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SEALING EFFECT ON CORROSION RESISTANCE OF BORIC SULFURIC ACID ANODIZING ON AA2024

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Aluminum is widely used due to its excellent properties, lightweight and thermal conductivity. However, when used in aircraft applications, it can cause corrosion and sticking, compromising safety. To address this issue, anodizing is used to improve aluminum's corrosion resistance and adhesion. In this study, the AA2024 material was anodized using the boron-sulfuric acid anodization (BSAA) process, followed by a sealing process using acetic acid. This sealing process forms an oxide layer on the aluminum's surface, which reduces the corrosion rate. The study investigated the effects of anodization voltage and time on the results of BSAA anodization through quantitative and qualitative measurements, including corrosion resistance, potentiodynamic polarization, scanning electron microscopy (SEM), and energy dispersive X-ray spectroscopy (EDS). The results showed that samples anodized with a gasket could reduce the corrosion rate by up to 85 % compared to those without a gasket and substrate. The most significant reduction in corrosion rates occurred at an anodization voltage of 10 V and an anodization time of 15 min. The potentiodynamic test results indicated that the Tafel plot during sealing lies in the cathodic region where the corrosion current density decreases with increasing voltage. SEM observations revealed that the anodizing process could provide an oxide layer on the samples' surface, while the sealing process creates a smooth surface. EDS analysis showed that an oxide compound was formed in an oxide bond state after the sample surface was subjected to the sealing treatment. Overall, the study demonstrates the effectiveness of BSAA anodization in improving corrosion resistance and highlights the importance of considering the anodization parameters

***Keywords:** corrosion resistance, acetic acid, AA2024, boric sulfuric acid anodization*

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1. Introduction

Aluminum alloys in the 2xxx series are particularly useful for their high strength-to-weight ratio, making them ideal for aircraft structures. They also have good fatigue resistance and excellent machinability, which makes them popular for aerospace applications [1]. Moreover, aluminum alloys in the 2xxx series have good weldability and formability, which allows them to be shaped into complex parts with relative ease. Additionally, they are relatively inexpensive and widely available, making them a cost-effective solution for many applications. Overall, aluminum and its alloys are an important and versatile group of materials that are widely used in industry due to their desirable properties and cost-effectiveness.

The use of the 2xxx series aluminum alloys in aircraft structures has significantly contributed to the aerospace industry's advancements. Due to their high strength-to-weight ratio and excellent resistance to fatigue crack growth [2], these alloys have enabled aircraft manufacturers to design and build lighter, more fuel-efficient, and more durable aircraft. However, the presence of copper in these alloys can negatively impact their corrosion resistance and fatigue strength [3]. This is a significant issue since aircraft operate in harsh environments that expose them to various corrosive elements and high stress loads.

To address these issues, several methods have been developed to enhance the corrosion resistance and fatigue strength of 2xxx series aluminum alloys. One of these methods is the impressed current cathodic protection (ICCP) system [4], which involves the application of an electrical current to the metal to prevent corrosion. Coating is another effective method that can be used to protect aluminum from corrosion by forming a protective barrier between the metal and the environment [5].

Anodizing is a process that is commonly used to enhance the corrosion resistance of aluminum alloys, including the 2xxx series. This process involves immersing the metal in an acid bath and applying an electrical current to produce a protective oxide layer on the surface of the metal [6]. This coating not only provides excellent corrosion protection but also improves the metal's adhesion properties, making it more suitable for bonding with other materials [7]. The process uses oxidation to alter the chemical composition of the material's surface. The specific requirements for anodizing aluminum alloys depend on their composition, which determines the appropriate current and voltage densities [8]. During the anodizing process, the substrate composition, current density, voltage changes, and temperature all play a role in the formation of the aluminum oxide layer [9]. Anodizing creates two different types of oxide layers: a non-porous, thin, and sturdy barrier layer called the barrier layer, and a thicker, porous oxide layer called the porous layer [10].

Boric sulfuric acid anodizing is a widely used surface treatment technique that improves the corrosion resistance and durability of aluminum and its alloys. However, the corrosion resistance of the anodized layer can be further enhanced by applying a sealing treatment to the anodized surface [11]. The sealing effect on corrosion resistance refers to the process of closing the pores and voids in the anodized layer by using various sealing agents such as hot water, nickel acetate, sodium dichromate, and others [12]. This process enhances the barrier properties of the anodized layer and protects the underlying metal from corrosive environments.

Several studies are still conducting experiments to provide a coating on metal to inhibit the corrosion rate. One coated metal type is aluminum because the material is light and easy to form. However, one of the weaknesses of aluminum is its mechanical properties, which tend to be weak. In addition, this material will easily corrode in an inappropriate environment. therefore, research on the development of the corrosion rate by sealing the material with the anodizing method is relevant.

2. Literature review and problem statement

Anodization is essential in automotive assembly, aircraft, and other metal products. Anodizing is done to get a thicker and uniform oxide layer on the aluminum surface. In recent years, several studies on anodization research have been carried out. First, the relationship between the forming conditions of the alumina film and the breakdown voltage is examined [13]. This study's results indicate that the breakdown voltage's magnitude during the anodizing process depends on the electrolyte solution. Furthermore, the influence of substrate composition, current density, voltage, and temperature changes during the anodizing process was investigated [9]. In addition, this study's results indicate that the electrolyte's temperature increases with increasing applied current density, while the resulting film thickness is more influenced by the magnitude of the anodizing voltage and is independent of the substrate.

The Boeing Company is developing BSAA (Boric Sulfuric Acid Anodization) anodizing and has met the technical requirements as a substitute for chromic acid anodizing [11, 14]. This anodization is environmentally friendly and also offers high corrosion resistance. Over the past two decades, researchers have conducted many experiments to optimize BSAA coatings on a wide range of aluminum alloys [15]. The paper [16] presents the effect of the composition of the electrolyte solution on the aluminum anodizing process. The results showed that the thickness of the aluminum oxide layer produced varied greatly depending on the type and composition of the electrolyte used. This research also mentions that temperature differences in the electrolyte cause the resulting viscosity.

The paper [17] highlights a study on the anodizing process of the 1100 series aluminum using variable voltage and anodizing time. The results indicate that the characteristics of the resulting aluminum oxide layer depend on several factors, including the electrolyte temperature, current density, and the surface polishing process of the substrate. Furthermore, the paper investigates the growth mechanism of the porous aluminum oxide layer formed using an electrolytic sulfuric acid solution. The study revealed that the porosity of the coating is influenced by the substrate material rather than the level of anodizing stress. This finding is essential as it indicates that, to control the porosity of the coating, it is crucial to choose the right substrate material [18]. Additionally, this study's findings can be useful in developing improved anodizing techniques that can be used to control the porosity of the coating and, therefore, optimize the anodized aluminum's properties.

