An oxide coating impedance measurement during micro-arc oxidation

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Abstract. The impedance instrument converter of oxide layers formed by micro-arc oxidation (MAO) is developed. It allows continuous non-destructive testing of electric parameters of the synthesized coatings (resistance and capacitance) and electrolyte conductivity to assess the degree of degradation. The instrument converter consists of a generator, a measuring circuit, a repeater and has a digital output. The modified ammeter-voltmeter method is used as the impedance measurement technique. High accuracy of resistance, capacitance and conductivity measurements (the total relative error is no more than 0.5%) is achieved by performed functional and metrological analysis of the converter measurement channels, as well as metrological tests.

1. Introduction
Currently, impedance spectroscopy is widely used in industry and science. Because of its high information content and the possibility of non-destructive measurements, this method finds many applications. It is used in medicine for the human body composition assessment and diagnosis of various diseases [1, 2]; for food quality control [3]; in forestry to analyze wood structure and improve the efficiency of logging [4]; for the study of conducting liquids [5] and thermoelectric materials [6]; for monitoring and adjusting the operation of DC microgrids [7], etc.

The promising way of impedance spectroscopy application is the automation of the technological processes of protective coatings deposition to the surface of machine parts and devices, in particular, micro-arc oxidation (MAO). This process requires continuous non-destructive testing of the synthesized coatings properties, what cannot be achieved with traditional measurement techniques. However, the application of impedance spectroscopy allows to bypass this limitation. The impedance instrument converter developed in [8] allows making impedance measurements during MAO processing using a pulse test signal with a frequency sweep. The advantage of this approach is the high speed of measurements, but it has low accuracy. To eliminate this disadvantage, an impedance instrument converter integrated to the intelligent automated research technological MAO installation [9] was developed. This article is devoted to the consideration of the structure and metrological support of this instrument converter.

2. Structure of the MAO coating impedance instrument converter
The impedance instrument converter (figure 1) is designed to measure the impedance of a galvanic cell in the frequency range in order to determine the electrical characteristics of oxide layers (resistance and
capacity) and electrolyte conductivity to monitor the degree of its depletion. The instrument converter implements a modified ammeter-voltmeter method. The test sinusoidal signal $U_{in}$ of the controlled frequency from the generator is fed to the measuring circuit MC. MC is a capacitor voltage divider, the upper shoulder of which is the investigated sample (galvanic cell or two additional electrodes for the electrolyte conductivity measuring). The lower arm of divider is the exemplary measure (RC-circuit with switchable component values). Input $U_{in}$ and output $U_{out}$ voltage of the measuring circuit are fed to the ADC. Then the impedance is calculated by software.

![Image](https://via.placeholder.com/150)

**Figure 1.** Structure of the impedance instrument converter. OA1 – operational amplifier; M – multiplexer; ADC – analog-to-digital converter.

The impedance of the galvanic cell has active and capacitive components [10]. For this reason, it can be thought of as a parallel RC-circuit that contains a resistor $R_x$ and a capacitor $C_x$. They describe the resistance and capacitance of the coating, respectively. It is convenient to analyze such an RC-circuit through the complex conductivity $Y_x$. The exemplary measure, in the simplest case, is also an RC-circuit, which is formed by elements $R_0$ and $C_0$, and has a complex conductivity $Y_0$. Using Ohm’s law and the voltage divider formula, we obtain an analytical functional model of the measured value:

$$Y_x = Y_0 \cdot \frac{U_{out}}{U_{in} - U_{out}} = Y_0 \cdot K,$$

where $U_{in}$ and $U_{out}$ – input and output voltage of the instrument converter, respectively; $K$ – complex coefficient (sensitivity), calculated as follows:

$$K = \text{Re}(K) + i \cdot \text{Im}(K),$$

$$\text{Re}(K) = \frac{U_{in}U_{out}\cos(\varphi_1 - \varphi_2) - U_{out}^2}{U_{in}^2 + U_{out}^2 - 2U_{in}U_{out}\cos(\varphi_1 - \varphi_2)},$$

