On the effect of plasma electrolytic oxidation on the fatigue strength of V96Ts1 (Al-Zn-Mg-Cu) aluminum alloy

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Abstract. One of the tasks of modern material science is to obtain new functional high-strength resistant coatings on structural materials like aluminum alloys. This paper shows the effect of plasma electrolytic oxidation (PEO) on the fatigue strength of V96Ts1 (Al-Zn-Mg-Cu) aluminum alloy. Fatigue tests were carried out with uniaxial loading in accordance with the harmonic law with a constant stress of 205 MPa. The modification of the PEO-coated surface layer led to a 5-fold decrease in the cyclic durability of the V96Ts1 alloy. The dependencies of the temperature of dissipative heating and the accumulated strain on the amplitude of the effective load were obtained for the inelastic cyclic strain of V96Ts alloy samples, with and without a PEO-coated surface layer. These dependencies describing material degradation correlate with the data of the fatigue tests. A fractographic analysis of the destroyed samples was also performed.

1. Introduction

In aircraft and rocket construction, aluminum alloys are widely used due to their high strength to mass ratio [1-4]. High-strength Al-Zn-Mg-Cu aluminum alloys are the main structural materials of both contemporary and upcoming aviation equipment. V95 (7075) is the most common alloy. V95pch (7175) and V95och (7475) alloys of greater purity [5] with increased resistance to cracking and ductility (20÷40%) are also widely used. The high-strength aluminum alloy V96Ts (7055, 7449) was developed for the nuclear industry, surpassing the V95 alloy in tensile and yield strength by 20% and 30%, respectively. V96Ts1 alloy is the most durable and highly alloyed aluminum alloy (tensile strength equals 730 MPa) [6]. However, it is pressed semi-finished products that have maximum strength characteristics. In leading industries, in addition to high strength and fatigue resistance, high tribological properties of a material are required.

This paper studies the effect of a PEO-coated surface layer on mechanical properties.

Due to microplasma discharges, a layered ceramic-like coating with a thickness of 5÷250 μm, containing refractory phases of aluminum oxide, is formed on the surface of the parts made of aluminum alloys [7]. The surface microhardness can reach 20 GPa. This unique property allows for the use of parts made of aluminum alloys in those areas of engineering where parts of steel and cast iron are prevalent. For example, in the production of various structural elements of an internal
combustion engine [7, 8]. While porosity, hardness, and wear resistance of an oxide layer have been studied in detail [9], the strength properties of the PEO-coated part have not yet been sufficiently studied. It was shown in [1] that PEO-coatings have a higher fatigue resistance than surfaces made of 7475 alloy and treated by solid-state anodic oxidation [10, 11]. It was noted in [2, 3] that epoxy resin in the PEO-coating increases the fatigue and corrosion resistance of the samples made of 2024, 7075 aluminum alloys. However, fatigue tests were performed during bending with rotation.

This paper continues studies of the effect of the surface layer on the strain and fatigue resistance [12]. In parallel with the fatigue tests, energy dissipation and strain accumulation were studied using the accelerated method described in [13, 14].

This paper analyzes and compares results of studies on: energy dissipation and strain accumulation occurring under uniaxial cyclic loading with a linearly increasing stress amplitude; classical fatigue tests of samples made of V96Ts1 high-strength aluminum alloy without coating and with PEO-coating; the microstructures of the fractured surface after fatigue tests. The test results are suitable for further use in theoretical research.

2. Material, samples, equipment and methods

2.1. Material and samples for studies
An extra-durable V96Ts1 aluminum alloy was studied. It has the following chemical composition: up to 0.3% Fe, up to 0.3% Si, 0.3÷0.8% Mn, 83.84÷87.3% Al, 2÷2.6% Cu, 0.1÷0.16% Zr, 2.3÷3% Mg, 8÷9% Zn, up to 0.03% Ti. The material and heat treatment is chosen due to its maximum specific strength among aluminum alloys of about 220.7 m2/s2. The material was provided in the form of rods with a diameter of 95 mm.

To study the PEO characteristics, two series of samples were made of rods. The first series contains samples with a PEO solid protective coating, and the second series includes uncoated samples. Samples were made by turning and grinding.

The samples of type II were used due to [15]. The gage length of the sample is 37.5 mm, which makes it possible to put two extensometers for measuring axial and transverse strains. The split samples were studied (split along the pressing axis).

The heat treatment of the rod was performed according to T2 mode: quenching at 465 °C, artificial two-stage aging at 120 °C (first step, for 3÷10 hours) and 165 °C (second step, for 3÷20 hours). Heat treatment according to T2 mode with a decrease in strength characteristics by 10% increases viscosity by 10% compared to heat treatment to a maximum strength according to T1 mode (quenching at 465 °C, artificial aging at 140 °C, 16 hours). The mechanical properties of V96Ts1: ultimate tensile strength is 615.1 MPa, creep strength is 554.6 MPa, elastic modulus is 71360 MPa.