Sealing is traditionally accomplished by immersion in boiling deionized water, known as hot water sealing. However, high temperatures and slow kinetics are required, which means significant energy consumption [19]. As a result, the hot water process has been successively replaced by cold sealing since the 1980s [20]. Sealing dichromate and nickel acetate is considered the most effective for corrosion prevention, but Cr(VI) is toxic [21, 22].

Recent studies have explored various sealants to protect anodic aluminum oxide from corrosion, including nickel acetate, nickel fluoride, cerium acetate, sol-gel sealing, and a complex sealing process with PTFE. For example, one study found that cold nickel acetate and hot water sealing decreased the pore size, while hot nickel acetate filled the pores resulting in a low porosity and small mean pore radius [23]. Another study showed that boiling water and potassium dichromate sealed films provided higher corrosion resistance in acidic solutions, while nickel fluoride sealed film was better in basic solutions [24, 25]. Cerium acetate was found to be effective in protecting insulator pins in low-pH and high-corrosion-rate environments, potentially leading to the development of eco-friendly anti-corrosion coatings for power industries [12]. Sol-gel sealing was shown to delay the access of aggressive species to the barrier layer [26, 27], while a complex sealing process with PTFE had a large positive reverse pump rate but may not extend its service life before leakage [28].

The research paper referenced as [13] examined the relationship between bound water and the stability of anodic oxide films on aluminum by measuring the quantity of bound water and its stability through various experimental methods. However, it did not investigate bound water's formation mechanisms or impurities' impact on the oxide film's stability. Another paper referenced as [19] investigated the effect of different sealing processes on the corrosion resistance of coatings formed on 2024 aluminum alloy via tartaric-sulfuric anodizing. However, it did not examine the effects of other anodizing parameters on the coatings' corrosion resistance or the coatings or the microstructure and composition of the coatings and their impact on the sealing process and corrosion resistance. The effectiveness of a novel sealing process on the corrosion resistance of an anodic oxide coating was evaluated in the article [21]. However, the study did not examine the mechanical properties of the coating or how the coating affected the mechanical properties of the substrate. The article [22] investigated the effects of two sealing treatments on the corrosion behavior of Al-based amorphous/nanocrystalline coatings using electrochemical impedance spectroscopy and potentiodynamic polarization measurements. The article did not thoroughly explain the observed corrosion behavior and recommended further research to explore the underlying mechanisms.

Previous research on the anodizing process has primarily focused on the impact of various parameters, such as temperature, time, current density, solution composition, and pre-treatment, on the thickness of the aluminum oxide layer produced. The thickness of the oxide layer is essential for determining the material's corrosion resistance. However, further research is necessary to investigate the impact of anodizing on other material properties, such as mechanical and physical properties and corrosion resistance. Such studies can help optimize the anodizing process for specific applications. Additionally, there is a need to develop new sealant methods that can improve the corrosion resistance of anodized aluminum. Based on these problems, all this allows to assert that it is expedient to conduct a study on the corrosion resistance of boric sulfuric acid anodizing on AA2024 with acetic acid sealing process.

3. The aim and objectives of the study

The aim of the study is to inhibit the corrosion rate of AA2024 with boric sulfuric acid anodizing (BSAA) with and without sealing process using acetic acid (CH_3COOH) solution.

To achieve this aim, the following objectives are accomplished:

– to study the effect of sealing using boric sulfuric acid anodizing on the corrosion rate of AA2024;

– to study the effect of sealing using boric sulfuric acid anodizing on potentiodynamic polarization of AA2024;

– to study the effect of sealing using boric sulfuric acid anodizing on micrographs with SEM and changes in the compound elements of AA2024.

4. Materials and methods of experiment

4. 1. Object and hypothesis of the study

The object of research on the sealing effect on corrosion resistance of boric sulfuric acid anodizing on AA2024 is to investigate the effect of the sealing process on the corrosion resistance of anodized AA2024 samples. The study's main hypothesis was that the sealing process using an acetic acid solution after the boric sulfuric acid anodizing process would improve the corrosion resistance and adhesion of the AA2024 material. Some of the assumptions made in this study include:

1) anodizing with boron-sulfuric acid improves the corrosion resistance and adhesion of AA2024;

2) sealing the anodized samples with acetic acid solution further enhances the corrosion resistance of the material;

3) anodization voltage and anodization time affect the corrosion resistance of the samples;

4) samples anodized with a gasket can reduce the corrosion rate compared to samples anodized without the gasket and its substrate.

In general, scientific studies often involve simplifying assumptions to make the research more manageable or to isolate specific factors of interest. Some possible simplifications that could have been adopted in this study on the sealing effect on corrosion resistance of boric sulfuric acid anodizing on AA2024 are:

– simplified sample geometry: the study may have used simple geometric shapes (e.g., flat disks) instead of more complex shapes to simplify the anodizing and sealing processes;

– controlled environmental conditions: the study may have controlled the environmental conditions (e. g., temperature, humidity, etc.) to minimize the effect of external factors on the corrosion resistance of the samples;

– homogeneous material composition: the study may have assumed that the AA2024 material used was homogeneous throughout, without any defects or impurities affecting the corrosion resistance;

– idealized sealing process: the study may have assumed an idealized sealing process that completely covers the anodized surface with a uniform layer of oxide without any defects or variations in thickness;

– single anodizing and sealing parameter: the study may have focused on a single anodizing voltage and time and a single-sealing solution and time to simplify the experimental setup and isolate the effect of sealing on the corrosion resistance.

4. 2. Material

In this study, an aluminum alloy AA2024 with a diameter of 12 mm and thickness of 3 mm was used. The alloy was subjected to anodizing at room temperature with constant voltage of 10 V and 15 V using a BSAA electrolyte mixture containing 45 gr/l sulfuric acid and 8 g/l boric acid. The anodizing was performed for 10 and 15 minutes, and the selected mechanical variables were the surface roughness of the coating and the corrosion rate after anodizing and sealing. The chemical composition of aluminum AA2024 was determined using an optical emission spectrometer (OES), and it was found that AA2024 is an aluminum alloy with copper as the main alloying element. Table 1 provides details of the chemical composition of aluminum AA2024.

