Measurement of oxide coating thickness in the micro-arc oxidation process

P E Golubkov, E A Pecherskaya, T O Zinchenko, V A Baranov, G V Kozlov and Y V Shepeleva

Department of Information and measuring equipment and metrology, Penza State University, Penza, 440026, Russia

E-mail: pea1@list.ru

Abstract. The requirements to methods for measuring the oxide coatings thickness are formulated, allowing measurements to be carried out directly in the process of their formation in real time, which is a necessary condition for the controlled synthesis of coatings with desired properties. Based on a systematic approach, the existing methods for measuring the oxide coatings thickness and their metrological characteristics are analyzed. As a result, it was found that the most suitable for measuring the oxide coatings thickness during micro-arc oxidation are electrical methods, which include the method proposed by the authors based on a frequency integrating scanning transducer. The method proposed by authors allows to measure the thickness of the oxide layers directly in the process of their formation with high accuracy. This method is implemented in an intelligent automated system for the controlled synthesis of oxide coatings.

1. Introduction

Currently, oxide coatings with improved properties (high micro-hardness, wear resistance, corrosion resistance, heat resistance, etc.) obtained by micro-arc oxidation (MAO) are in demand in many industries [1-4]. New application areas of such coatings appear, such as layers sensitive to radioactive radiation and antibacterial biocompatible coatings for bone implants, etc. [5, 6]. In Russia and the world there are already about 10 companies using this technology, however, its widespread introduction into the industry is still difficult at present, since a large number of simultaneously influencing heterogeneous factors create an obstacle to the effective control of the MAO process and its automation [7-9]. Research teams are carrying out research to eliminate this drawback and improve technological equipment [3, 10-12], for which intelligent algorithms have been increasingly used [13, 14]. For example, in [15-17] a concept for constructing an intelligent automated system for the controlled synthesis of MAO coatings, which is based on the use of intelligent techniques that allow real-time measurement of technological parameters and oxide coatings properties to control and correct their deviation from setpoints is proposed. This indicates the need to develop appropriate measuring instruments. In this paper, as a result of an analytical review and systematization of methods for measuring the thickness of dielectric coatings on a metal substrate, the most suitable method for its implementation in an intelligent automated system for the controlled synthesis of MAO coatings is selected.
2. Systematization of methods for measuring the thickness of dielectric coatings on a metal substrate

We analyze the existing methods for measuring the oxide coatings thickness, taking into account the following requirements:

- the ability to measure in real time;
- the ability to measure the coatings thickness during their formation, i.e. without removing the part from the galvanic cell and without turning off the technological current source;
- thickness measurement range - from 1 to 300 μm;
- relative error of thickness measurement - not more than 1%.

The most common methods for measuring the thickness of dielectric coatings on a metal substrate include the following:

- eddy current method;
- thermo-graphic method;
- radiometric β-reflection method;
- ultrasonic methods: echo-pulse and resonance;
- optical methods: polarizing; interference; colorimetric; light section method; shadow section method;
- electrical methods: probe methods; capacitive method; thickness measurement methods using measuring bridges and voltage dividers.

The eddy current method for measuring thickness is based on the interaction of the coating material with high frequency currents (eddy currents) allows to determine the thickness of dielectric coatings on a non-ferromagnetic conductive substrate in the range from 0 to 500 μm with a basic error of 3%. The method is used to study finished samples with MAO coatings, but it is not suitable for measuring the thickness of these coatings during their formation, since it allows only discrete measurements.

The thermo-graphic method for determining the thickness is based on measuring the parameters of the thermal fields of the studied samples. The measurement range for the thickness of dielectric coatings on metal substrates is from a few microns to 1 mm. The disadvantage of this method is the dependence of the measurement result on thermo-physical (heat conductivity, density, reflectivity, etc.) and geometric (surface roughness) parameters of the coating, as well as on the adhesion of the coating to the substrate [6].

The radiometric β-reflection method makes it possible to measure the thickness of both metal and dielectric coatings on metal and dielectric substrates in the range from hundredths to hundreds of micrometers. Interfering parameters for measuring thickness by this method are the density and atomic number of the coating and substrate materials, their surface roughness, but the most important drawback of this method is the use of radioactive isotopes, which is dangerous for humans [18].

