

DYNAMICS OF THE PLASMA ELECTROLYTIC POLISHING PROCESS OF AUSTENITIC STEEL AISI 304 IN A SOLUTION OF AMMONIUM SULFATE

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Modern industrial technological processes together with scientific research activities require the special preparation of the metal surfaces for the further treatments. At present time the variety of techniques for the metal surface processing exist. Among them one could outline the plasma electrolytic polishing (PEP) due to its capability together with the decoration effect to enhance the surface properties. Despite the number of papers published in different journals the PEP stays an innovative surface treatment and requires further study to obtain optimal processing regimes. In present contribution the experimental study of PEP process dynamics with further numerical analysis of physical aspects for steel AISI 304 in a solution of ammonium sulfate with the mass fraction of 3% is given. After analyzing the experimental results the optimal PEP regime for the steel AISI 304 in a solution of ammonium sulfate with the mass fraction of 3% was identified.

INTRODUCTION

A large number of industrial technological processes as well as scientific research activities need the special preparation of the metal surfaces for further treatments. That preparation includes dimensional processing, cleaning of the surface from contaminations that were brought during the previous technological processes, polishing, etching, etc. At present time the variety of metal surface treatment techniques exist [1]. Among them one could outline the plasma electrolytic processes [2–5], especially the PEP due to its capability together with decoration effect to enhance the surface properties [6]. The other advantages of PEP are the ecological cleanliness, relatively low power consumption and low time consumption for the surface modification, simple automatization of the process, low price of the device together with low main expenses. Despite the number of papers published in different journals the PEP is an innovative surface treatment [5] that highly needs to be studied in details in order to improve our understanding of the physical processes standing behind it. That will be helpful in optimization of PEP process by itself and in developing of a new surface modification technologies.

In present paper the study of the physical aspects of PEP process dynamics of steel AISI 304 [7, 8] in a solution of ammonium sulfate is given. Such kind of steel is widely used to manufacture the parts of high-technology equipment, for food and chemical industries. The other application of AISI 304 is a decorate-constructional elements for consumer goods.

To perform the experiment the standard scheme of plasma electrolytic processes device is chosen [1, 5, 6, 9]. In this scheme the workpiece put inside the reservoir plays the role of anode and the body of the reservoir filled with the electrolyte becomes a cathode. In our case the electrolyte is the solution of ammonium sulfate [9, 10] with the mass fraction of 3%. This percentage is a threshold value, starting with that the valuable

polishing effect is observed. One of the important point is to chose the optimal startup scenario for the polishing process in order to avoid gas liberation and spark ignition regimes [1, 11] and to pass directly to the continuous plasma envelope regime. To do that the power was turned on before the sample was immersed the electrolyte that guaranteed the preliminary electric potential difference between the sample and the electrolyte. After the rated voltage was reached the immersing system places the sample in the reservoir.

In order to cover all tasks of interest the experiment was splitted in two campaigns. In the first campaign the workpieces were processed one by one during 600 s each. During this experiment the strong correlation between the current drop on a sample and rising of electrolyte temperature was observed. The numerical analysis of the experimental data showed that the linear correlation coefficient called Pearson correlation coefficient has the value of -0.97 that is associated with very high correlation.

The correlated current density dependence on electrolyte temperature allows to present three different regimes of polishing. The first regime is low temperature regime with high current value with large fluctuations that reach 50% from mean current value. With the increasing of the temperature the process passes to the second regime – the transition phase. The rapid current drop is observed and the fluctuation of the current is reduced to the 25...30% from the average value. With further increasing of the temperature the process passes to the third regime with the significant current drop and reduced value of current fluctuations around 10%. In the last regime the continuous plasma envelope surrounding the sample is observed. These regimes are studied in details and described in the chapter with the experimental results.

During the second campaign a separate sample was processed repeatedly during 30 s each time in the third polishing regime. It was done on purpose to observe the modification of the surface during the polishing. After

each 30 s process the sample was removed from the electrolyte and the detailed study of the surface including photographing was done. After the analysis of the experimental results from both campaigns the third polishing regime is pointed as the optimal regime for the steel AISI 304 in a solution of ammonium sulfate with the mass fraction of 3%.

