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Diagnostics of the PEO-coating growth on a zirconium alloy by the acoustic spectrum

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Abstract. Plasma-electrolytic oxidation is accompanied by microdischarges, which determine the main mechanism of coating growth. These microdischarges, along with electrolyte boiling, generate acoustic vibrations that are closely related to the coating growth process. In this paper, authors investigated the acoustic characteristics and surface properties of a zirconium alloy during plasma-electrolytic oxidation, specifically focusing on the constant voltage mode. The study identified informative ranges within the acoustic spectrum that corresponded to two distinct stages of microdischarge development. Furthermore, regression models were created to establish a strong correlation between the increase in coating thickness and changes in current density and peak amplitudes within the acoustic spectrum. The acoustic spectrum ranges of 4000-5000 Hz and 17000-18000 Hz were found to be the most informative for coating thickness diagnostic.

1 Introduction

Plasma-electrolytic oxidation (PEO) is a modern environmentally friendly technological process that allows to obtain multifunctional coatings on the surface of metals and alloys, characterized by high corrosion and wear resistance [1]. PEO finds relevance in the medical field for creating biomimetic coatings on implants. [2]. The growth rates of the coating are interrelated with a complex of physical processes in which, along with the processes of electrochemical oxidation at the metal-oxide interface and dissolution at the oxide-electrolyte interface, the coating and substrate are melted at the locations of the micro-discharge, and the molten metal is ejected onto the surface of the part, oxidized and deposited [3]. The nature of micro-discharges changes during processing, which affects the properties of the oxide layer. Researchers analyze video images [4], optical spectra [5,6], electrical signals, and acoustic signals [7] to develop methods for controlling coating growth. The PEO process involves electrolyte boiling and micro-discharge flashes, which generate acoustic vibrations interconnected to coating growth.

Our research is devoted to the study of acoustic signal during the PEO of a zirconium alloy in a constant voltage mode. This PEO mode is chosen to eliminate frequency harmonics that would occur in pulse voltage mode. The selection of the alloy is based on its mechanical and biocompatibility prospects for medical implants [8]. The study is aimed at finding

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regularities that allow one to indirectly estimate the coating thickness during PEO of a zirconium alloy.

2 Materials and methods

The substrate material used for the investigation was zirconium Zr-1%Nb. Before the PEO process, the samples were polished up to P4000 grit SiC paper.

We used the PEO equipment (USATU, Russia) in the DC mode. The voltage amplitude was 480 V. The electrolyte compositions was water solution of 15 gl-1 Na3PO4·12 H2O +25 gl-1 Ca(CH3COO)2+1 gl-1 NaOH + 1 gl-1 H3BO3. The PEO process was performed in a 6 liter tank at the electrolyte temperature of 20±1 °C kept constant by heating and cooling system under microcontroller regulation (TRM202, Owen, Russia). The treatment time was 600 s.

The automated recording system was used for recording the instantaneous and average values of current and voltage.

The coating thickness was measured by DeFelsko Positector 6000 eddy current gauge with an N-type sensor.

The analysis of sound signal was carried out by two independent sensors: miniDSP UMIK1 and Superlux E302. The frequency response of the sensors is approximately linear. An example of the frequency response for the E302 sensor is shown in figure 1. The sensors are designed to record a signal in the frequency range of 20 Hz – 20 kHz. UMIK1 was connected to a PC via USB, recording was performed using the Audacity program in Wav format with a frequency of 48 kHz. The second sensor was connected via a microphone input to another independent PC. To organize the recording, a special program block of control program was developed in the Labview environment. The start and end of the recording is synchronized with the start and stop of the PEO process.

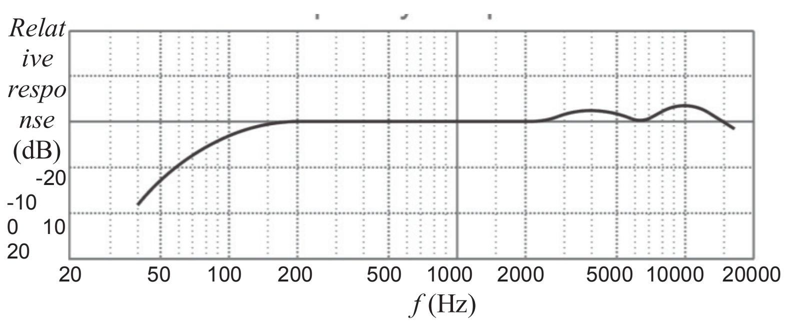


Fig. 1. Frequency response of the Superlux E302 sensor.