Table 1

Chemical composition (wt %) of aluminum AA2024

AA2024	Si	Fe	Cu	Mg
	0.5	0.5	3.9	1.5
	Zn	Cr	Ti	Al
	0.25	0.1	0.15	92.5

In addition, the base metal is also tested for tensile strength to determine the strength of the base metal. The results of the tensile test show that it still has a maximum tensile stress (σT) of 463 MPa, yield stress (σy) of 360 MPa, and elongation (ε) of 17.8 %, which is still within the material limits of the AA2024 standard.

4. 3. Anodizing process

Aluminum alloy AA2024 has a diameter of 12x3 mm thick with constant voltage, 10 V and 15 V for 10, 15, and 20 minutes at room temperature 27 °C. The controlled variable was the BSAA electrolyte used as an electrolyte mixture of 45 gr/l sulfuric acid and 8 gr/l boric acid. The distance between the anode and cathode was 5 cm. The electrolyte mixture ratio for anodizing aluminum ranges from 30.5 to 52.0 g/l sulfuric acid and 5.2 to 10.7 g/l boric acid. This study focused on a mixture of 45 gr/l of sulfuric acid and 8 gr/l and boric acid, as recommended [12].

Fig. 1 outlines the various steps involved in preparing for the anodizing process. The first step involves preparing the material. The process involves cleaning/degreasing with alkaline solution (sodium hydroxide, potassium hydroxide, and trisodium phosphate), rinsing with RO water, etching with 100 gr/l caustic soda, rinsing, desmut with a mixture of 75 % phosphoric acid (H_3PO_4), 15 % sulfuric acid (H_2SO_4), and 10 % acetic acid (CH_3COOH) at room temperature for 2 minutes, rinsing, anodizing with a mixture of 45 gr/l H_2SO_4 and 8 gr/l boric acid, rinsing again, and finally sealing with a mixture of 50 gr/l acetic acid (CH_3COOH). Acetic acid is a popular choice for filling oxide films due to its ability to dissolve in water and form a stable solution, penetrate pores of the oxide film, react with the oxide layer, and form a dense and uniform aluminum oxide layer. Moreover, acetic acid has a low boiling point and is relatively safe to handle, making it a practical choice for the anodizing process.

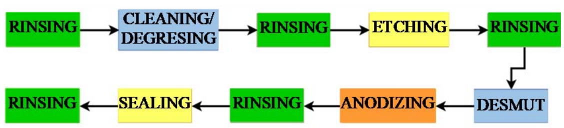


Fig. 1. Preparation scheme for anodizing process

In this anodizing tool, the anode is typically made of the material to be anodized, while the cathode is usually made of a material that doesn't react with the electrolyte. The anode and cathode are placed in the electrolyte, which is a solution that conducts electricity. The direct current source is then used to apply a voltage to the anode and cathode, which initiates the anodizing process. The voltage can be adjusted depending on the desired thickness and properties of the anodized layer. The schematic also shows other components in Fig. 2, such as the container that holds the electrolyte and the mechanism for circulating the electrolyte to ensure uniform anodizing. The anodizing tool can be designed in different shapes and sizes depending on the specific application and requirements.

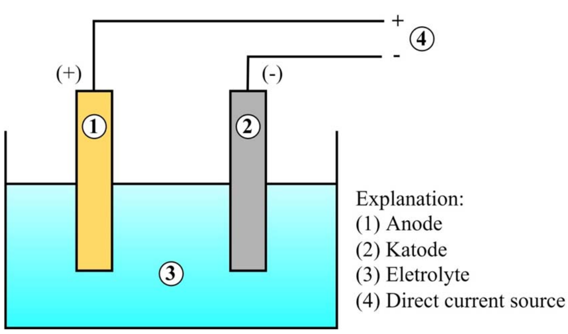


Fig. 2. Schematic illustration of anodizing tool

During the anodizing process, the two specimens of the AA2024 aluminum alloy were immersed in the electrolyte solution, with one serving as the anode (+) and the other as the cathode (–). A voltage source was connected to the cathode to supply a constant voltage of 10 volts for a duration of 10, 15, and 20 minutes. The electrolyte solution was a mixture of sulfuric acid and boric acid with concentrations of 45 gr/l sulfuric acid and 8 gr/l boric acid. The anodizing process involved the formation of a protective oxide layer on the surface of the aluminum alloy through the process of electrolysis. The duration of the process and the concentration of the electrolyte solution were varied to investigate their effect on the resulting properties of the oxide layer, such as its thickness, porosity, and corrosion resistance.

4. 4. Specimen test method

Compositional and tensile tests are performed to determine material properties. A material composition test was performed using a spectrometer to determine the type and specifications of materials used. Aluminum alloy AA2024 A tensile test to determine the strength of the base metal to obtain the following mechanical properties of the test material: Elastic limit, yield point, and tensile strength.

The process of testing corrosion with anodizing is depicted in Fig. 3. In the first step, the material is prepared as described earlier. In the second step, the corrosion test is conducted on the AA2024 specimen by utilizing the boric sulfuric acid anodizing (BSAA) process for durations of 10, 15, and 20 minutes, and the voltage utilized is either 10 or 15 volts. Finally, in the third step, the specimens are tested, and both qualitative and quantitative measurements, including corrosion rate and polarization, are conducted using Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDS) techniques.

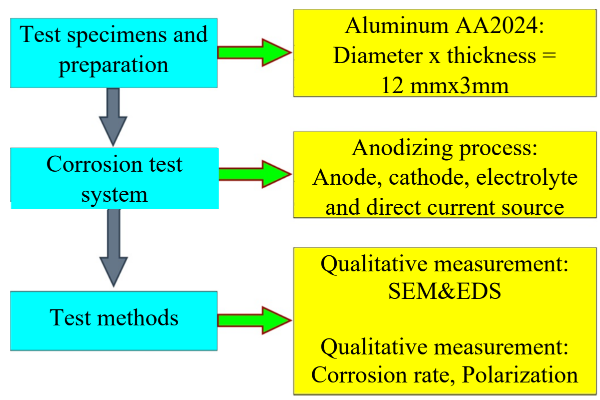


Fig. 3. Corrosion testing flow by an anodizing process

In addition to the SEM and EDS tests, the study conducted various other tests to evaluate the effectiveness of the anodizing process. The ASTM G102 standard was utilized to measure the corrosion rate and polarization of the specimen. This standard involves measuring the specimen's potential, current, and corrosion rate under specific conditions. By collecting this data, the study aimed to evaluate the corrosion resistance of the anodized aluminum samples.