1. EXPERIMENT

1.1. EXPERIMENTAL SETUP

The experiment was performed on a standard PEP setup [1, 5, 6, 9] that consists of the plastic reservoir with the technical window, which provides the direct access to the workpiece handling system connected to the immersion system, as shown in Fig. 1.

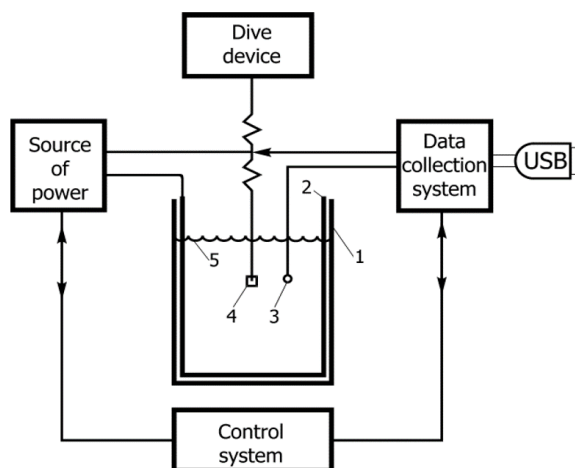


Fig. 1. The principal scheme of the experimental PEP setup: 1 – cathode connected to DC power supplier; 2 – the plastic reservoir with the technical window for direct access to the detectors 3; 4 – workpiece; 5 – the reservoir is filled with the solution of ammonium sulfate

The power supplier is actually the process energy DC source connected by wires to anode and cathode. The process control system together with measurement and data acquisition systems are installed as well. The reservoir is filled with the solution of ammonium sulfate [9, 10] $[(\text{NH}_4)_2\text{SO}_4]$ with concentration of 31 g/l. The volume of reservoir is 60 l and the size of cathode is $3 \cdot 10^3 \text{ cm}^2$. The immersion system allows to move the workpiece automatically to the necessary depth with the speed of 5 mm/s. The power supplier is based on a classical scheme of the three-phase Larionov bridge (controlled three-phase full-wave bridge) [12] with galvanic isolation by a three-phase transformer. The DC output voltage is filtered by capacitors with a total capacitance of 1200 μF . The no-load voltage is 280 V and the output resistance is 0.18 Ω . The power supplier is equipped with a short-circuit protection for emergency case when the current exceeds 100 A. The process control system provides the prescribed operation regime of the setup, controls and indicates the current operation parameters. The measurement and data acquisition systems allow to control the voltage and the current on workpiece together with the temperature of electrolyte around the process area. The accuracy of measurements for voltage and current is 0.5%, and for

the temperature is 1%. The measurement is performed each 20 ms and the data transforms automatically to the computer that monitors the total process.

1.2. WORKPIECES

In the framework of current experimental campaigns the plane workpieces made of austenitic steel AISI 304 [7, 8] of rectangular shape were used. The size of each sample was chosen 22x50x2 mm in order to make its area much less than the area of cathode and to minimize the edge effects.

1.3. EXPERIMENT PERFORMANCE

At the first step of the experiment the workpiece described above was fixed on a handle system connected with the immersion system. The crucial point at this step is to guarantee the stable electrical contact of the sample with the power supplier. After that, while the sample is not immersed in the electrolyte, the power was turned on in order to provide the preliminary electric potential difference between the workpiece and electrolyte. After the rated voltage is reached on a filtering capacitors the immersing system places the sample on a necessary depth in the reservoir. Such startup allows to avoid gas liberation and spark ignition regimes and to pass directly to continuous plasma envelope regime according to the conventional current-voltage diagram for PEP [1], as shown in Fig. 2.

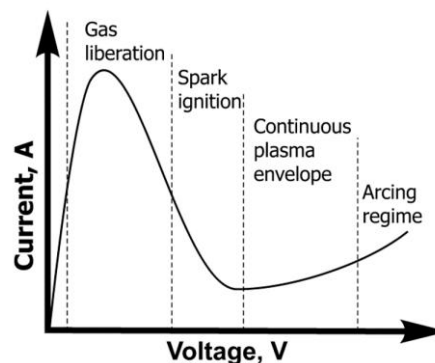


Fig. 2. Conventional voltage-current characteristics of the PEP process

The immersing depth of the sample was 25 mm and was keeping the same for each experiment.

