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Electrical characteristics identification of dielectric film breakdown during plasma electrolytic oxidation process



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ABSTRACT

To identify the electrical characteristics of dielectric breakdown of growing oxide film during plasma electrolytic oxidation (PEO) process, a real-time identification method based on pulse test technique was presented. The voltage–current characteristic curve and impedance curve of pulse tests were analyzed. A sharp increase in pulse current was observed when applied potential increased to critical breakdown voltage, and the breakdown voltage and absolute impedance value of oxide film increased with processing progress. Therefore, the electrical characteristics identification technique could be used for process diagnostics and self-adaptive control of PEO process.

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1. Introduction

Distinct discharge events undergoing plasma electrolytic oxidation (PEO) processing, caused by dielectric breakdown of growing oxide coatings at high voltage electrolysis, are thought to be crucial for the coating formation mechanism and strongly affect both microstructure and properties of the coating [1]. Recently, several approaches, such as optical [2–4], spectral [2–5], electrical [1,3,5,6], acoustic [7], frequency response [8] and electrochemical impedance spectroscopy [9,10], are employed for PEO process diagnostics. These monitoring techniques are effective in distinguishing key stages of the process, and certain basic characteristics of individual discharge event are becoming clearer. However, real-time adjustment of output electrical regime according to instantaneous state of coating has never been investigated. Therefore, in order to optimize automatic control of PEO process, the inter-relationship between externally-imposed electrical conditions and coating behaviors should be studied. This work aims to introduce a feasible approach to identify the state of dielectric film during PEO process in terms of electrical characteristic monitoring and provide a possible application of self-adaptive control method.

2. Experimental

6061 Aluminum alloy samples (60 mm × 60 mm × 2 mm) and a 2-L water-cooled stainless steel tank, served as a counter-electrode,

were used. For the electrolytes, potassium hydroxide (5 g L⁻¹) and sodium silicate (10 g L⁻¹) were dissolved in distilled water. The PEO process was performed by a 20 kW power supply at around 25 °C, which could provide voltage from DC in range –1000 to 1000 V to pulses with the same magnitude and frequencies up to 10 kHz.

The voltage waveform (Fig. 1) used in this work is a combination of potentiostatic and pulse test regime, and the electrical parameters of pulse tests are listed in Table 1. Initially, a wide-range test (test 1) was performed, followed by anodizing by voltage sweep up to 360 V at a rate of 3 V s⁻¹. Then, a short-range test (test 2) was implemented, prior to stepwise potentiostatic processes (steps 1–8) from 370 V to 440 V with amplification (ΔU_1) of 10 V (2 min)⁻¹. During potentiostatic process at 370 V (step 1), same tests (tests 3–6) were performed every 0.5 min. Finally, different tests (tests 7–12) were performed between every two adjacent potentiostatic processes until the work time was over. The voltage and current data were recorded with a sampling frequency of 1 MHz using a data acquisition card NI 6133 controlled by Labview software.

3. Results and discussions

Fig. 2a and b depict voltage and current waveforms of typical pulse 1 (350 V) and pulse 2 (450 V) of test 1, respectively. In Fig. 2a, current increases sharply when pulse voltage is applied, due to active-capacitive behavior of electrolyte load [11]. After showing a peak current (I_p), the current decreases sharply, observed within time t_s , and a nearly constant current (I_c) flows in time t_{ss} . The I_p is several times of I_c because of charging of equivalent capacitance.

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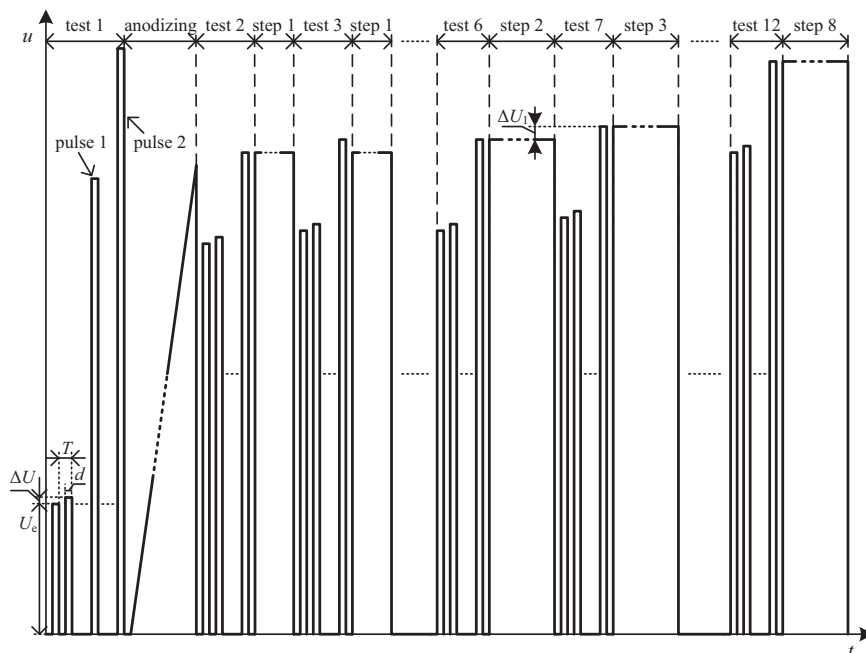