The corrosion testing results were carefully analyzed to identify the optimal anodizing process parameters that could deliver the required level of corrosion resistance. By comparing the corrosion rates of samples subjected to different anodizing process parameters, the researchers could determine which process parameters produced the most effective corrosion resistance. This information can help guide the development of new anodizing processes that can deliver superior corrosion resistance in various applications.

5. Results of the experiment corrosion rate using boric sulfuric acid anodizing of AA2024

5. 1. Corrosion rate on aluminum AA2024 with boric sulfuric acid anodizing

The corrosion rate is one of the quantitative measurements carried out on AA2024 with the BASS method. This corrosion rate measurement is applied to raw materials, materials without sealing, and materials with sealing. Anodizing time also determines how fast or slow the corrosion rate is on the material. Fig. 4, *a, b* show the corrosion rate with an anodizing voltage of 10 and 15 volts, respectively.

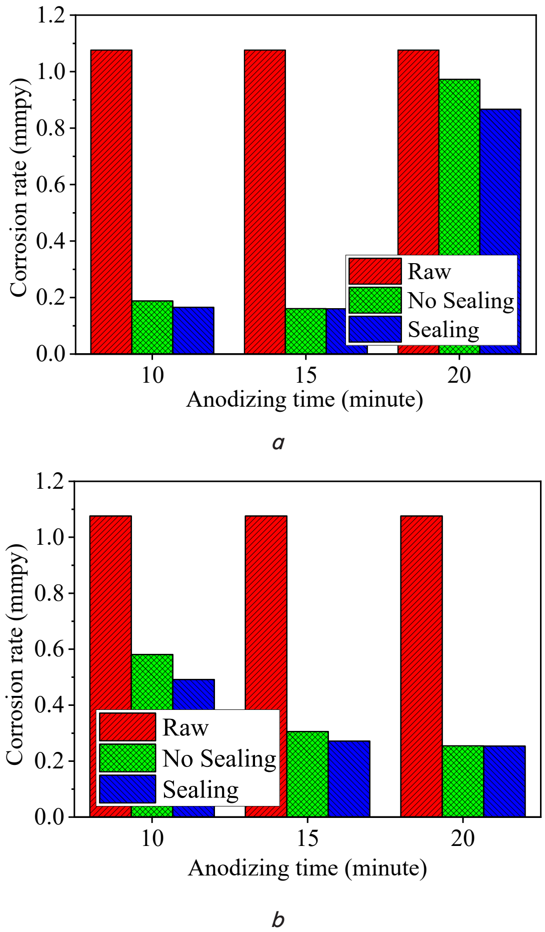


Fig. 4. Corrosion rate with anodizing voltage: *a* – 10 volts; *b* – 15 volts

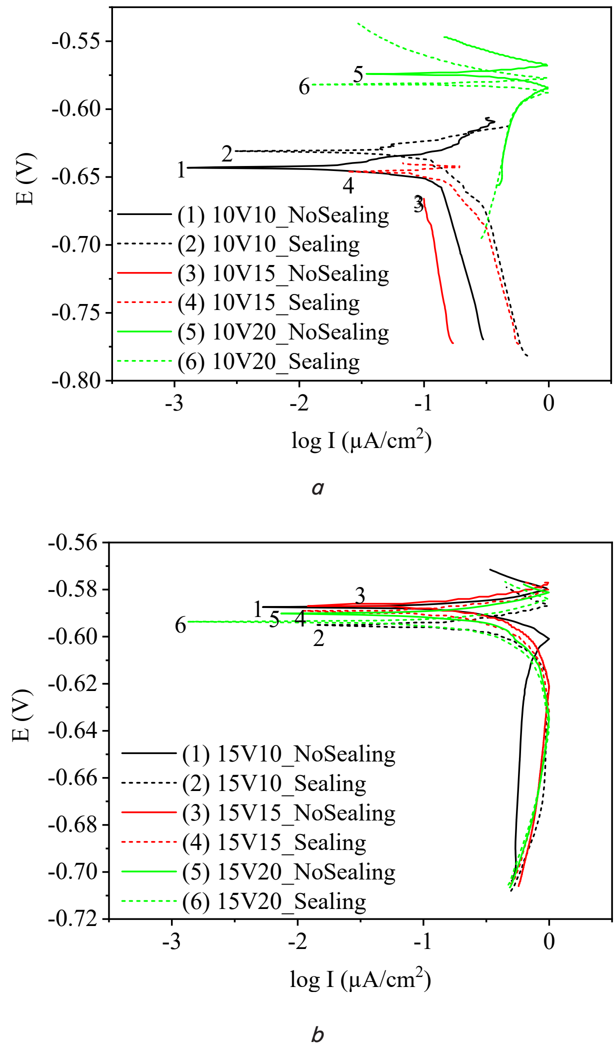


Fig. 5. Polarization curves with anodizing voltage: *a* – 10 volts; *b* – 15 volts

The anodizing process with a voltage of 10 volts showed decreased corrosion rate on specimens without sealing and samples with sealing at each anodizing time variation. At anodizing time of 10 minutes, the corrosion rate decreased by up to 84 % of the raw material. Furthermore, at anodizing time of 15 minutes, the corrosion rate decreased by up to 85 %. Meanwhile, at anodizing time of 20 minutes, the corrosion rate only reduced by 19 %. The corrosion rate also decreased with an anodizing voltage of 15 volts and anodizing time of 10 minutes, which was 54 %. Anodizing time of 15 minutes reduced the corrosion rate by up to 74 %, while at 20 minutes, the corrosion rate decreased by 76 % compared to the raw material.

5. 2. Potentiodynamic polarization on aluminum AA2024 with boric sulfuric acid anodizing

The results of the electrochemical polarization observations are depicted as a Tafel curve plot shown in Fig. 5. At an anodizing voltage of 10 volts (Fig. 5, *a*), there are two types of specimens: specimens without sealing and samples with sealing. Variations in the anodizing time used are 10 minutes, 15 minutes, and 20 minutes. There is an increase in potential in the sealed specimen compared to the non-sealed specimen. Meanwhile, the longer anodizing time will also increase the corrosion potential.