3 Results and discussion

3.1 Electrical response of PEO of zirconium alloy

The voltage *U* was smoothly increased from zero to the operating value of 470 V for 200 s, then maintained at a constant level (Figure 2). The current density *j* initially slowly increased with increasing voltage; the current growth was hindered by the resistance of the growing oxide layer. When the voltage reaches 330-370 V, a current surge is observed due to the ignition of microdischarges. Intense dielectric breakdowns of microdischarges and an actively formed coating lead to current fluctuations. After reaching the operating voltage, the current density gradually decreases due to the electrical resistance of the PEO coating.

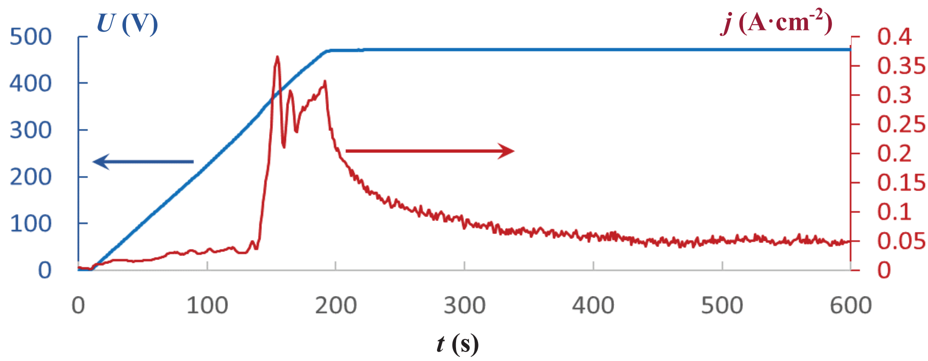


Fig. 2. Evolution of voltage and current density during PEO of Zr-1%Nb alloy.

3.2 Acoustic response

Figure 3 shows the dynamics of the sound spectrum during PEO in the constant voltage mode. The greatest sound vibrations are observed in the range of 7500-11000 Hz (figure 3, a). When the microdischarge ignition voltage is reached, this peak in the spectrum begins to increase in amplitude. It was found that the spectrum peak in the frequency range of 17000-18000 Hz decreases during processing (figure 3, b), and, the spectral peak in the range of 4000-5000 Hz increases with the growth of PEO-coating (figure 3, c). This may be due to a change in the type of microdischarges. During PEO, spark microdischarges are first ignited, which can be characterized by a higher frequency oscillation compared to microarc discharges that occur with a thicker coating.

3.3 Model for coating thickness diagnostic

The dynamics of the coating thickness for PEO in the constant voltage mode is shown in figure 4. Experimental data h (μm) were approximated by the equation with high accuracy:

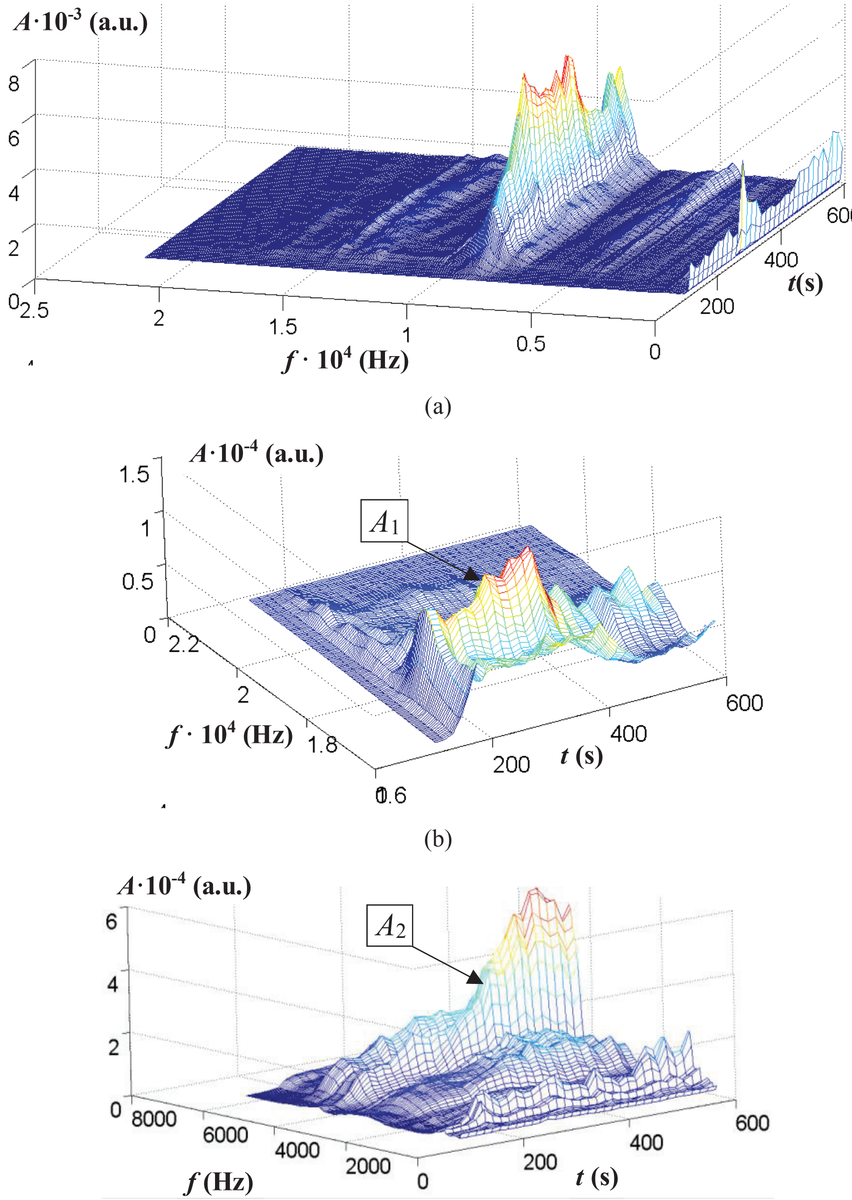
$$h = 15.66\exp(0.0004224 \cdot t) - 371.7\exp(-0.02123 \cdot t). \quad (1)$$

