Computer Aided Measurement System of Electrical Parameters of Pulsed Microplasma Processes in Electrolytic Solutions

A.I. Mamaev, V. N. Borikov*, V. A. Mamaeva, T.I. Dorofeeva

Institute of Strength Physics and Materials Science of SB RAS, 8/2 Academicheskii Ave., Tomsk, 634070, Russia,
Phone: (3822)-492091, Fax: (3822)-492576, E-mail: aim@galore.tomsk.ru
**Tomsk Polytechnic University, Tomsk, Russia*

Abstract – The new equipment – Computer Aided Measurement System (CAMS) as a tool for investigation of the physics-chemical characteristics of the ceramic layers by high current pulsed microplasma processes was developed. The CAMS has enhanced technical and metrological characteristics. The CAMS allows to record the volt-ampere characteristics of the pulsed microplasma processes at voltage up to 3000 V, the increased rate voltage up to 10^8 V/s, currents up to 100 A and control of the voltage and current with step-type behaviour of 25 mV and 1 mA accordingly.

The developed method for recording of the volt-ampere characteristics uses the original trapezoid form of the pulsed polarization voltage. This method allows to separate an active and capacity parts of current during one pulse. The measured data can be used to predict a quality of ceramic layer: thickness, porosity, roughness, and dynamics of their changes.

Some results of development and operation of the CAMS for electrical signals of the high current pulsed microplasma processes in the electrolytic solutions are submitted. Measurement of high-voltage and high-current signals of electrochemical cells is presented. The dependencies of the electrical signal form changes of the pulsed microplasma processes from the electrolyte composition, the layer formation time and working electrode material was carried out.

Introduction

The microplasma oxidation method allows to form the ceramic layers on a surface of aluminium, titanium, zirconium and their alloys for various purposes. These layers have various porosity, high hardness, resistance, high protective and electro-insulation properties [1–4]. Combination of the function layer properties with a decorative kind allows to use the parts from aluminium, titanium, zirconium treated by the microplasma oxidation in various fields of human activity: building construction, medicine, shipbuilding, aircraft and motor industry.

The modelling of the gradient oxide layer and control of the microplasma process parameters with means of CAMS allows to control the technological formation process of oxide, composition, ceramic and bio ceramic layers of various composition on aluminium, titanium surface and their alloys.

At present, there is not standard equipment for recording of the volt-ampere characteristics of the microplasma processes. Previously published [5] described the information-measuring complex, allowing to measure the active part of current and to estimate one. At the same time the volt-ampere characteristics recording of the microplasma processes is problematic in connection with low resolution of the instrument [5], since the researched signal measuring will be carried out with frequency 1 MHz. That reduces opportunities of using it both for carrying out of researches, and for technological control of the high-current pulsed microplasma processes.

The graphs fields conforming to the pulses fronts represent a special interest for the microplasma processes research, as they allow to record the volt-ampere dependences of the high-current pulsed microplasma processes, which are basic in the electrochemical processes research.

Information from the volt-ampere characteristics depends on the instrument resolution. The electrical parameter values of the microplasma processes *I* and *U* from time *t* are shown in Fig. 1. In this case for achievement of the instrument resolution in 50 mV for the trapezoid form signal with the increase front rate in 50 V/mks, the nano-range signal sampling – 1 GHz is necessary. In this case the CAMS allows to show of the electrical parameters of the microplasma oxidation process, with sampling frequency 2 GHz.

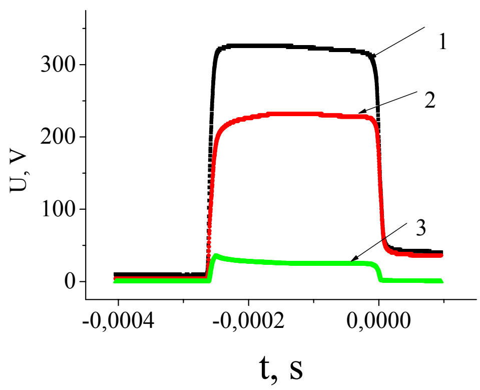

Fig. 1. Dependencies of voltage and current vs. time of the pulsed microplasma oxidation: 1 – voltage; 2 – the polarized voltage; 3 – current flowed through a cell

The CAMS diagram for the microplasma process characteristics investigation contains a generator of pulses (GP). Shapes a pulse of the trapezoid form with a voltage from zero up to 3 kV with frequency from zero up to 10 kHz and range of pulse's duration from 10 μs up to 2000 μs. The voltage pulses of GP moves on a sample placed in a current-carrying bath with electrolyte.