Fig. 1. Typical patterns of applied voltage during process.

Table 1
Parameters of pulse tests used in the experiment.

Test number	Test time (t, min)	Frequency (f, Hz)	Duty ratio (d)	Pulse number	Amplification (ΔU_i , V)	Basic value (U_e , V)
Test 1	0	100	0.5	45	8	100
Test 2	2	200	0.5	10	8	300
Test 3	2.5	200	0.5	10	8	310
Test 4	3	200	0.5	10	8	310
Test 5	3.5	200	0.5	10	8	310
Test 6	4	200	0.5	10	8	310
Test 7	6	200	0.5	10	8	320
Test 8	8	200	0.5	10	8	330
Test 9	10	200	0.5	10	8	340
Test 10	12	200	0.5	10	8	350
Test 11	14	200	0.5	10	8	360
Test 12	16	200	0.5	10	8	370

Fig. 2b exhibits almost similar current transient behavior within t_{ss} . The value of I_p increases with an increase in applied voltage, due to greater charging current at higher potential. However, the transient behavior within t_{ss} is different. For pulse 2, the I_c is almost five times of pulse 1 and the current profile oscillates severely. The reason of this is that higher potential results in stronger electric field strength between coating and electrolyte, leading to violent arc discharge and electrochemical reactions. As shown in Fig. 2c, the dynamic voltammograms of pulses 1 and 2 are characterized by loop circle. When higher potential is applied, it leads to higher peak current and larger loop area.

The impedance curve, which determines the resistance to current, was used to represent the dynamic process of oxide film. The magnitude of impedance (Z) was calculated as Eq. (1):

$$|Z(\omega)| = \frac{|U(\omega)|}{|I(\omega)|} \quad (1)$$

Impedance values (Fig. 2d) of pulses 1 and 2 are significantly different. The magnitude of Z of pulse 1 is almost constant (order of $10^2 \Omega$), indicating that the kinetics of the electrode process is dynamic equilibrium and the system conforms to Faraday's laws at

relatively low voltages. However, the magnitude of Z of pulse 2 fluctuates, which represents that the current–voltage characteristics vary according to Ohm's law.

To achieve adequate approximation accuracy over the voltage–current characteristic of pulse test process, appropriate pulse approximations were calculated for each pulse, by converting the pulse data into root mean square format. And also the approximation of pulse impedance was calculated as Eq. (1). The voltage–current characteristic and their impedance curves of test 1 (listed in Table 1), shown in Fig. 3, were obtained in this way.

Fig. 3a shows that initially the change in current is insignificant, and Fig. 3b shows relatively high value of Z . As the pulse potential reaches the corrosion potential of the material, the current increases slightly and Z appears to decrease due to the dissolution of the previously formed passive film. When the cell tends to be stable, current drops slowly and Z increases distinctly due to growth of oxide film. At point V_b , an abrupt current increase occurs since the electric field strength in the oxide film reaches a critical value which causes dielectric breakdown of anodic oxide film. In the region $V_b - V_u$, the current increases sharply and Z drops sharply with increasing potential. It means that higher voltage leads to formation of more discharge channels. Beyond the point V_u , pulse waveform looks like Fig. 2b and the oscillation amplitude is directly proportion to potential, meanwhile it is accompanied by an intensive low-frequency noise. In this region, powerful arc discharges may happen, which may cause destructive effects. Therefore, the applied voltage should not exceed V_u at this moment.

Similarly Fig. 4a and b show the voltage–current characteristic and their impedance curves obtained from tests 3–6, respectively. Both the voltage–current characteristic and impedance curves of different pulse tests are rather similar. The magnitude of impedance tends to increase and breakdown voltage ($V_{b1} - V_{b3}$) increases with time, which is consistent with conventional process understanding: the effective value of current decreases with time as the coating grows [12]. This trend becomes more obvious in Fig. 4c and d. The coating exhibits higher impedance state and becomes more difficult to break with increase in processing time because the second dielectric breakdown is significantly suppressed by preceding

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