The dependence of the amplitude at the peak frequency in the frequency range 17000-18000 Hz on the duration of the PEO is denoted by the variable $A_1(t)$, and in the 4000-5000 Hz range we denote the variable $A_2(t)$.

To indirectly measure the coating thickness h, regression models were built in which the input parameters were $A_1(t)$, $A_2(t)$ and current density $j(t)$. The input and output parameters were scaled to equalize the influence of factors and analyze the significance of the coefficients of regression models according to the formula [9]:

$$x_i' = (X_i - \frac{X_{i\max} + X_{i\min}}{2}) \cdot (X_{i\max} - \frac{X_{i\max} + X_{i\min}}{2})^{-1}, \text{ where } i = 1, 2, 3 \text{ is } A_1, A_2, j. \quad (2)$$

$$h' = (h - \frac{h_{\max} + h_{\min}}{2}) \cdot (h_{i\max} - \frac{h_{\max} + h_{\min}}{2})^{-1} \quad (3)$$



(a)

(b)

(c)

Fig. 3. Evolution of the acoustic spectrum in the range between: 0 and 22 kHz (a), 16 and 22 kHz (b), 0 and 6 kHz (c).

The proposed regression model is described by the formula:

$$h' = A_1 \cdot a + A_2 \cdot b + j \cdot c + d \ , \tag{4}$$

coefficients of the regression model *a*, *b*, *c*, *d* are given in the table 1.

The coefficients a, b and c are of the same order, which means the significance of all the selected parameters. The coefficient of determination $R^2 = 0.97$.

Table 1. Coefficients of the regression model.

a	b	c	d
0.159	0.187	-0.591	0.022

Figure 4 shows the graphs of the change in the thickness of the coating constructed from experimental measurements, results of approximation of experimental data and regression model with acoustic informative parameters. The graphs match within the confidence interval.

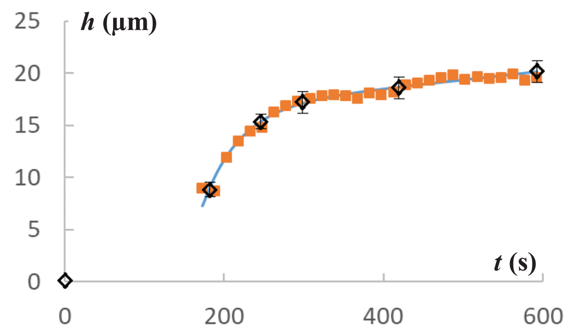


Fig. 4. Coating thickness growth: ◊ - experimental points; — approximation curve of experimental data, ■■■ regression model points.

4 Conclusion

Experimental studies of PEO of zirconium alloy Zr-1%Nb have been carried out. After 10 minutes of treatment, a thickness of 20.2±1.6 µm was achieved. The stages of the technological process with a characteristic acoustic spectrum reflecting the type of microdischarges are revealed. An exponential equation for the approximation of the experimental data on the coating thickness is obtained. Informative frequency ranges of 17000-18000 Hz and 4000-5000 Hz are found, which characterize oscillations caused by spark and microarc discharges. A regression model has been constructed that makes it possible to determine the coating thickness from the values of informative parameters of the acoustic spectrum and current density.

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