The CAMS Diagram

The block-diagram of the carrying out measures using of computer system consist of the pulse generator (GP), the CAMS, working electrode, reference electrode, auxiliary electrode, bath. The computer system of the measuring voltage *U*, acting with GP on a sample, the polarization voltage U_n and U_n,*, taken out with a reference electrode and the microplasma process current *I*, taken out with the help of the contact less converter current-voltage. U_n and U_n,* are the same voltage, but U_n will be used for the measuring signal with the rasping resolution, and U_n,* – with the exact resolution.

The given parameters*: I, U,* U_n, U_n,* of the three-electrode electrochemical schema are sufficient for definition active and capacitor current parts describing the microplasma process formation of the ceramic coatings in electrolytes correctly.

The voltage *U* from the pulse generator moves through the voltage divider 1:100 on one of four terminal of the analog-digital converter (ADC) and allows to supervise the target voltage on the electrode (Fig. 2). The polarisation voltage on a reference electrode – U_n moves in one case through a voltage divider 1:100 on the second orifice ADC for the measuring voltage parameters with the rasping resolution. In the second case (U_n,*) through a divider 1:100 on an electron magnifying lens for the measuring voltage parameters with the raised resolution. The electron magnifying lens manufactures shift of the researched signal part to a voltage 0 V and provide this signal on the third orifice of ADC. On the fourth orifice ADC the voltage from the transducer the current – voltage (*I–U*) moves which is directly proportional to a current in the electrochemical cell.

As the nominal dividers of the Tektronix P-5100 oscillograph, current gauge feeler Tektronix A-622, and in quality ADC a four line oscillograph of firm Tektronix TDS2024 are used. All four signals are converted in digital form by eight signals by discharges also are transferred in the computer through the asynchronous consecutive interface RS232, or through the high-speed instrument interface GPIB. As the result of the system work into the computer is introduced simultaneously data conforming to input voltage signal, voltage on a reference electrode and data of a current proceeding through a sample, Fig. 2.

Measurements both in a mode of averaging and single pulse are one of the important advantages of the developed system.

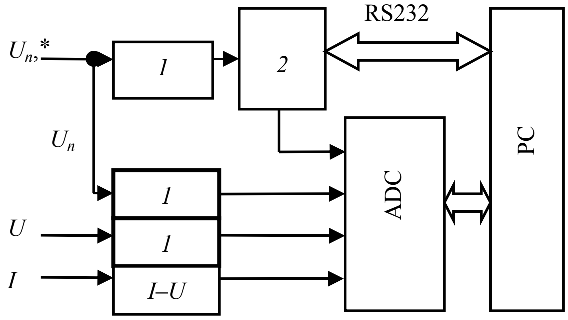

Fig. 2. The CAMS block-diagram

The block diagram of the electron magnifying lens is shown in Fig. 3.

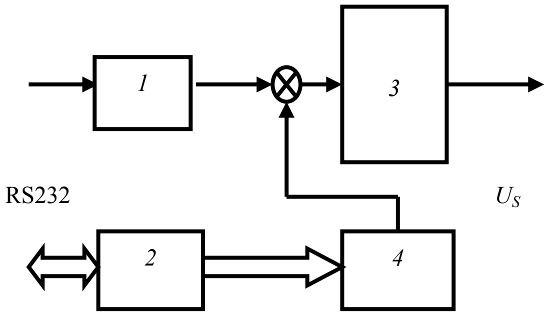

Fig. 3. The block diagram of an electron magnifying lens. *1* – voltage divider 1:3; *2* – microprocessor; *3* – control amplifier; *4* – digital-to-analog converter

In the measurement system the large current resolution in the electrochemical cell is stipulated. Thus the signal from the current-voltage converter to submit on the orifice of the electron magnifying lens (*2*) is necessary.