Two experimental campaigns were performed. In the first campaign five workpieces were processed one by one during 600 s each. The long processing time was chosen to avoid the effect coming from the finite time of sample immersing to the rated depth. The immersing time is 5 s that is two orders less than the processing time.

During the second campaign each sample was processed repeatedly by 30 s each time. It was done on purpose to observe the modification of the surface during the polishing. After each 30 s process the sample was removed from the electrolyte and the detailed study of the surface including photographing was done.

2. EXPERIMENTAL RESULTS

As far as during the first experimental campaign the samples were polished one by one the temperature of

electrolyte was continuously increasing. As one can see in the Fig. 3, the first workpiece was immersed in the electrolyte with the temperature of $T_{1s} = 22\text{ }^{\circ}\text{C}$. After the first 600 s of processing the temperature of electrolyte was reaching the value higher than $63\text{ }^{\circ}\text{C}$ and the polishing of the next sample was starting at the temperature $T_{2s} = 63.2\text{ }^{\circ}\text{C}$. The process starting temperatures for other three samples were $T_{3s} = 70.4\text{ }^{\circ}\text{C}$, $T_{4s} = 72.8\text{ }^{\circ}\text{C}$, and $T_{5s} = 74\text{ }^{\circ}\text{C}$ respectively. The power released in the reservoir was spent to the electrolyte heating. Hence the temperature regime of the process was changing as well. As the result the characteristics of the process were modified that is reflecting in the time dependence of the anode current drop as is presented in Fig. 4.

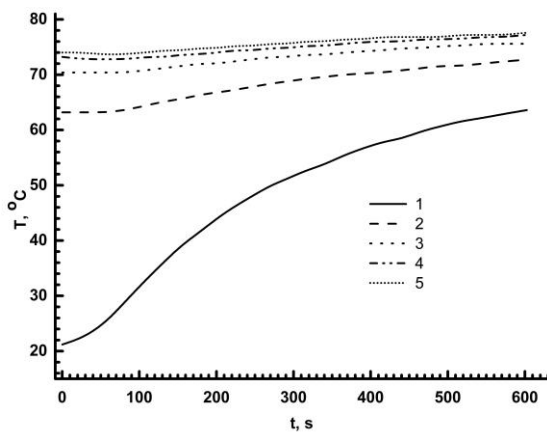


Fig. 3. Rising electrolyte temperature during the first experimental campaign for five workpieces

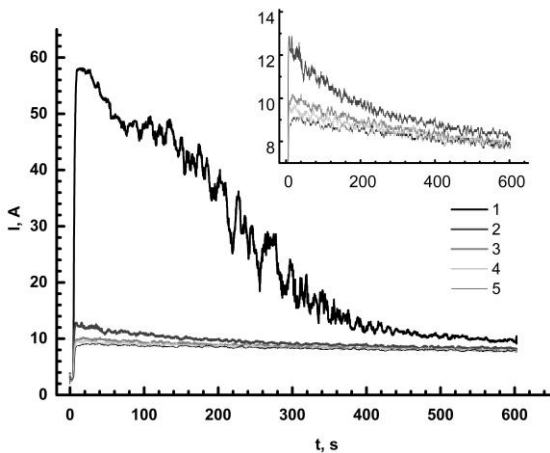


Fig. 4. Anode current drop during the first experimental campaign for five workpieces

As one can see from the dependencies, the beginning values of the anode current for each sample are decreasing together with the decreasing of running current value during the polishing processes. That is related with the flattening of the workpiece surface leading to the reducing of the effective surface area, and with the increasing of the electrolyte temperature. One can observe the correlation between increasing of electrolyte temperature and decreasing of current density. The linear correlation coefficient known as Pearson's product-moment coefficient or as it is sometimes called Pearson correlation coefficient [13] has the value of -0.97 that is associated with very high

correlation. In Fig. 5 the correlated current density dependence on electrolyte temperature is presented.