Ultrasonic methods for measuring thickness are based on the use of various acoustic effects arising from the passage of ultrasonic vibrations through the medium under study. There are two acoustic methods: pulse-echo and resonance. The range of measurement of coating thickness by ultrasonic thickness gauges is from 100 μm and above. The indisputable advantage of these devices is the ability to measure the thickness of coatings in hard-to-reach places or in products of a closed type (pipes, vessels), however, in galvanic production this method has not found wide application due to the large value of the minimum measured coating thickness. The main error of ultrasonic methods for measuring thickness is 1-2%, a significant contribution to which is the methodological error due to the difference in the speed of sound in different objects and the nonlinearity error.

Optical methods include the following: polarization (ellipsometric) method, interference methods, light and shadow section methods [18]. Analysis of various modifications of this method showed its
inapplicability to the process of micro-arc oxidation over the measuring range. In particular, the high-
precision ellipsometry method is designed to measure layer thicknesses in the range from 10 to 1000
nm with an error of (3-5)% and 0.5% in the thickness range from 1 to 10 nm. The disadvantage of the
colometric method is a large subjective error (up to 90%), as well as a small thickness of the measured
films.

When using electrical measurement methods, an indirect determination of the thickness is used
according to the measurement results of the following parameters: resistance, capacitance, resonant
frequency. One of the possible options for measuring the coating thickness by the resistance of the
coated sample is the four-probe method. Four probes – two current and two potential are brought to
the sample surface. An electric current \( I \) is passed through current probes, and the voltage drop across
the sample \( U \) is measured between potential probes. The coating thickness \( d \) is calculated by the
formula:

\[
d = \frac{\rho I}{U},
\]

where \( \rho \) is the specific volume resistance of the coating.

The total error in measuring the thickness of oxide films on aluminum by the four-probe method is
±15%. This method allows only discrete measurements, which is its drawback.

Measuring bridges and voltage dividers are also used to measure thickness. The thickness
measurement by the bridge method is carried out by balancing the measuring bridge, then from the
equilibrium condition the unknown complex resistance is expressed, from which the dielectric
thickness \( d \) is expressed.

3. Thickness measurement method based on frequency integrating scanning transducer

One of the options for electrical methods for measuring the coatings thickness using a capacitor
voltage divider is a method based on a frequency integrating scanning transducer (FIST), developed
specifically for the MAO process [19]. The structure of the measuring channel of the coating thickness
by this method is presented in figure 1.

![Figure 1. The structure of the measuring channel of the thickness of the MAO coating: TCS - technological current source, A - anode, C - cathode, FIST - frequency integrating scanning transducer.](image)

A metal sample with an oxide coating and a measuring electrode located at a distance \( d \) from it
form the investigated capacitor, which serves as the lower arm of the divider. The upper arm of the
divider is a reference capacitor $C_0$ with a known capacitance. The divider is connected to a frequency integrating scanning converter, which serves to convert capacitance to frequency according to the expression:

$$f_{out} = \frac{1}{4R_0C_d} + \left(\frac{C_{eq} - C_0}{C_0 + C_{eq}}\right) \cdot \frac{1}{4R_1C_d} = f_0 + \Delta f,$$

(2)

where $C_{eq}$ is the capacitance of the studied capacitor, $C_d$, $R_0$, $R_1$ are the parameters of the components of the integrated circuits of the FIST, $f_0$ is the initial frequency, $\Delta f$ is the frequency deviation (if $C_{eq} < C_0$, $f_{out}$ decreases, and when $C_{eq} > C_0$ it increases) (figure 2).

![Figure 2. The dependence of the frequency of the output pulses FIST on the capacitance of the investigated sample $C_{eq}$.](image)

The coating thickness $d_1$ is calculated by the following formula:

$$d_1 = \left(\frac{1}{C_{eq}} - \frac{1}{C_3}\right) \cdot \frac{1}{S_{dc}},$$

(3)

where $S_{dc}$ is the sensitivity of the primary transducer (the studied capacitor), $C_3$ is the capacitance of the capacitor with the electrolyte as the dielectric and having the same geometric parameters as the studied capacitor. Capacitance $C_3$ is measured during the MAO process using the second FIST module.