The surface of the sample, designed to measure temperature using a thermal imager, was covered with a thin layer of amorphous carbon, which makes it possible to bring the thermal emissivity closer to unity.

2.2. Equipment
The Instron 8801 universal testing system was used to determine the strain properties of V96Ts1 samples under periodic loading. To measure the increment of the components of the total strain tensor in situ, standard extensometers were used (No. 2620-601 “Dynamic Extensometer”, and “Transverse / Diametral Extensometer” No. W-E-404-F). The temperature was measured using the TKVr-IFP “SVIT” thermal imager. The thermal stress fatigue was analyzed using scanning electron microscopy (SEM), for which the fragments of the fractured samples were prepared properly: they were sonicated in ethanol and dried. The test was carried out using the “MERLIN Compact VP” microscope.

2.3. The PEO-coating process
PEO-processing of samples was carried out in the workshop of ZK-Motor LLC. The method of surface treatment according to the classification given in the monograph [16] can be characterized as a
bipolar plasma-electrolytic oxidation of the V96Ts1 valve aluminum alloy, which forms n-type oxides, with the largest stress drop across the oxide layer. The process was carried out in an electrolyzer with a counter electrode which significantly exceeds the sample in the area.

A capacitor power source was used. A water-alkaline solution with the addition of silicates was used as the electrolyte. As a result of the processing, a coating with a total thickness of 140÷145 μm was formed in the gage area of the sample. After removing the mullite layer, the thickness of the solid layer was 80÷90 μm.

As for the surface structure, after PEO the following was observed: the mullite (technological) layer thickens the sample by 40÷50 microns. This layer is not durable or loose; it can be removed with a corundum abrasive tool (sandpaper or a polymer-abrasive grinding wheel). The hard corundum layer grows inward from the initial size of the part and cannot be processed with corundum abrasive tools. Samples were tested after removing the mullite (technological) layer.

2.4. Fatigue testing and determining the strain characteristics of the material under cyclic loading

While studying new or modified materials, ultimate stresses are usually determined through the destructive method. Permissible stresses, which are less than the ultimate stresses, are used in the calculation of the strength taking into account the service conditions of the structure. In our studies, the permissible stress during cyclic loading was 2/3 of the ultimate strength of the material [17]. Fatigue testing of the samples was carried out at the same loading with a cycle stress amplitude of 205 MPa, with the asymmetry coefficient of R = 0, at a frequency of 4 Hz. Fatigue tests were carried out in air at room temperature.

At the same time, a study was performed according to an accelerated method [13, 14]. The sample, in thermodynamic equilibrium, was subjected to zero light loading with a monotonically increasing stress. This made it possible to obtain the dependences of the strain and temperature on the stress amplitude.

Figure 1 shows a typical loading. The loading consisted of two steps in which soft loading was performed with a stepwise increase in the stress amplitude (Step 1) and unloading (Step 2).

Step 1 contained 3760 load cycles under the harmonic law with a frequency of 4 Hz with a total test duration of 940 seconds. The increment of the load amplitude for each step \( \sigma_{a\ step} \) was calculated by the formula: \( \sigma_{a\ step} = \sigma_{a\ max} / N_{cycle} \), where \( \sigma_{a\ max} \) is the maximum stress amplitude in step 1 (\( \sigma_{a\ max} = \sigma_{0.2} \)), \( N_{cycle} \) is the number of cycles in the first step. The load, axial, and transverse strain and radiation temperature of the working surface of the sample were simultaneously measured during the tests. The tests were carried out at room temperature.

![Figure 1. Loading. Step 1 is cyclic loading with increasing stress amplitude (zero cycle); Step 2 is unloading to zero stress.](image)

After completing the test program, the measurement data was analyzed. The extreme values of strain and temperature were determined for each extreme stress. Experimental studies performed with the measurement of longitudinal strain and radiation surface temperature make it possible to ensure the completeness of the data when studying the accumulation of irreversible strain and energy dissipation in a sample.
3. Main results and discussion

3.1. Fatigue tests
Statistical processing of the fatigue tests’ results was carried out. For each series of experiments, the average number of cycles before deterioration was determined: the expected value is \( \bar{N} = \frac{N_i}{n} \).

Fatigue endurance (at \( R = 0, \sigma = 205 \text{ MPa} \)) of circular samples, made of the V96Ts1 alloy, with a PEO-coating and without it, is 5973 and 29619 cycles respectively. Obviously, the PEO-coated layer had a significant effect on the durability of the samples. The average value of the durability at the studied stress level for PEO-coated samples is 20% of the base of the corresponding average value of samples without the coating.