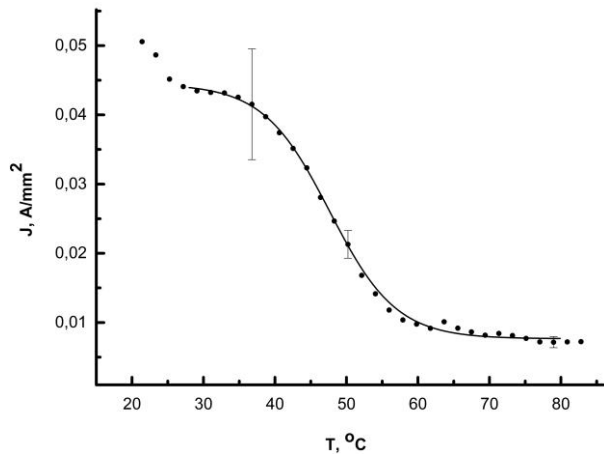


Fig. 5. The correlated current density dependence on electrolyte temperature for five samples processed one after another during 600 s each. The error bars are displayed for several points in order to avoid puzzling the graph

The presented data in the interval from $28\text{ }^{\circ}\text{C}$ till $80\text{ }^{\circ}\text{C}$ is well fitted by the following analytical expression:

$$y(x) = A_2 + \frac{A_1 - A_2}{1 + \exp\left(\frac{-x - x_0}{\sigma}\right)}, \quad (1)$$

where A_1, A_2, σ and x_0 are fitting coefficients that have the values $0.045, 0.008\text{ A/mm}^2; 4.6$ and $47.6\text{ }^{\circ}\text{C}$ respectively. This reflects the fact that with the temperature increasing the physical and chemical properties of the electrolyte are changing, and for the given experimental conditions the current density asymptotically approaches to the finite value A_2 .

In this graph, one can select several areas. The first area is the area of high current densities that could be found between 20 and $35\text{ }^{\circ}\text{C}$. Within this temperature interval high current fluctuations that reach 50% from average current value are observed. Visually the process looks like the gas bubbles formation that occurs due to gas liberation. At the same time the continuous plasma envelope surrounding the sample does not appear at this stage. With the increasing of the temperature the process passes to the second stage – the transition phase between 35 and $60\text{ }^{\circ}\text{C}$. The fluctuation of the current is reduced to the 25...30% from the average current value. Starting from $60\text{ }^{\circ}\text{C}$ the third phase starts. The average current value in this area is five times lower than in the first area and the fluctuations are around 10% from the average value. Visually the continuous plasma envelope surrounding the sample completely is observed.

During the PEP process the loss of sample weight occurs. For example for the workpiece immersed in the electrolyte with temperature of $21.2\text{ }^{\circ}\text{C}$ the loss of the weight is $(0.9 \pm 0.01)\text{ g}$, and with the increasing of the initial temperature of electrolyte the decreasing of the weight loss is observed. Hence for the samples immersed in the electrolyte with the initial temperatures 72.8 and $73.6\text{ }^{\circ}\text{C}$ the weight loss was $(0.3 \pm 0.01)\text{ g}$. At the same time the correlation between the input power

and total exhausted energy in the electrolyte volume during the processing is observed. That correlation is presented in Table. Here in the second column T_s is the temperature of the electrolyte at the beginning of the polishing of corresponding sample and T_f is the temperature at the finishing of polishing. As one can see from the presented data with the increase of the

electrolyte temperature the current drop is observed and as a result the decreasing of mean input power and mass loss. From the data presented in the table one could conclude that the most effective polishing process, from the point of view of minimum mean input power rate, starts from the electrolyte temperature of 70 °C.

Correlation between PEP parameters during continuous processing of five samples

Sample number	T_{s_s} , °C	T_f , °C	Mass loss, g	Mean electrolyte temperature T_m , °C	Total exhausted energy Q , MJ	Mean input power P , W
1	21.2	63.6	0.91	48	4.31	7160
2	62.8	72.8	0.41	68.3	1.63	2700
3	70.4	76	0.34	73.2	1.50	2500
4	72.8	77.2	0.3	74.9	1.47	2400
5	73.6	77.6	0.3	75.7	1.44	2400

Furthermore to provide the visual test of the polishing surface the extra sample was removed from the electrolyte after each polishing regime and was investigated. Optical electron microscopy was carried out using the METAM P-1. On Fig. 6 one can see the corresponding evolution of the workpiece surface after each of three polishing regimes. The granular structure of the surface changes and has a significant modification after the third regime.

