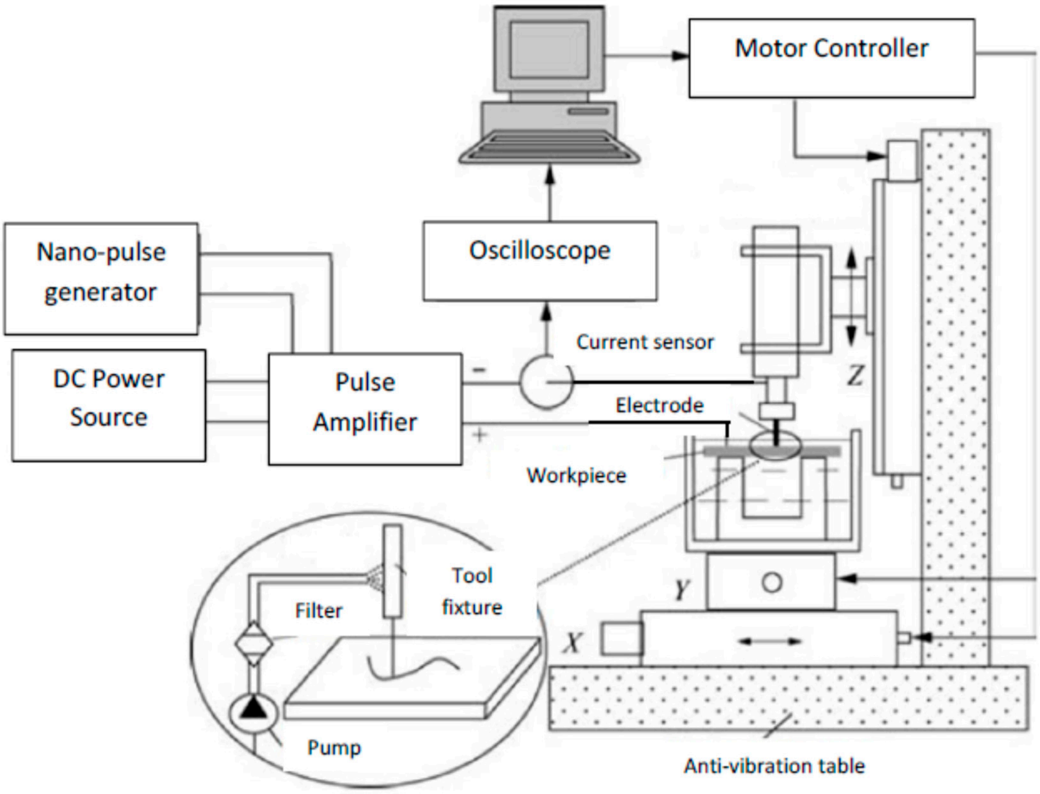

Figure 1. Sketch of the equipment used for experiments

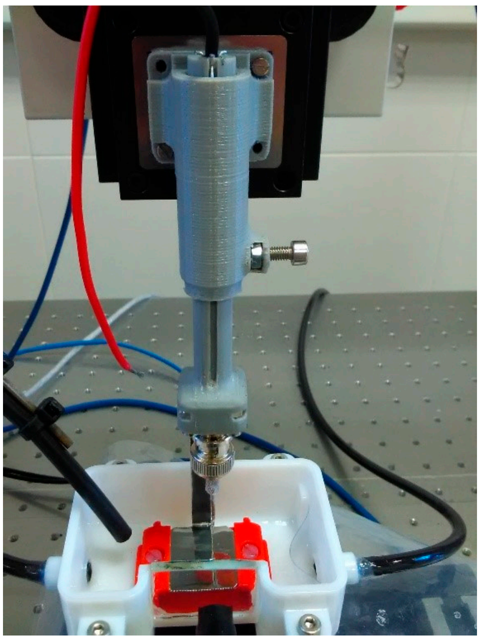

Figure 2. Electrochemical cell used for experiments

In order to apply the voltage pulses to the system, a Function Generator Agilent 33250 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 voltage pulses. The signal applied by the Generator passes through a 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. In Figure 3 the graphs of voltage and current between electrodes for a machining process are shown.