The graph mentioned in the text represents the corrosion rate of aluminum samples under different anodization conditions. The y-axis represents the potential corrosion (V), while the *x*-axis represents the current density (μA/cm^2). The graph shows that the corrosion rate of the samples decreases with increasing anodization voltage and time. Based on the graph above, an odd number with a solid line on the curve indicates an anodized sample without sealing, while an even number with a dotted line is an anodized sample with sealing. The results show that sealing the samples after the anodization process can significantly reduce the corrosion rate of aluminum. The difference between the solid and dotted lines for the same anodization conditions indicates the effect of sealing on the corrosion rate.

The study involved various corrosion parameters in analyzing the effects of anodization and sealing on aluminum. The results showed that at an anodizing voltage of 15 volts, the potential for corrosion increased for each specimen parameter without sealing, and the specimen with the seal was not significantly affected. Corrosion testing yielded various parameters, including potential corrosion (*Ecorr*), current density corrosion (*Icorr*), cathodic (β*c*), anodic (β*a*), Tafel slope, and (*IE*).

The potentiodynamic test results revealed that the Tafel plot using sealing is in the cathodic region where the stress increases, reducing the corrosion current density and indicating a decrease in the corrosion rate. The highest corrosion potential (Ecorr) value was –0.537 V obtained at the anodizing time of 10 minutes, while the lowest corrosion potential (*Ecorr*) value was –0.708 *V* with the anodizing time of 15 minutes. The highest and lowest values for the current density corrosion (*Icorr*) were –8.7E-05 and –2.87515, respectively, at 15 minutes of anodizing time. These results suggest that the anodizing and sealing processes can significantly affect the corrosion behavior of aluminum, and proper parameter selection is crucial to achieving optimal corrosion resistance.

It can be observed that the corrosion rate decreases as the anodization voltage and time increase, and the lowest corrosion rate is obtained at an anodization voltage of 10 V and an anodization time of 15 minutes. Additionally, samples anodized with a gasket have a lower corrosion rate than those without the gasket and substrate. This reduction in corrosion rate is attributed to the formation of an oxide layer on the surface of the aluminum during anodization, which is further sealed by acetic acid treatment, resulting in a smooth surface and increased corrosion resistance.

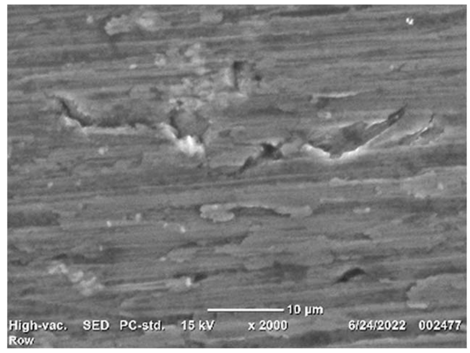
5. 3. Scanning electron microscopy and energy dispersive X-ray spectroscopy on aluminum AA2024 with boric sulfuric acid anodizing

The AA2024 aluminum alloy is characterized by a thin layer of oxide (Al_2O_3) prior to anodizing. The SEM images depicted in Fig. 6, *a, b* show that the surface of the aluminum alloy without anodizing appears flat. However, after undergoing the anodizing process, the surface layer becomes uneven and porous, as evident in the SEM images. This porous surface is a characteristic feature of materials that have undergone the anodizing process.

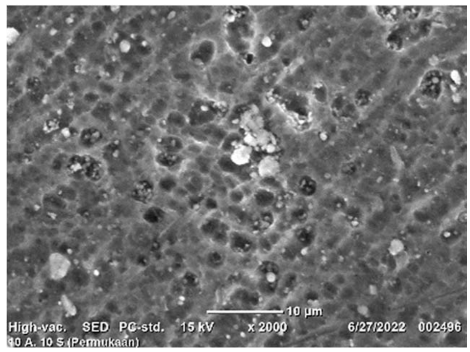
The pores shown in the photomicrographs are larger than those typically formed on anodized aluminum because they are not proper anodic pores but rather areas where the coating is not present or incomplete due to copper, zinc, iron, or their compounds with aluminum. These areas can be more susceptible to corrosion and may impact the overall effectiveness of the anodizing process in preventing corrosion. It would require further analysis and investigation to confirm the exact cause of these larger pores.

The aluminum oxide layer significantly formed influences the corrosion resistance properties of the AA2024 aluminum alloy. However, if the resulting pores are too large or exceed their optimum size, the corrosion resistance will not be optimal or will increase again. To minimize the resulting pores, a 50 g/l vinegar/acetic acid solution was used to close the open pores in the oxide layer from the anodization process to reduce the pores formed. Helpful seals are performed. The results are better where it can be seen that the surface of the specimen is smoother when sealing is done, as shown in Fig. 7, *a*. Meanwhile, if sealing is not carried out, the surface of the specimen will be rougher, as shown in Fig. 7, *b*.

The SEM-EDS test is required to determine the formation of an oxide layer in the anodizing process, where its main function is used to obtain information on topography and morphology. Topography is the surface and textural characteristics of the specimen. Meanwhile, morphology is the shape and size of the particles making up the object and composition, namely the semi-quantitative data of the elements and compounds contained in the anodizing process. Fig. 8 shows the EDS results on specimens without and with seals.

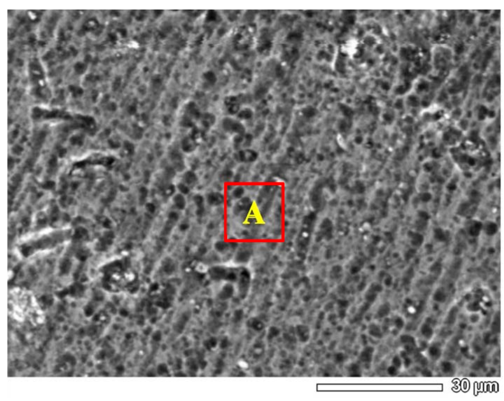


a

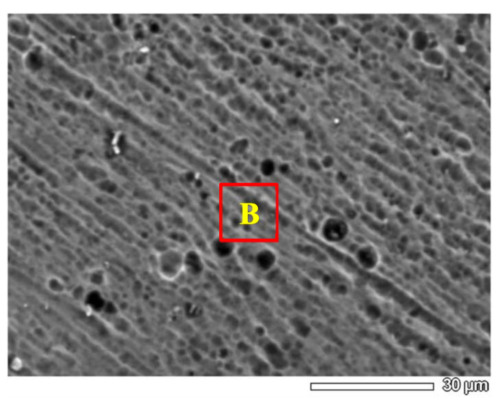


b

Fig. 6. Scanning electron microscopy micrography for a specimen: *a* – without anodizing; *b* – with anodizing



a



b

Fig. 7. Scanning electron microscopy micrography for a specimen: *a* – without sealing; *b* – with sealing