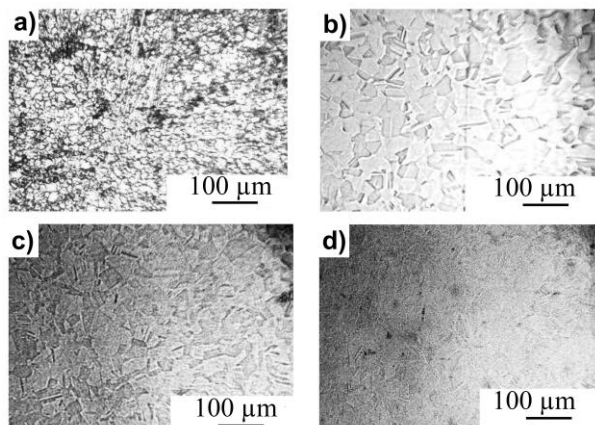


Fig. 6. The modification of the workpiece surface during the processes that passes through all three polishing regimes presented on Fig. 5: a – the beginning shape of the surface; b – the surface after the first regime; c – after the second regime, and d – after the third regime

The second experimental campaign was undertaken for the small variation of the electrolyte temperature that is related to the third polishing regime according to the Fig. 5. The polishing process was interrupted every 30 s in order to extract the sample and to analyze the roughness of the surface [14, 15]. On Fig. 7 the modification of surface of the workpiece is presented for different time values. On presented photos at the edges the depth of the surface roughness is well observed. It appears due to optical characteristics of the microscope and small area of illumination of the sample. A qualitative characteristic allows one to judge

microroughness along grain boundaries on sample surfaces.

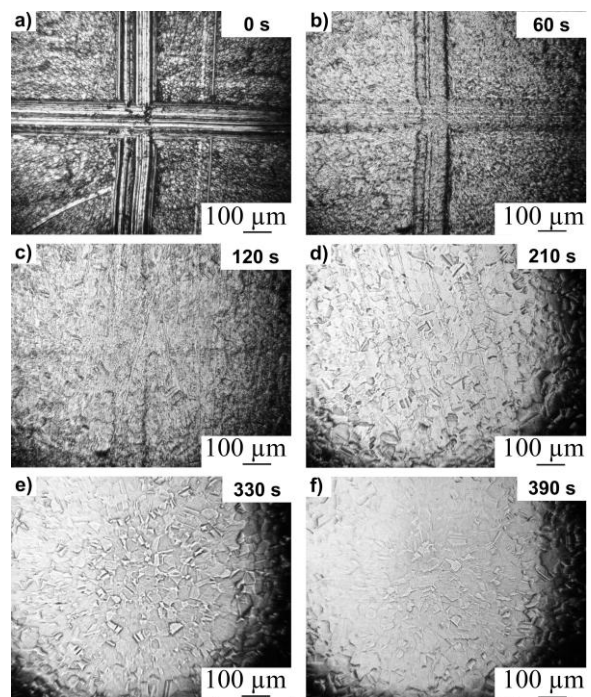


Fig. 7. The modification of the workpiece surface during the processes in third polishing regime: a – the beginning shape of the surface; b – the surface after 60 s of polishing; c – after 120 s of polishing; d – after 210 s of polishing; e – after 330 s of polishing, and f – after 390 s of polishing that is the end of the process

Before the polishing process the cross mark of the 20 μm depth was made on a sample surface. As one can see from the photos after 210 s of polishing the mark vanishes. After 390 s of process the roughness of the sample surface was not modified, that is considered as the end of polishing process. Fig. 8 shows the surface of the sample before (curve 1) and after the processing (curve 2). Studies of the surface profile were carried out using a multifunctional device “Mikron-gamma” [16]. Curve 2 corresponds to the end of the polishing process

since the further process of PEP does not lead to a qualitative and quantitative change in the characteristics of the surface profile. The surface roughness parameters do not change after 390 s. Thus, further processing only leads to the removal of the material of the workpiece without reducing the level of roughness of its surface. That is considered as the end of polishing process.