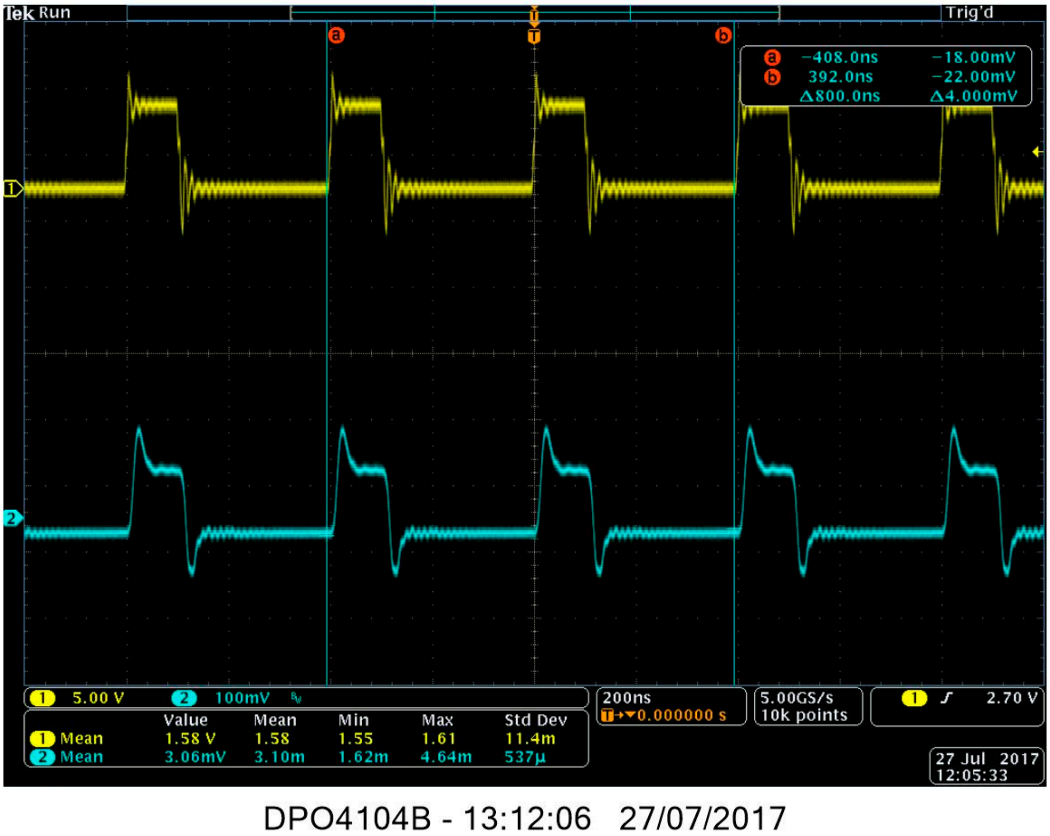

Figure 3. Signals of voltage and current between electrodes in the process of machining. Signal 1: Voltage (V), signal 2: current (mA)

The electrochemical process is observed by means of a Supereyes USB Portable Digital Miroscope B008 connected to a computer in which the amplified image of the tool and the area of the part being machined can be seen. This microscope is also helpful to set the approach the tool to the workpiece in order to set the reference of distance.