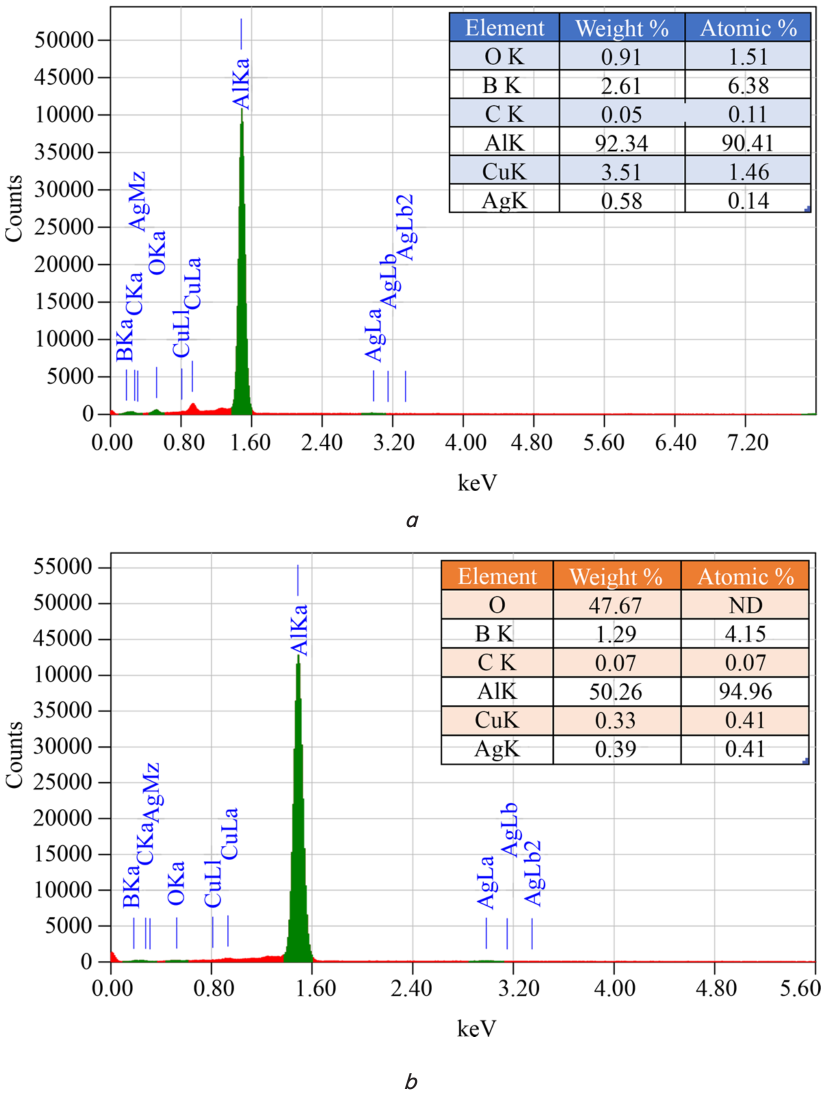


Fig. 8. Energy dispersive x-ray spectroscopy analysis of the selected area: *a* — without sealing; *b* — with sealing

The EDS test conducted on the samples provided valuable information on the composition of the aluminum oxide layer formed after the anodizing process. As expected, the results confirmed that the layer consisted of pure aluminum and oxygen. In addition, as shown in Fig. 8, *a*, the EDS spectrum revealed peaks corresponding to aluminum, oxygen, and trace amounts of other elements such as copper and zinc. These elements likely originate from the underlying aluminum alloy substrate.

Furthermore, Fig. 8, *b* shows that the elements in the oxide layer form oxide compounds, which are in an oxide bond state. This is consistent with the expected formation of a dense and uniform aluminum oxide layer during the anodizing process. The results of the EDS test support the effectiveness of the anodizing process in producing a stable and uniform oxide layer on the surface of the aluminum alloy substrate.

6. Discussion of the experiment corrosion rate using boric sulfuric acid anodizing of AA2024

Further analysis of the preliminary test results indicates that sealing has a significant impact on the corrosion rate of AA2024. Corrosion rate tests were carried out on the unsealed and sealed anodized samples and the base material based on Fig. 4. The anodizing time was varied for three samples, namely 10, 15, and 20 minutes. In addition, two different anodizing voltages, namely 10 volts and 15 volts, were used. The anodizing process causes the formation of pores on the aluminum layer, resulting in a decrease in the corrosion rate of the specimen. The size of the pores is influenced by the anodizing stress and process time. The results of this study suggest that an anodizing voltage of 10 volts and an anodizing time of 10 or 15 minutes are more effective in reducing the corrosion rate than an anodizing voltage of 15 volts (Fig. 4, *a*). However, an anodizing time of 20 minutes combined with an anodizing voltage of 15 volts is more effective in reducing the corrosion rate (Fig. 4, *b*). Another study [29] found that anodizing stress affects the formation of pores in the material, with a higher anodizing stress resulting in larger pore geometry. Therefore, the decrease in the corrosion rate in this study can be attributed to the increased formation of pores with the use of a 10 V anodizing voltage. Additionally, it is likely that longer anodizing times will lead to the formation of larger pores [30].

In addition to the improvement in corrosion potential observed in the anodizing samples, the study also found that sealing the anodized specimens resulted in a slightly higher corrosion potential compared to unsealed samples based on Fig. 5. However, increasing the anodizing stress may also increase the corrosion potential of the material. Despite this, the tested parameters all demonstrated a higher corrosion potential, indicating that the addition of sealing after anodizing can effectively enhance the corrosion resistance of the material. It is worth noting that previous research has also shown that applying the BSAA process to AA2024 can lead to a lower current density in anodized samples [21], further supporting the positive effects of anodizing on the corrosion resistance of the material.

The formation of aluminum oxide layer during the anodizing process is determined by the size and number of pores present in the material (Fig. 6, *a*). However, the surface microstructure of the aluminum oxide layer formed is observed to be uneven. This unevenness is attributed to the excessive current and energy that cause decay of the aluminum oxide layer back into the electrolyte solution. This decay results in the formation of pores in the aluminum oxide layer that are too large, leading to its unevenness. As a result, the aluminum oxide layer cannot be detected on the base material using SEM or EDX. However, after the anodizing process, aluminum oxide pores are visible, which vary in size and number and are not evenly distributed, but the thickness of the oxide formed increases (Fig. 6, *b*). The presence of these pores explains how the aluminum content decreases after the anodizing process. In addition, the quality of anodized aluminum is also affected by the potential difference provided. A significant potential difference affects the width and thickness of the aluminum oxide pores formed, leading to variations in the resulting material's properties [31].

