The research of the features destruction of the oxide coatings on aluminum alloy by using the method of acoustic emission

X Li¹, O V Bashkov², F Bao², V A Kim², C Zhou¹ and O G Shakirova²

¹ Heilongjiang University of Science and Technology, Harbin, China
² Komsomolsk-on-Amur State University, Komsomolsk-on-Amur, Russia

E-mail: bashkov@knastu.ru

Abstract. The influence of oxidizing the aluminum product 6070 with different times of micron exposure on the registration of acoustic emission signals (AE) during mechanical testing of samples was investigated. The results of microstructural studies after mechanical tests confirm the mechanism for generating AE signals associated with the destruction of coatings and the effects of plastic deformation on microarc oxidation, the newly formed surfaces of the substrates.

1. Introduction

Micro-arc oxidation (MAO) is a method of superficial treatment of metals and alloys of valve group, that consists in oxidizing of surface in an electrolyte at treatment the electric impulses of high amplitude. The micro-arc oxidation is divided by four stages: the stage of anodization, stage of spark digit, stage of micro-arc oxidation and stage of arc digit [1].

Two main component in generated oxide membrane are: α-Al₂O₃ (trigonal form of crystalline grate), γ-Al₂O₃ (cube form of crystalline grate). α-phase is the most stable structure, its temperature of melting is 2000 °C. γ-phase is porous and it can transform in α-phase at a temperature higher 1200 °C [2]. The oxide coating is formed by MAO consists of surface layer, basic layer and transitional layer, which is added to substrate. The surface layer is porous, loose and rough with low content of α-phase. The structure of basic layer is dense with high concentration of α-phase. It is the most essential part of oxide layer and determines descriptions of oxide coating. The main components in the transition layer are the metal of the substrate and γ-phase. This layer is irregularly jagged and closely connected with substrate.

The most common metals which can be covered by MAO are aluminum, titanium, magnesium and their alloys. Oxide coating formed by MAO, use to protect the surface from exposure to high temperatures, as dielectric coatings, as well as the solid porous basis for applying other types of functional coatings.

High adhesion and structural heterogeneity of the coatings formed by MAO, make it difficult to use standard methods for determining the quality of coatings. The method of acoustic emission (AE) is one of the methods of passive diagnostics of structural changes that occur in materials under external influence. The method is used in many areas, including the analysis of the kinetics of damage accumulation and the determination of the characteristics of materials and coatings [3].
The paper proposes the use of the acoustic emission method for the analysis of deposition modes and characteristics of oxide coatings during deformation of samples with oxide coatings deposited by MAO. In order to study the parameters of oxide coating obtained with certain modes of MAO, they were applied to samples that were subjected to mechanical tensile tests with simultaneous registration of AE.

2. Methodology of the experiment
Micro-arc oxidation was performed on 6070 aluminum alloy specimens designed for mechanical tensile testing. The specimens were made of sheet material with a thickness of 3 mm in the form of a double blade with a size of the working part of $3 \text{ mm} \times 3 \text{ mm} \times 20 \text{ mm}$. The amplitude of the voltage pulses was 400 V. The specimens were subjected to micro-arc effects for 10, 20, 30 and 40 minutes in electrolyte solution of the composition $\text{Na}_2\text{SiO}_3 (20 \text{ g/l}) + \text{NaF} (2 \text{ g/l}) + \text{KOH} (1 \text{ g/l})$.

After oxidation, the specimens were washed and dried. Before the beginning of the test, the AE converter was installed on the smooth wide surface of the specimens outside its working part near the grip of the testing machine (figure 1). The AE GT301 broadband sensor was used as a PEA. AE registrations were performed on the AE-Pro2.0 software / hardware complex [4]. The strain rate was 1 mm/min.

Figure 1. The specimens with the coating formed by MAO in the process of mechanical testing statically stretched to destruction.

3. Results and discussion
As a result of oxidation with a period of micro-arc exposure of 10, 20, 30 and 40 minutes, an oxide layer with different thickness and morphology was formed on the specimens. The microstructures for cross-sections of specimens with different microarc exposure time after mechanical testing in figure 2 are presented. Coatings are uneven in thickness and porosity in the transition layer, which are characteristic of the MAO. In some places between the coating and the substrate there are voids filled during the study with the composition for the preparation of thin sections.