The voltage applied to the cell as well as the current that passes through it is measured by means of a digital oscilloscope Tektronic DPO 4104, which allows to visualize several signals with up to 3 GHz by using a maximum sample rate of 5 Gs/s. It also permits measuring mean values of signals, 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 of the position of the tool is taken in 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 the IEG decreases. However, when there is electrical contact, the current increases suddenly to a very high value. This phenomenon allows to find the reference 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 in applying a potential difference between the tool and the workpiece so that an electrochemical reaction arises that 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 give place to the precipitate of ferrous hydroxide $Fe(OH)_2$. Simultaneously, the hydrolysis causes the molecules of water gain electrons from the cathode and they separate into free hydrogen gas and hydroxyl ion (16). The reactions can be summarized in the following equations:

$$Fe \rightarrow Fe^{2+} + 2e^{-}, \quad (1)$$

$$H_2O + 2e^{-} \rightarrow H_2 \uparrow + 2OH^{-} \quad (2)$$

$$Fe^{2+} + 2OH^{-} \rightarrow Fe(OH)_2 \quad (3)$$

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 process signal. The other one is using ultrashort voltage pulses and a very low interelectrode gap (IEG). The second method is used by several researchers (8,11,12), since it is easier to use in case that 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 and otherwise the edge will be rounded. This phenomenon was studied by machining slots with several values of pulse on-time and maintaining constant the voltage and the period. By observing the size of the machined area, an assessment of the confinement of current can be achieved. In Table 1 the conditions for the experiments are shown.

Table 1. Conditions of the experiments for assessing current confinement

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
R-05	1	16	80	370	5.6

It can be observed that the average current decreases as the pulse on-time is lower, since the current only flows in the voltage pulse periods. In Figure 4 a photograph of the holes machined in experiments R-01 to R-04 is presented. 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 µm to 70 µm approximately, as it can be seen in the image. This is a consequence of the spreading of the current, which will be higher when the pulse on-time increases. In addition, it can be seen clearly that the roundness of the edges is higher when the pulse on-time grows.

Figure 5 shows the slot machined in experiment R-05, in which the pulse on-time was lowest. It can be observed in the figure that there is an area around the edge which seems worn. This fact suggests that the current was spread also outside the hole and hence some material was removed from that area. Therefore, it can be deduced that there is always a spreading of current outside the area under the tool tip, even if the edges are sharp.