In parallel with fatigue tests, cyclic loading of the samples was carried out according to the procedure described in paragraph 2.4. For the first (coated) and second (uncoated) samples, figure 5 compares the dependences of the temperature increments (\( \Delta T_a \)) and the cumulative amplitude of irreversible longitudinal strain (\( \varepsilon_{ax}^p \)), respectively, on the stress amplitude (\( \sigma_a \)). Here \( \varepsilon_{ax}^p = \varepsilon_{ax} - \varepsilon_{ax0} \), where \( \varepsilon_{ax} = (\varepsilon_{ax \text{ max}} - \varepsilon_{ax \text{ min}}) / 2 \) is the amplitude of the total longitudinal strain; \( \varepsilon_{ax \text{ max}}, \varepsilon_{ax \text{ min}} \) are the extreme values of the longitudinal strain; \( \varepsilon_{ax0} = \sigma_a / E_0 \) is the longitudinal strain at the stress amplitude \( \sigma_a \); \( E_0 \) is the tangential modulus of elasticity calculated at the beginning of Step 1, where inelastic strain is insignificant.

In figure 2, the dependences are indicated by the numbers “1” and “2” as being with and without a PEO-coated layer, respectively. The data presented in figure 5 make it possible to evaluate the effect of the PEO-coated layer on the stress amplitude at which dissipative heating begins, and the process of accumulation of irreversible strain starts.

Figure 2 shows that, while undergoing periodic strain, the PEO-coated layer reduces the stress amplitude at which the process of plastic strain is activated. An increase in the temperature of the dissipative heating of the sample is observed. This observation is consistent with the known softening effect.

The diagram presented in figure 2 makes it possible to estimate the cyclic elastic limit of V96Ts1. Irreversible plastic strain in uncoated samples and temperature increment occurs at a stress amplitude above 100 MPa.

At a stress amplitude of 205 MPa (figure 2), the amplitudes of irreversible longitudinal strain (\( \varepsilon_{sx}^p \)) and temperature increments (\( \Delta T_a \)) are 3 times greater for PEO-coated samples than for samples without the coating.
3.2. Metallographic studies of fractography

To explain the results of the fatigue tests presented in paragraph 3.2, images of the destroyed parts of the samples were considered figures 3, 4).

Figure 3a shows a typical crack initiation, where a fatigue crack is initiated from the surface of the sample. The arrows indicate the directions of the fatigue crack propagation. Figure 3a shows fatigue development (right) and accelerated development – rupture area (left).
Figure 3b shows a fracture of a PEO-coated V96Ts1 alloy of 90 μm thick. There are many sites of crack initiation along the perimeter of the sample cross-section (Figure 3b, 4a).

![Figure 4a](image1.png)  ![Figure 4b](image2.png)

**Figure 4.** Fracture of a coated sample tested at a stress amplitude of 205 MPa.

Figure 4 shows a fracture of a PEO-coated V96Ts1 alloy of 90 μm thick. There are many sites of crack initiation along the perimeter of the sample cross-section (figure 4 a, b). Chemical analysis showed that cracks started at the boundary between the coating and the base layer (Figure 4 a, b, table 1, 2). Figure 4b and table 1, 2 show that a crack separates the alloy (spectrum 1, table 1) and the coating (spectrum 2, table 2).

| Table 1. Chemical spectrum 1. | Table 2. Chemical spectrum 2. |
|-----------------------------|-----------------------------|
| **Element** | **Weight %** | **Atomic%** | **Element** | **Weight %** | **Atomic%** |
| Mg           | 2.99          | 3.53         | O            | 55.23        | 67.50        |
| Al           | 86.30         | 91.78        | Mg           | 1.01         | 0.81         |
| Mn           | 0.09          | 0.05         | Al           | 43.25        | 31.34        |
| Cu           | 1.92          | 0.87         | Si           | 0.51         | 0.35         |
| Zn           | 8.41          | 3.69         | Results      | 100.00       |              |
| Zr           | 0.30          | 0.09         | Results      | 100.00       |              |

A fractographic study showed that a decrease in the cyclic durability of PEO-coated samples is connected with many stress concentrators which act as sites of crack initiation. These concentrators are probably related to the features of the PEO-coating. To clarify the causes of the formation of multiple initial cracks on the surface of the PEO-coated samples, additional studies are needed. They will determine the microrelief at the boundary between the alloy and the oxide layer.

The studies show that PEO-coated samples withstand 5 times fewer loading cycles than untreated ones. This result suggests that parts of this material operating at stress above 200 MPa can only be used on equipment with a low assigned fatigue life. However, the endurance of the samples at stress less than 80 MPa remains unconsidered, where, as figure 2 shows, there is no accumulation of plastic strain and dissipative heating.

4. **Conclusion**

The effect of the PEO-coated layer on the durability of the V96Ts1 aluminum alloy under uniaxial zero loading was studied. It was experimentally established that:
1) PEO-coating (90 μm) reduces the cyclic durability of samples by 5 times with a stress amplitude of 205 MPa;
2) coated surface activates the degradation of material at the stress less than or equal to 100 MPa;
3) on the PEO-coated sample, cracks are initiated along the entire perimeter of the sample cross-section, in contrast to the uncoated sample, where cracks are initiated only in one place.

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