Thickness measurement range by this method is from 0 to 300 μm, and the error does not exceed 1%. The indisputable advantage of this method is the possibility of continuous measurement of the coating thickness directly during MAO processing.

4. Conclusion
Currently, a significant number of methods for measuring the thickness of dielectric films on metal substrates have become widespread, however, for various reasons, not all of them satisfy the requirements for the micro-arc oxidation process. The most suitable for use in an intelligent automated system for the controlled synthesis of MAO coatings are electrical methods, for example, the capacitive method and especially the method of frequency integrating scanning transducer, taking into account the specifics of the micro-arc oxidation process. The application of this method will allow not only to obtain the most accurate and reliable dependences of the thickness of oxide layers on technological parameters, but also to ensure its continuous monitoring throughout the entire MAO processing.

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References
[1] Markov M A, Gerashchenkov D A, Krasikov A V, Ulin I V, Bykova A D, Shishkova M L and
Yakovleva N V 2018 Glass Ceram. 75(7–8) 258-63
[2] Haghighat-Shishavan B, Azari-Khosrowshahi R, Haghighat-Shishavan S, Nazarian-Samani M and Parvini-Ahmedi N 2019 Appl. Surf. Sci. 481 108-19
[3] Golubkov P E, Pecherskaya E A, Artamonov D V and Shepeleva J V 2019 J. of Phys.: Conf. Ser. 1393 012083
[4] Legostaeva E V, Kulyashova K S, Komarova E G et al. 2013 Materialwiss. Werkstofftech. 44(2-3) 188–97
[5] Santos J S, Rodrigues A, Simon A P, Ferreira C H, Santos V A Q, Sikora M S, Cruz N C, Mambrini G P and Trivinho-Strixino F 2019 Adv. Eng. Mater. 21 1900119
[6] Zolotarjovs A, Smits K, Laganovska K, Bite I, Grigorjeva L, Auzins K, Millers D and Skuja L 2019 Rad. Meas. 124 29-34
[7] Mikheev A, Girn A, Vakhteev E, Alekseeva E and Ravodina D. 2015 IOP Conf. Ser.: Mater. Sci. and Eng. 70 012002
[8] Golubkov P E, Pecherskaya E A, Shepeleva Y V, Martynov A V, Zinchenko T O and Artamonov D V 2018 J. of Phys.: Conf. Ser. 1124 081014
[9] Golubkov P E, Pecherskaya E A, Kochevarov I I, Safronov M I and Shepeleva J V 2019 Proc. XXII Int. Conf. on Soft Computing and Measurements (SCM-2019) vol 1 (St. Petersburg) pp 200-03
[10] Borikov V N, Baranov P F and Bezshlyakh A D 2009 Proc. of SIBCON-2009 pp 275-79
[11] Bolshenko A V, Pavlenko A V, Puzin V S and Panenko I N 2014 Life Science J. 11(1s) 263-68
[12] Mamaev A I, Mamaeva V A, Kolenchin N F et al 2016 Russian Physics J. 58(12) 1720-25
[13] Borikov V 2006 Materialwiss. Werkstofftech. 37 915-18
[14] Ashhab M S S, Oimat A N and Shaban N A 2011 Sensors & Transducers J. 128(5) 55-66
[15] Golubkov P E, Pecherskaya E A, Karpanin O V, Shepeleva Y V, Zinchenko T O and Artamonov D V 2017 J. of Phys.: Conf. Ser. 917 092021
[16] Pecherskaya E, Golubkov P, Karpanin O, Safronov M, Shepeleva Ju and Bibarsova A 2019 Proc. Conf. of Open Innovation Association, FRUCT vol 24 (Moscow) pp 96-103
[17] Pecherskaya E A, Golubkov P E, Artamonov D V and Shepeleva J V 2019 J. of Phys.: Conf. Ser. 1393(1) 012083
[18] Babadzhanov L S and Babadzhanova M L 1999 Meas. Tech. 42 559
[19] Schuur R V 1977 ElectroComponent Science and Technology 3 203-08
[20] Golubkov P E, Pecherskaya E A, Gromkov N V, Zinchenko T O, Artamonov D V and Kochevarov I I 2019 Proc. XXII Int. Conf. on Soft Computing and Measurements (SCM-2019) vol 1 (St. Petersburg) pp 204-07