The input signal with the voltage divider 1:3 help and control amplifier up to voltage 10 V, and the digital-to-analog converter (DAC) frames a voltage of shift U_S for the input signal. With the control amplifier on DAC orifice the part of the input signal determined by voltage of shift enters. The control amplifier carries out a functions of electron gates limits target signal of the power supply voltage of the control amplifier and displaces researched field of signal to voltage 0 V as ADC of system allows to look through with the large resolutions only signals closely zero voltage. In result, setting a various voltage of shift, it is possible to look through various fields of electrical signals of the electrochemical cell with the large resolution ADC.

The DAC is assembled on a integrated circuit (IC) of Analog Devices AD660 and represents 16-digit DAC. With the DAC it is possible to set a target voltage with step-type behaviour $\Delta = 10\text{V}/(2^{16}) = 152\ \mu\text{V}$. Taking into account the quotient of the divider – 1:100 and quotient of the control amplifier – 3, it is possible to carry out viewing of an input voltage in 3000 V with step-type behaviour of 0.2 mV through every 50 mV. The step-type behaviour of measurement is defined by sensitivity of a digital oscillograph used in quality ADC.

The representation of the information as the current and voltage graphs is provided by the software «WaveStar», supplied complete with an oscillograph. The control DAC is carried out through the second asynchronous consecutive interface RS232 by control of the software «Terminal», written on DELPHI. The voltage of shift is introduced from the computer keyboard and is automatically pitched in the electron magnifying lens block.

Metrological Characteristics

The CAMS of the electrical parameters of the pulsed microplasma processes in electrolytic solutions has the following characteristics:
Range of the voltages – from 0 up to 3000 V;
Range of the currents – from 50 mA up to 100 A;
Quantity of measuring canals – 4;
Error of measuring of a voltage – 3%;
Sampling frequency – from 5 Hz up to 2 GHz;
Error of time measuring in the averaging mode – 0.6 ns;
Error of transformation of current to voltage:
– in a range from 50 mA up to 10 A – 3%;
– in a range from 500 mA up to 40 A – 3%;
– in a range from 40 up to 100 A – 15%.

Experimental Investigations

The developed CAMS allows to carry out the researched high-voltage (up to 3000 V) volt-ampere characteristics of the pulsed electrochemical and microplasma processes on liquid/liquid interface [6] using of the trapezoid voltage pulse form with change voltage rate $1.5 \cdot 10^9$ V/s.

For research volt-ampere characteristics on liquid/liquid interface the four-electrode cell with two reference electrodes was used. The polarization potential of liquid/liquid interface was defined as the potential difference of the reference electrodes, which are taking place in aqueous and organic phases. For this purpose one of the compare electrodes, which are taking place in an organic phase was connected in the working electrode socket of the diagram in Fig. 2. The electron-magnifying lens allows to investigate any range of potentials in details.

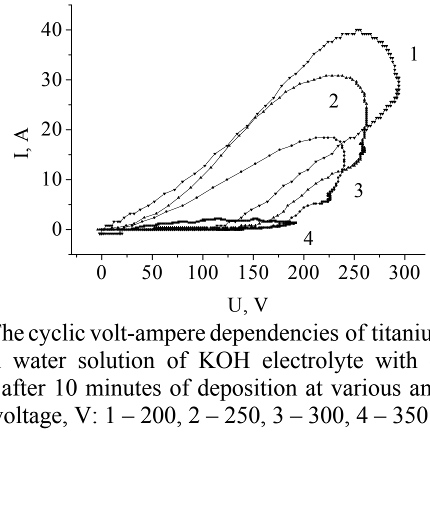

Fig. 4. The cyclic volt-ampere dependencies of titanium alloy VT-5 in water solution of KOH electrolyte with calcium fluorate after 10 minutes of deposition at various amplitude voltage, V: 1 – 200, 2 – 250, 3 – 300, 4 – 350

The CAMS allows to find out the behaviour of volt-ampere curves of the high-current pulsed microplasma processes on metal-electrolyte interface in dependence from the voltage amplitude (Fig. 4), the concentration of components keeping in electrolyte (Fig. 5).