Aluminum anodic coatings are widely used in aviation technology for various purposes, such as corrosion resistance, wear resistance, and electrical insulation. These coatings can provide a protective barrier that prevents corrosion and prolongs the lifespan of aircraft components, especially those exposed to harsh environmental conditions such as moisture and salt water. In addition, anodized aluminum can improve the adhesion of paints and other coatings, making it a popular choice for aircraft exterior and interior parts. Anodizing can also increase the hardness and durability of aluminum, making it more resistant to scratches, abrasion, and wear. Overall, using aluminum anodic coatings in aviation technology can improve the safety, reliability, and efficiency of aircraft operation.

Iron, zinc, and copper in aluminum alloys are not subject to electrochemical oxidation during anodization. To address this, a pre-treatment process is typically employed to remove these impurities from the surface of the alloy. Additionally, the anodization process can be adjusted to produce a thicker and more porous oxide layer on the surface of the alloy, which can provide increased protection against corrosion (Fig. 7, *a*). Regarding the issue of porosity, it is possible that some areas of the alloy containing phases or intermetallics of aluminum with copper are not fully anodized, leading to variations in the porosity of the surface (Fig. 7, *b*). This can result in uneven corrosion resistance across the surface of the alloy. As for the reported decrease in corrosion rate, it is important to note that anodization is not a foolproof solution for corrosion protection, and it is possible that the oxide film does not cover the entire aluminum alloy surface. Therefore, while anodization can significantly reduce the corrosion rate, it may not eliminate it completely.

In spite of the valuable insights gained from this study, it should be noted that there are still some limitations that need to be addressed. One of the main limitations is that the test samples were only taken once, whereas it is generally recommended to collect samples at least three times to ensure the accuracy of the results. This is particularly important for validating the test data. Additionally, SEM and EDS tests were only performed for one of the study parameters, which means that the SEM and EDS results are only applicable to the tested parameters. Other parameters may exhibit different properties when tested. Thus, including SEM and EDS test results for other parameters can further enhance the discussion and results of this study.

7. Conclusions

1. Anodizing treatment was carried out on aluminum with the BSAA process without sealing and has been successfully carried out with sealing. The decrease in corrosion rate was more significant for specimens with sealing. With an anodizing voltage of 10 V and an anodizing time of 15 minutes, the corrosion rate decreased by up to 85 % compared to the base material.

2. Potentiodynamic polarization indicates a difference between unsealed and sealed specimens. An increase in the corrosion potential occurs in specimens with sealing. The taffel plot shows sealing in the cathodic region where the stress increases, thereby reducing the corrosion current density.

3. SEM testing showed that the specimens with anodized surface experienced a porous surface, while the sealing treatment provided an oxide layer and a smoother surface. The EDS results also indicated that oxide compounds were formed on the specimens by sealing.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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Data availability

Data will be made available on reasonable request.

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References

1. Prasad, N. E., Wanhill, R. J. H. (Eds.) (2017). Aerospace Materials and Material Technologies. Springer. doi: https://doi.org/10.1007/978-981-10-2134-3
2. Tunes, M. A., Stemper, L., Greaves, G., Uggowitzer, P. J., Pogatscher, S. (2020). Prototypic Lightweight Alloy Design for Stellar-Radiation Environments. Advanced Science, 7 (22), 2002397. doi: https://doi.org/10.1002/advs.202002397
3. Iglesias-Rubianes, L., Garcia-Vergara, S. J., Skeldon, P., Thompson, G. E., Ferguson, J., Beneke, M. (2007). Cyclic oxidation processes during anodizing of Al–Cu alloys. Electrochimica Acta, 52 (24), 7148–7157. doi: https://doi.org/10.1016/j.electacta.2007.05.052
4. Hamsir, H., Sutresman, O., Arsyad, H., Syahid, M., Widyianto, A. (2022). Suppression of corrosion on stainless steel 303 with automatic impressed current cathodic protection (a-ICCP) method in simulated seawater. Eastern-European Journal of Enterprise Technologies, 6 (12 (120)), 13–21. doi: https://doi.org/10.15587/1729-4061.2022.267264