The coatings applied for 30 and 40 minutes have a higher uniformity in thickness and continuity. Transverse cracks in the coatings were formed during of mechanical tensile testing. In addition, the results of an analysis of the accumulation of the number and energy of AE signals (figure 3) recorded during mechanical tests have showed a significant difference in the different stages of deformation from the time of the micro-arc effect. There were no significant differences in the mechanical properties of the samples for the investigated coating thicknesses [5].
Figure 2. The microstructure of the oxide coating applied at different times MAO: (a) – 10 min, (b) – 20 min, (c) – 30 min, (d) – 40 min.

The diagram presented in figure 3(a) shows that starting from the stage of onset of plastic deformation (II), the total AE increases with an increase in the oxidation time. The most active increase in the number of AE signals for samples with an oxidation time of 30 and 40 min is observed at the macrolocalization stage of deformation (IV).

Figure 3. Diagrams of accumulation of AE signals (a), registered for specimens with different microarc exposure time: 1 – 10 min; 2 – 20 min; 3 – 30 min; 4 – 40 min and the energy diagram of the recorded AE signals for the sample with an oxidation time of 40 min (b).

The highest value of the energy of the AE signals is observed at stage II, which is explained by the cracking of the coating with incompatibility of deformation of the plastic substrate and the hard coating. The decrease in the energy of the AE signals in stage IV is associated with the predominant plastic deformation on the surfaces newly formed after cracking due to an increase in the rate of local deformation. The numerical values of the parameters of the recorded AE signals at each stage are given in table 1.
Table 1. AE signal parameters.

| AE parameter | Oxidation time |
|--------------|----------------|
|              | 10 min | 20 min | 30 min | 40 min |
| Stage II     |         |        |        |        |
| Median frequency, kHz | - | 350 | 320 | 320 |
| E, mV²s      | - | 0.002 | 0.01 | 0.01 |
| Amp, mV      | - | 16 | 30 | 20 |
| Total AE_{II} | - | 56 | 80 | 43 |
| Total AE_{II} / Total AE % | - | 0.38 | 0.214 | 0.075 |
| Stage III    |         |        |        |        |
| Median frequency, kHz | - | 400 | 400 | 390 |
| E, mV²s      | - | 0.0003 | 0.0006 | 0.001 |
| Amp, mV      | - | 9 | 10 | 10 |
| Total AE_{III} | - | 29 | 49 | 145 |
| Total AE_{III} / Total AE % | - | 0.199 | 0.131 | 0.253 |
| Stage IV     |         |        |        |        |
| Median frequency, kHz | - | 400 | 380 | 360 |
| E, mV²s      | - | 0.0006 | 0.0008 | 0.0015 |
| Amp, mV      | - | 10 | 15 | 20 |
| Total AE_{IV} | - | 61 | 244 | 385 |
| Total AE_{IV} / Total AE % | - | 0.418 | 0.654 | 0.672 |

The analysis of the numerical values of the AE parameters (table 1) shows that the decrease in the part of the total AE in stage II from the total AE over the entire period of deformation with an increase in the oxidation time is associated with an increase in the period of cracks in the coating. The increase in the energy of AE at stage IV with the increase in the oxidation time can be explained by the fact that after cracking the coating with a period of 40 minutes micro-arc impact the rate of local deformation of the newly formed surfaces is accompanied by the excitation of a large number of AE signals with high energy values.

4. Conclusions

Analysis of AE parameters makes it possible to assess the nature of the formation and destruction of coatings formed by the MAO method. Coatings with a different period of microarc oxidation can be distinguished by the total accumulation and energy of the recorded AE signals for different stages of deformation. The results will be used to develop a method for rapid assessment of the adhesion and homogeneity of oxide-arc coatings using registered AE signals.

References
[1] Gordienko P S 1999 *Electrochemical formation of coatings on aluminum and its alloys under the electric breakdowns conditions* (Vladivostok: Dalnauka) 232
[2] Tian J, Luo Z and Qi S 2002 *Surface and coatings Technology* 154 7
[3] Bashkov O V, Panin S V and Byakov A V 2010 *Physical mesomechanics* 6 73–80
[4] Bashkov O V, Murav’yev V I, Lnochakov S Z and Frolov A V 2015 *Steel in Translation* 45(12) (New York: Allerton Press) 932–7
[5] Dudareva N U, Butusov I A and Kalschikov R V 2014 *Mechanics* 12 912–8
[6] Bashkov O V, Bashkova T I and Popkova A A 2015 *Advances in Acoustic Emission Technology* 158 283–91