$$\text{Im}(K) = \frac{U_{in}U_{out}\sin(\varphi_2 - \varphi_1)}{U_{in}^2 + U_{out}^2 - 2U_{in}U_{out}\cos(\varphi_1 - \varphi_2)},$$

where $\text{Re}(K)$ and $\text{Im}(K)$ – real and imaginary part; $i$ – imaginary unit; $U_{in}$, $\varphi_1$, $U_{out}$, $\varphi_2$ – module and argument (amplitude and phase) of the instrument converter input and output voltage, respectively.

The complex admittance of the sample under investigation has the form:
\[ Y_x = \text{Re}(Y_x) + i \cdot \text{Im}(Y_x) = \left[ G_0 \text{Re}(K) - B_0 \text{Im}(K) \right] + i \cdot \left[ G_0 \text{Im}(K) + B_0 \text{Re}(K) \right], \]  

\[ \text{Re}(Y_x) = \frac{U_{in}U_{out}}{U_{in}^2 + U_{out}^2 - 2U_{in}U_{out}} \left[ G_0 \cos(\varphi_1 - \varphi_2) - B_0 \sin(\varphi_1 - \varphi_2) \right] - G_0 U_{out}^2, \]  

\[ \text{Im}(Y_x) = \frac{U_{in}U_{out}}{U_{in}^2 + U_{out}^2 - 2U_{in}U_{out}} \left[ G_0 \sin(\varphi_2 - \varphi_1) + B_0 \cos(\varphi_1 - \varphi_2) \right] - B_0 U_{out}, \]  

where \( G_0 \) and \( B_0 \) – conductance and susceptance of the exemplary measure.

3. Functional and metrological analysis of the impedance instrument converter

The structural metrological model of the impedance instrument converter is shown in Figure 2. Since, according to equation (1), for the indirect measurement of the complex admittance it is necessary to know the input and output voltages of the measuring circuit, the instrument converter includes two instrumentation channels of these quantities.

Figure 2. Structural metrological model of the impedance instrument converter. (a) and (b) – measurement channels of input and output voltage; \( S_{MC}, S_i, S_M, \delta_{MC}, \delta_M, \delta_{S1}, \delta_{S2}, \delta_{SM}, A^{MC}_i, A^{i} \) – sensitivity of the measuring circuit, voltage repeater and multiplexer, their multiplicative and additive errors; \( \delta_{m1}, \delta_{m2} \) – relative errors in matching the measuring circuit with the voltage repeater and the voltage repeater with the multiplexer; \( q_{ADC} \) and \( \delta_{ADC} \) – nominal ADC quantization step and its relative error; \( A^{ADC}_i, A^{ADC}_{S1}, A^{ADC}_q \) – additive, ADC nonlinearity and quantization error, respectively.

Based on the metrological model, the conversion functions of the input and output voltage, reduced to the ADC input, are obtained and determined by equations (8) and (9), respectively:

\[ U_{out1} = N_{in}q_{ADC} = U_{in}S_M, \]  

\[ U_{out2} = N_{out}q_{ADC} = U_{in}S_{MC}S_iS_M, \]
where $U_{\text{in}}$ – input voltage; $U_{\text{out}1}$ and $U_{\text{out}2}$ – output voltage of measurement channels; $N_{\text{in}}$ and $N_{\text{out}}$ – ADC code corresponding to input and output voltage; $q_{\text{ADC}}$ – nominal ADC quantization step; $S_{\text{MC}}$, $S_{r}$, $S_{M}$ – sensitivity of the measuring circuit, voltage repeater and multiplexer, respectively.