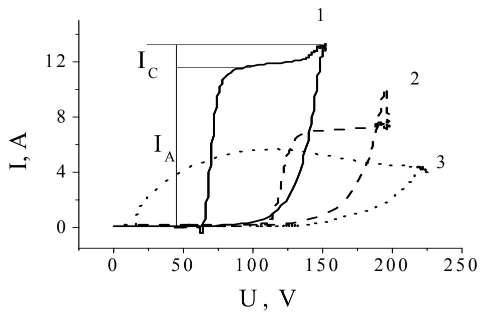

Fig. 5. The cyclic volt-ampere dependencies of aluminium alloy 2021 from the borate concentration, g/l: 1 – 6; 2 – 12; 3 – 18

The change of the volt-ampere characteristic form is observed with the electrolyte component concentration changes.

At the time from zero up to 25 mks (Fig. 1) the current value is determined by the pseudo-capacity charge processes.

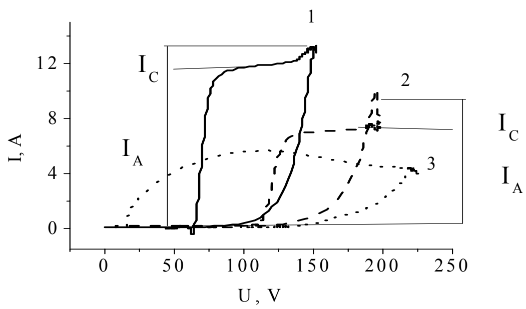

Fig. 6. The cyclic volt-ampere dependencies of microplasma treatment of aluminium alloy 2021 from time of process of deposition, min: 1 – 1; 2 – 3; 3 – 6

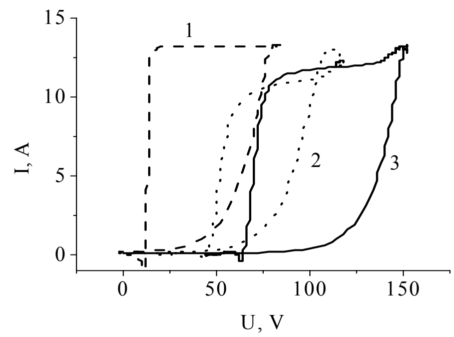

Fig. 7. The cyclic volt-ampere dependencies at the initial moment of the microplasma process time from material of electrode: 1 – AZ91D; 2 – D16; 3 – 2021

At the time part from 25 up to 270 mks the derivative of polarised voltage is decreased up to zero. In this time, the capacitor current of charge of pseudo-capacity is decreased to zero, and the active current is determined the current of all process. These laws find reflectance on volt-ampere dependences at the trapezoid form of polarised signal. The current value at the maximum of polarised potential corresponds to the sum of the capacity and active current $I = I_A - + I_C$, and the current I_A corresponds to active current of process.

Conclusion

Thus, the developed computer system allows to receive the cyclic volt-ampere characteristics of the microplasma processes up to 3000 V in electrolytic solutions with high accuracy and resolution using the voltage change rate 10^8 V/s.

The trapezoid voltage pulse is use to record the cyclic volt-ampere characteristics at the anodic and cathodic ranges of potential at electrode-electrolyte and liquid-liquid interface. The high speed of measurements and using the electron magnifying lens allows to show the most interesting parts of the volt-ampere curves for detailed research of electrochemical and microplasma processes which are taking place on the electrode at these potentials with step-type behaviour 0.2 mV.

The opportunity of research the dynamics of the change volt-ampere characteristics in dependence of voltage amplitude, time of process, concentration of components of electrolyte, nature of a substrate was shown.

The computer system of measurement opens a new opportunities in investigations and laws of the high current pulsed microplasma processes and the mechanism formation of ceramic layers.

References

[1] A.I. Mamaev, J.M. Ramazanova, *Patent RU 2046157*, 1995.

[2] A.I. Mamaev, V.A. Mamaeva., S.N. Vibornova, Advanced materials **6**, 31 (1998).

[3] Yu.Yu. Budnitskay, A.I. Mamaev, V.A. Mamaeva, S.N. Vibornova, Advanced materials **3**, 48 (2002).

[4] A.I. Mamaev, *Phisical-chemical laws high-current pulsed processes in solutions at forming oxide and ceramic coatings and modifying of a surface*, Diss. Doctor of Science, Tomsk, 1999.

[5] A.I. Mamaev, J.M. Ramazanova, P.I. Butaygin, Protection of metals **32**, 203 (1996).

[6] A.I. Mamaev, V.A. Mamaeva, *Patent RU 2198025, C25*, 2003.