5. Subbotina, V., Sobol, O., Belozerov, V., Subbotin, A., Smyrnova, Y. (2020). A study of the phase-structural engineering possibilities of coatings on D16 alloy during micro-arc oxidation in electrolytes of different types. Eastern-European Journal of Enterprise Technologies, 4 (12 (106)), 14–23. doi: https://doi.org/10.15587/1729-4061.2020.209722
6. Polmear, I. J. (2004). Aluminium Alloys – A Century of Age Hardening. Materials Forum. Available at: https://www.researchgate.net/publication/279898292_Aluminium_Alloys_-_A_Century_of_Age_Hardening
7. Bensalah, W., Feki, M., Wery, M., Ayedi, H. F. (2011). Chemical dissolution resistance of anodic oxide layers formed on aluminum. Transactions of Nonferrous Metals Society of China, 21 (7), 1673–1679. doi: https://doi.org/10.1016/s1003-6326(11)60913-8
8. Stevenson, M. F. (2013). Anodizing.
9. Fratila-Apachitei, L. E., Apachitei, I., Duszczyk, J. (2006). Thermal effects associated with hard anodizing of cast aluminum alloys. Journal of Applied Electrochemistry, 36 (4), 481–486. doi: https://doi.org/10.1007/s10800-005-9102-y
10. Poinern, G. E. J., Ali, N., Fawcett, D. (2011). Progress in Nano-Engineered Anodic Aluminum Oxide Membrane Development. Materials, 4 (3), 487–526. doi: https://doi.org/10.3390/ma4030487
11. Zhang, L., Thompson, G. E., Curioni, M., Skeldon, P. (2013). Anodizing of Aluminum in Sulfuric Acid/Boric Acid Mixed Electrolyte. Journal of The Electrochemical Society, 160 (4), C179–C184. doi: https://doi.org/10.1149/2.032306jes
12. Sanyal, S., Kim, T., Rabelo, M., Pham, D. P., Yi, J. (2022). Application of noble cerium-based anti-corrosion sealing coating approach applied on electrical insulators installed in industrial regions. Royal Society Open Science, 9 (4). doi: https://doi.org/10.1098/rsos.211786
13. Haruna, T., Ikeda, T., Miyazaki, M., Nishimoto, A., Hirohata, Y. (2015). Correlation between Bound Water and Stability of Anodic Oxide Film on Aluminum. MATERIALS TRANSACTIONS, 56 (12), 2000–2005. doi: https://doi.org/10.2320/matertrans.l-m2015831
14. Shen, Y. Z., Li, H. G., Tao, H. J., Ling, J., Wang, T., Tao, J. (2015). Effect of anodic films on corrosion resistance and fatigue crack initiator of 2060-T8 Al-Li alloy. International Journal of Electrochemical Science, 10 (1), 938–946. Available at: https://www.researchgate.net/publication/281996844_Effect_of_anodic_films_on_corrosion_resistance_and_fatigue_crack_initiator_of_2060-T8_Al-Li_alloy
15. Veys-Renaux, D., Chahboun, N., Rocca, E. (2016). Anodizing of multiphase aluminium alloys in sulfuric acid: in-situ electrochemical behaviour and oxide properties. Electrochimica Acta, 211, 1056–1065. doi: https://doi.org/10.1016/j.electacta.2016.06.131
16. Mukhurov, N. I., Zhvavyi, S. P., Terekhov, S. N., Panarin, A. Yu., Kotova, I. F., Pershukevich, P. P. et al. (2008). Influence of electrolyte composition on photoluminescent properties of anodic aluminum oxide. Journal of Applied Spectroscopy, 75 (2), 214–218. doi: https://doi.org/10.1007/s10812-008-9026-5
17. Pooladi, R., Rezaei, H., Aezami, M., Sayyar, M. R. (2009). Fabrication of anodic aluminum oxide nanotemplate and investigation of their anodization parameters. Transactions of the Indian Institute of Metals, 62 (3), 241–244. doi: https://doi.org/10.1007/s12666-009-0026-9
18. Fotovvati, B., Namdari, N., Dehghanghadikolaei, A. (2019). On Coating Techniques for Surface Protection: A Review. Journal of Manufacturing and Materials Processing, 3 (1), 28. doi: https://doi.org/10.3390/jmmp3010028
19. Wang, R., Wang, L., He, C., Lu, M., Sun, L. (2019). Studies on the sealing processes of corrosion resistant coatings formed on 2024 aluminium alloy with tartaric-sulfuric anodizing. Surface and Coatings Technology, 360, 369–375. doi: https://doi.org/10.1016/j.surfcoat.2018.12.092
20. Gonzalez, J. A., Lopez, V., Otero, E., Bautista, A., Lizarbe, R., Barba, C., Baldonedo, J. L. (1997). Overaging of sealed and unsealed aluminium oxide films. Corrosion Science, 39 (6), 1109–1118. doi: https://doi.org/10.1016/s0010-938x(97)00019-x
21. Yu, S., Wang, L., Wu, C., Feng, T., Cheng, Y., Bu, Z., Zhu, S. (2020). Studies on the corrosion performance of an effective and novel sealing anodic oxide coating. Journal of Alloys and Compounds, 817, 153257. https://doi.org/10.1016/j.jallcom.2019.153257 doi: https://doi.org/10.1016/j.jallcom.2019.153257
22. Zhang, L. M., Zhang, S. D., Ma, A. L., Hu, H. X., Zheng, Y. G., Yang, B. J., Wang, J. Q. (2018). Influence of sealing treatment on the corrosion behavior of HVAF sprayed Al-based amorphous/nanocrystalline coating. Surface and Coatings Technology, 353, 263–273. doi: https://doi.org/10.1016/j.surfcoat.2018.08.086
23. Hu, N., Dong, X., He, X., Browning, J. F., Schaefer, D. W. (2015). Effect of sealing on the morphology of anodized aluminum oxide. Corrosion Science, 97, 17–24. doi: https://doi.org/10.1016/j.corsci.2015.03.021
24. Kocabaş, M., Örnek, C., Curioni, M., Cansever, N. (2019). Nickel fluoride as a surface activation agent for electroless nickel coating of anodized AA1050 aluminum alloy. Surface and Coatings Technology, 364, 231–238. doi: https://doi.org/10.1016/j.surfcoat.2019.03.003
25. Hao, X.-L., Zhao, N., Jin, H.-H., Ma, W., Zhang, D.-H. (2020). Nickel-free sealing technology for anodic oxidation film of aluminum alloy at room temperature. Rare Metals, 40 (4), 968–974. doi: https://doi.org/10.1007/s12598-020-01410-8
26. Whelan, M., Cassidy, J., Duffy, B. (2013). Sol–gel sealing characteristics for corrosion resistance of anodised aluminium. Surface and Coatings Technology, 235, 86–96. doi: https://doi.org/10.1016/j.surfcoat.2013.07.018

27. Capelossi, V. R., Poelman, M., Recloux, I., Hernandez, R. P. B., de Melo, H. G., Olivier, M. G. (2014). Corrosion protection of clad 2024 aluminum alloy anodized in tartaric-sulfuric acid bath and protected with hybrid sol–gel coating. Electrochimica Acta, 124, 69–79. doi: https://doi.org/10.1016/j.electacta.2013.09.004
28. Huang, T.-C., Lin, C.-Y., Liao, K.-C. (2022). Sealing performance assessments of PTFE rotary lip seals based on the elasto-hydrodynamic analysis with the modified archard wear model. Tribology International, 176, 107917. doi: https://doi.org/10.1016/j.triboint.2022.107917
29. Abd-Elnaiem, A. M., Abbady, G., Ali, D., Asafa, T. B. (2019). Influence of anodizing voltage and electrolyte concentration on Al-1 wt% Si thin films anodized in H2SO4. Materials Research Express, 6 (8), 086468. doi: https://doi.org/10.1088/2053-1591/ab2848
30. Park, J., Son, K., Lee, J., Kim, D., Chung, W. (2021). Effects of Anodizing Conditions on Thermal Properties of Al 20XX Alloys for Aircraft. Symmetry, 13 (3), 433. doi: https://doi.org/10.3390/sym13030433
31. Araoyinbo, A. O., Noor, A. F. M., Sreekantan, S., Aziz, A. (2010). Voltage Effect on Electrochemical Anodization of Aluminum at Ambient Temperature. International Journal of Mechanical and Materials Engineering, 5 (1), 53–58. Available at: https://www.researchgate.net/publication/265811751_Voltage_effect_on_electrochemical_anodization_of_aluminum_at_ambient_temperature