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

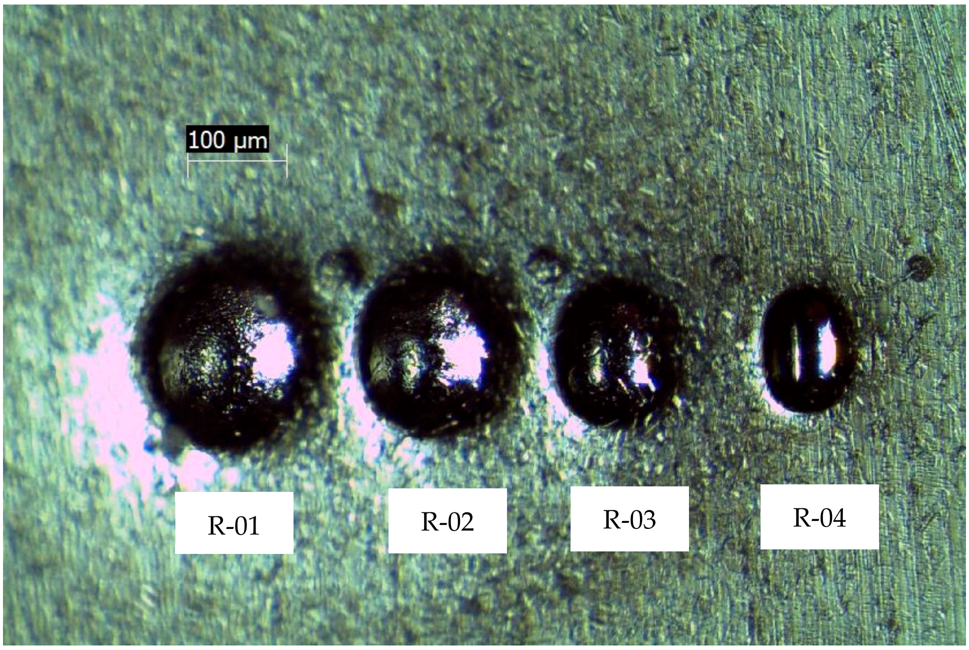

Figure 4. Slots machined in the experiments R-01 to R-04

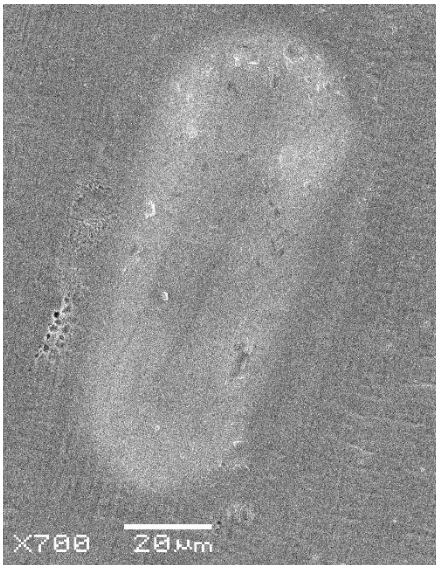

Figure 5. Slot machined in experiment R-05

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

It is observed that the electrochemical machining causes tiny craters in the workpiece surface, as a result of the localized current that flows 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 fact is clearly observed in Figure 5, which shows that the bottom surface of the slot is smoothest in that corresponding to R-04 and the roughness is increasingly higher

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in R-03, R-02 and R-01, i.e. when the pulse on-time grows. Therefore it can be concluded that a good result is achieved applying a voltage of 16 V and a pulse on-time of 80 ns and both confinement and surface roughness get worse when more aggressive values are used.

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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 ϕ, according to the Butler-Volmer equation:

$$i = i_0\left[e^{(1-\beta)\eta F/RT} - e^{-\beta\eta F/RT}\right], \quad (4)$$

where i is the current density, i_0 the equilibrium exchange current density, β the symmetry factor of the reaction, F Faraday's constant (96500 C), ϕ the overpotential, R the ideal gas constant and T the temperature in K. So, the amplitude of the voltage signal determines the current intensity. Nevertheless, as the voltage signal applied to the cell consists in pulses, what determines MRR is the mean value of the current, according to Faraday's law of electrolysis:

$$MRR = \dot{m} = \frac{A \cdot I}{Z \cdot F}, \quad (5)$$

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 the 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 vary 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 6, which shows the variation of the average current density as a function of the voltage for every value of the pulse on-time.

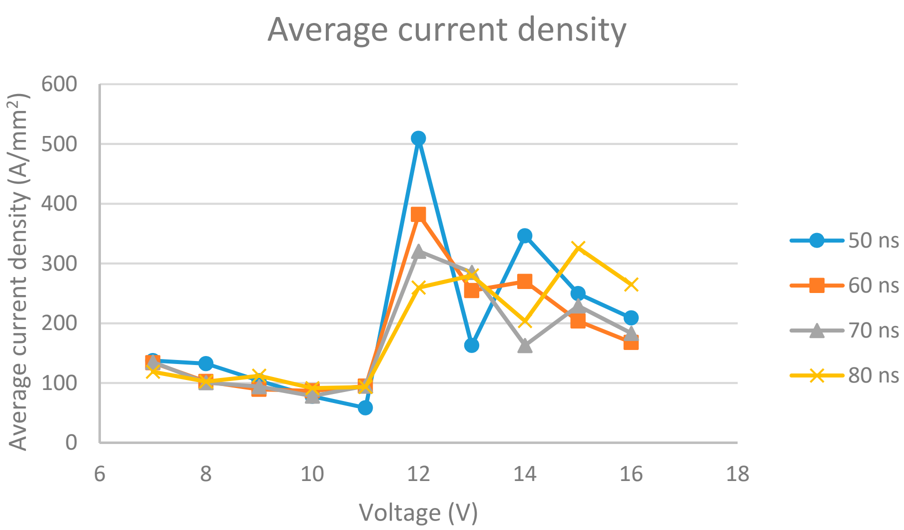

Figure 6. Average current density as a function of voltage amplitude and pulse on-time