Consider the errors of the measurement channel of the output voltage. The sum of the multiplicative errors reduced to the input has the form:

$$
\delta_{\text{mul}}^{U_{\text{out}}} = \left( \delta_{S_{\text{MC}}} \cdot A_{3} \right)^{2} + \left( \delta_{S_{r}} \cdot A_{4} \right)^{2} + \left( \delta_{S_{M}} \cdot A_{5} \right)^{2} + \left( \delta_{\text{ADC}} \cdot A_{6} \right)^{2} + \left( \delta_{\text{ADC}} \cdot A_{7} \right)^{2} + \left( \delta_{\text{ADC}} \cdot A_{8} \right)^{2} \right)^{\frac{1}{2}},
$$

where $A_{3} = U_{\text{in}}^{S_{\text{MC}}^{2}}, S_{S_{M}}$, $A_{4} = U_{\text{in}}^{S_{r}}, S_{S_{M}}$, $A_{5} = U_{\text{in}}^{S_{M}}, S_{S_{M}}$

and $A_{6} = \frac{A_{7}}{S_{\text{MC}}^{2}S_{S_{M}}}, A_{7} = \frac{A_{8}}{S_{MC}^{2}S_{S_{M}}}$.

The sum of the additive errors relative to the input:

$$
A_{A}^{U_{\text{out}}} = \left( A_{a}^{MC}S_{MC}S_{S_{M}} \right)^{2} + \left( A_{a}^{r}S_{r}S_{S_{M}} \right)^{2} + \left( A_{a}^{ADC}S_{MC}S_{S_{M}} \right)^{2} + \left( A_{a}^{ADC}S_{MC}S_{S_{M}} \right)^{2} \right)^{\frac{1}{2}},
$$

where $A_{a}^{MC}, A_{a}^{r}$ – additive errors of the measuring circuit and voltage repeater; $A_{a}^{ADC}, A_{q}^{ADC}$ – ADC additive and quantization error.

The linearity error relative to the input has the form:

$$
A_{l}^{U_{\text{out}}} = \frac{A_{l}^{ADC}}{S_{MC}S_{r}S_{S_{M}}},
$$

where $A_{l}^{ADC}$ – ADC linearity error.

Similarly, we find the errors of the input voltage measurement channel. The sum of the multiplicative errors relative to the input is:

$$
\delta_{\text{mul}}^{U_{\text{in}}} = \delta_{S_{MC}}U_{\text{in}} + \delta_{\text{ADC}}U_{\text{in}}.
$$

The sum of the additive errors relative to the input is:

$$
A_{A}^{U_{\text{in}}} = \frac{A_{a}^{ADC}}{S_{MC}} + \frac{A_{q}^{ADC}}{S_{S_{M}}}.
$$

Input linearity error is:

$$
A_{l}^{U_{\text{in}}} = \frac{A_{l}^{ADC}}{S_{MC}}.
$$

Main static instrumental error relative to output voltage $A_{\text{imp}}^{U_{\text{out}}}$ is:

$$
A_{\text{imp}}^{U_{\text{out}}} = \sqrt{\left( A_{l}^{U_{\text{out}}} \right)^{2} + \left( A_{l}^{U_{\text{out}}} \right)^{2} + \left( \delta_{\text{mul}}^{U_{\text{out}}} \right)^{2}}.
$$

Similarly, main static instrumental error relative to input voltage $A_{\text{imp}}^{U_{\text{in}}}$ is:
\[ \Delta_{imp}^{Uin} = \sqrt{\left( \Delta_{Uin}^{Uin} \right)^2 + \left( \Delta_{dU}^{Uin} \right)^2 + \left( \delta_{amb}^{Uin} \right)^2}. \]

Provided metrological test have shown that full error of MAO coating impedance instrument converter in relative form is less than 0.5 %.

4. Conclusion
Thus, the developed MAO coating impedance instrument converter provides high-precision measurements of the formed oxide layers the electrolyte electrical characteristics throughout the MAO process, which allows to study their dependence on various processing parameters. The developed instrument converter is planned to be used in the future to establish a correlation between the electrical and structural parameters of the coating (thickness and porosity), which will allow the use of impedance spectroscopy for effective control of the MAO process.

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