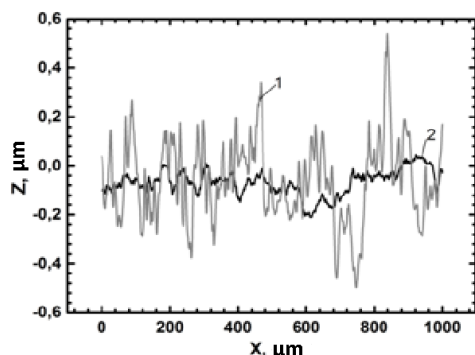


Fig. 8. Comparison of the workpiece surface roughness at the beginning of the polishing (1 curve) in third regime and in the end of the process (2 curve). The presented profilogram is related to the process presented on Fig. 7

CONCLUSIONS

Two series of experiments made it possible to perform the tasks set for determining the optimum regimes of electrolytic-plasma modification of the surface of AISI 304 steel in an ammonium sulfate solution of 3%, depending on the temperature of the electrolyte and the processing time. A strong correlation was found between the current flow on the sample and the temperature of the electrolyte. This correlation made it possible to present three different polishing regimes. Numerical analysis of the experimental data has shown that the most efficient process of energy consumption begins with a temperature exceeding 70 °C, which corresponds to the polishing conditions introduced in [1].

But it should be noted that in our experimental studies the polishing regime begins with a temperature above 35 °C. The second experimental campaign was performed to observe the dynamics of the sample surface change from the polishing time, and showed that after 390 s, further surface treatment does not lead to qualitative and quantitative changes in the relief. After analyzing the experimental results of both campaigns, the third polishing regime is indicated as the optimum mode for steel AISI 304 in a solution of ammonium sulfate with a mass fraction of 3%, a processing time of not more than 390 s and a temperature of more than 60 °C.

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ДИНАМИКА ПЛАЗМЕННОГО ЭЛЕКТРОЛИТНОГО ПРОЦЕССА ПОЛИРОВАНИЯ АУСТЕНИТНОЙ СТАЛИ AISI 304 В РАСТВОРЕ СУЛЬФАТА АММОНИЯ

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Современные производственно-технологические процессы вместе с научно-исследовательской деятельностью требуют специальной подготовки металлических поверхностей для дальнейшей обработки. В настоящее время существует множество методов обработки поверхности металла. Среди них можно выделить плазменную электролитическую полировку (РЕР) из-за ее способности улучшения свойств поверхности с эффектом полировки. Несмотря на число публикаций в разных журналах, РЕР остается инновационным методом обработки поверхности и требует дальнейшего изучения для получения оптимальных режимов полирования. В данной статье представлено экспериментальное исследование динамики процесса РЕР с дальнейшим численным анализом физических свойств стали AISI 304 в растворе сульфата аммония с массовой долей 3%. После анализа экспериментальных результатов был определен оптимальный режим РЕР для стали AISI 304 в растворе сульфата аммония с массовой долей 3%.

ДИНАМІКА ПЛАЗМОВОГО ЕЛЕКТРОЛІТНОГО ПРОЦЕСУ ПОЛІРУВАННЯ АУСТЕНІТНОЇ СТАЛІ AISI 304 У РОЗЧИНІ СУЛЬФАТУ АМОНІЮ

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Сучасні виробнично-технологічні процеси разом з науково-дослідницькою діяльністю вимагають спеціальної підготовки металевих поверхонь для подальшої обробки. На цей час існує безліч методів обробки поверхні металу. Серед них можна виділити плазмове електролітичне полірування (РЕР) через його здатність покращення властивостей поверхні з ефектом полірування. Незважаючи на число публікацій в різних журналах, РЕР залишається інноваційним методом обробки поверхні та потребує подальшого вивчення для отримання оптимальних параметрів полірування. У цій статті представлено експериментальне дослідження динаміки процесу РЕР з подальшим чисельним аналізом фізичних властивостей сталі AISI 304 у розчині сульфату амонію з масовою часткою 3%. Після аналізу експериментальних результатів було визначено оптимальний режим РЕР для сталі AISI 304 у розчині сульфату амонію з масовою часткою 3%.