As it can be seen in the graph, from 7 to 11 V there is a decrease in the current density as the voltage increases. This is due to the passivation phenomenon that occurs on the stainless steel surface. At the value of 12 V, the current density increases dramatically and then it remains approximately constant. This means that the range beyond 12 V is 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 of 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 way as the voltage increases, as it can be seen in Figure 7.

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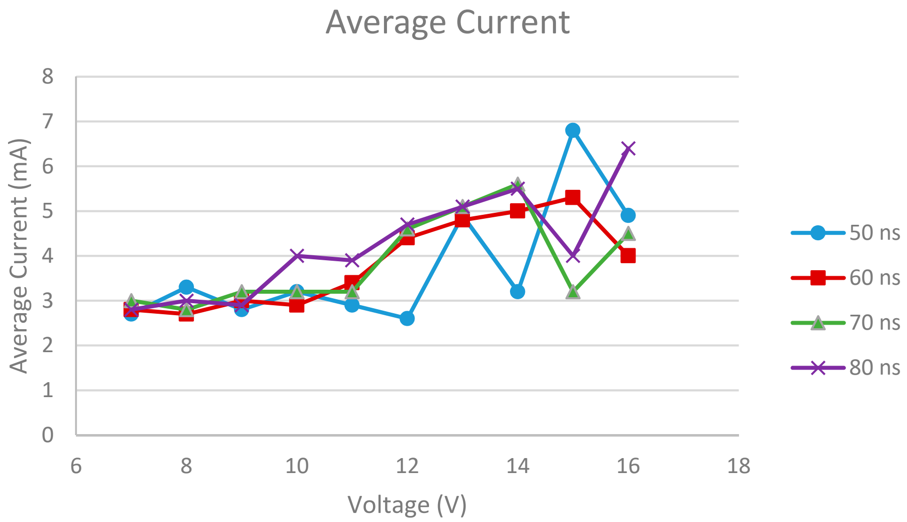

Figure 7. Average current as a function of voltage amplitude and pulse on-time

This effect determines that, in order to carry out a 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.

Another way of assessing the MRR is to observe the volume of material removed, which is determined by the geometry of the hole made in every experiment. Due to the tapering side walls of the hole and the rounded tip of the tool, the geometry of the hole can be assumed to be a cone with a rounded tip. By knowing the depth, the edge radius of the tool tip and the diameter of the hole in the surface, the real volume of material removed can be calculated.

The volume removed can be represented as a function of the voltage applied and the pulse on-time. These graphs are shown in Figure 8.

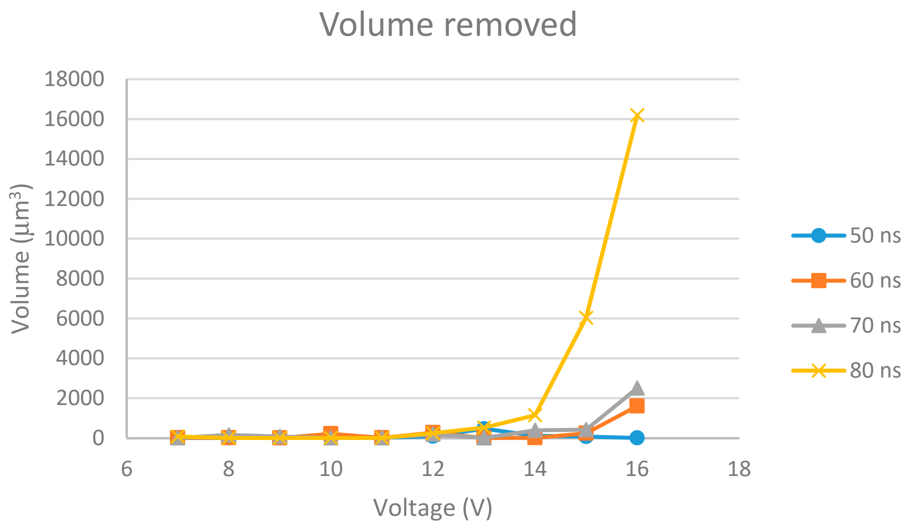

Figure 8. Volume removed as a function of voltage amplitude and pulse on-time

It can be observed in the graph that the volume removed increase 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. On the other hand, it can be seen that the increase is faster when the pulse on-time grows, so the value of this parameter should be as high as possible, keeping the conditions of confinement.

3.3. Formatting of Mathematical Components

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 (1) and can be obtained from the current in the process. The real value can be calculated from the geometry of the machined feature, as it was explained in section 3.2.

This characteristic of the process has a great importance for the economy of the process, most of all, at industrial level, and it should be optimized by choosing the fitting 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 9.

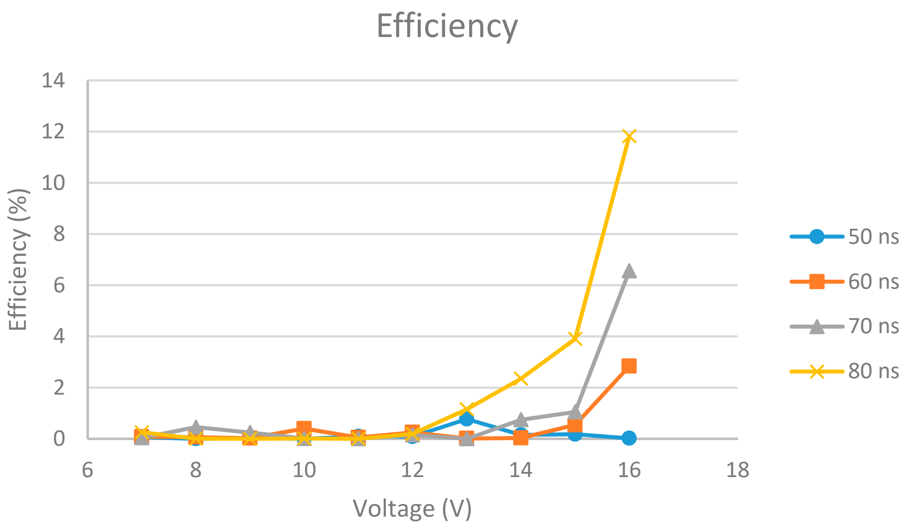

Figure 9. Efficiency of the process as a function of voltage amplitude and pulse on-time

In these graphs very low values of efficiency can be seen, because the maximum efficiency is lower than 12%. This fact is a consequence of the dispersion of the current, as the results presented in section 3.1 show. According to the graphs, the more the voltage and pulse on-time the more the confinement of the current in the area under the tool tip and hence a higher efficiency is attained.

These results can be analyzed 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 values of voltage higher than 14 V is not the increase of the current, but the clear increase in the efficiency of the process for those values of voltage. 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 that maintains the confinement and surface roughness within acceptable values.

5. 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. This variables were confinement, surface roughness, material removal rate and efficiency. Observing the results of the experiments it can be stated that surface roughness increase 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 it disappear 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 at which the surface roughness and the confinement are not acceptable. In this study, this limits have been set at 16 V and 80 ns respectively.

Author Contributions: The research was carried out by the three authors mentioned in the heading. The equipment set up, simulations and programming was done by D.H. The data analysis and the writing of the article was done by P.R. The supervision of the research was carried out by J.E.L.

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