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Tribological Characteristics of Pulse-Anodized Layers on Aluminum

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Abstract

This study develops advanced electrochemical synthesis methods for high-performance hard anodic coatings on Al-Cu-Mg alloy (AA2024/D16) substrates. The research systematically investigates the synergistic effects of chemical electrolyte modification via hydrogen peroxide or ozone additions, high-voltage pulse hard anodizing (PHA) modes, and post-synthetic thermal treatments on the coating kinetics and phase composition. It was established that lowering the electrolyte temperature to -5 °C suppresses acid-induced chemical surface dissolution, promoting the steady growth of a dense monohydrated boehmite phase ($Al_2O_3 \cdot H_2O$) with significantly enhanced microhardness. Conversely, elevating the synthesis temperature above 0 °C triggers a structural phase transition toward a trihydrated gibbsite phase ($Al_2O_3 \cdot 3H_2O$). Tribological evaluations under dry sliding and boundary lubrication conditions (mineral and synthetic oils) revealed a unique self-lubricating mechanism: the lower-hardness gibbsite phase undergoes mechanical shearing during sliding contact and forms a continuous, self-replenishing tribo-transfer solid lubricant film on both steel and ceramic counterfaces. Furthermore, a post-synthetic thermal treatment within 100–400 °C followed by hydrothermal boiling induces target matrix dehydration, doubling the coating's abrasive wear resistance and providing a 5- to 10-fold wear reduction relative to the untreated AA2024 aluminum substrate. The pulse modulation effectively mitigates local thermal concentrations within the pore channels, enabling optimal tribomechanical responses under diverse wear modes. Objective: To enhance the durability of aluminum alloys using surface engineering methods. Research methodology: A novel approach involving pulse-mode anodizing was employed to form a surface layer on the aluminum alloy; the physicochemical properties and wear resistance of this layer under friction conditions were investigated. Scientific novelty: Synthesis conditions and electrolyte composition were determined for forming an anodized layer with a specific phase composition. Practical significance: Pulse-mode anodizing and heat treatment ensure improved wear resistance of the aluminum alloy.

Keywords: hard anodizing; aluminum alloys; boehmite; gibbsite; thermal treatment; tribo-transfer film; boundary lubrication, friction.

Introduction: Problem Statement and Signifigance

Aluminum and its alloys are extensively utilized across diverse industrial sectors for modern equipment manufacturing. They serve as critical structural materials in aerospace and aviation vehicles, automotive engineering, rail and maritime transport, and general machinery. However, their inherently low wear resistance and poor tribological performance severely restrict wider industrial application. Currently, the deployment of aluminum alloys has become essential for unmanned aerial vehicle (UAV) manufacturing. The rapid advancement of UAV technologies demands lightweight aluminum alloys (such as Al-Mg, Al-Cu-Mg, and Al-Si series) for critical components, including internal combustion engine elements, gimbal suspension mounting systems, transmission components, and gearbox housings. From a military perspective, drones operate under extreme environmental conditions, such as sandstorms, marine salt fog, and severe engine thermal loads. Conventional anodization techniques fail to provide sufficient protection against abrasive wear and fragmentation impacts. From a civilian standpoint, extending the operational lifespan and reducing structural weight by replacing steel components with surface-hardened aluminum significantly improves payload capacity and flight endurance. To enhance the physical, mechanical, and tribological properties of structural aluminum materials, high-voltage pulse

hard anodization (PHA) is widely adopted. Nevertheless, conventional pulse anodization exhibits significant drawbacks, including a low growth rate of theoxide/hydroxide layer, insufficient hardness, and suboptimal wear resistance. To improve the functional performance of the anodized layers, this study proposes modifying the sulfuric acid electrolyte with hydrogen peroxide H_2O_2 combined with post-synthetic thermal treatment of the formed anodic oxide layer.

Literature review

Pulse Hard Anodizing (PHA) and Self-Lubricating Tribo-Films. Conventional direct current (DC) anodizing frequently fails under extreme sliding conditions due to local thermal concentration inside the structural pores, which leads to macro-defects and micro-cracking. To eliminate this issue, Pulse Hard Anodizing (PHA) has emerged as a frontline technological breakthrough [1 - 5]. Current publications demonstrate that pulse current modulations provide a crucial "cooling relaxation time" during the off-period of the pulse cycle. This effectively mitigates spatial heat accumulation at the pore base, facilitating the uniform synthesis of a less-defective oxide matrix over extended processing periods. From a tribological standpoint, recent boundary lubrication and dry sliding investigations (published in Wear and Coatings) indicate a shifting paradigm: high hardness is no longer the sole requirement for wear mitigation. Researchers have discovered that when a hydrated phase like gibbsite participates in oscillatory sliding contact against steel or ceramic counterfaces, it undergoes controlled mechanical shearing [6 - 8]. This process leads to the deposition of a stable tribo-transfer film (solid lubricant) within the contact interface. This dynamic interfacial film undergoes continuous renewal, dramatically lowering the wear rate even when working under boundary lubrication with mineral or advanced synthetic oils [8 - 12].

Aim of the Research

The aim of this work is to establish the mechanisms governing parameter variations and to optimize the high-voltage pulse anodization process during the synthesis of hard anodic oxide layers on an AD0 aluminum alloy as a baseline material.

Materials and Methods

Flat specimens with dimensions of 20 × 20 × 5 mm were prepared from AA2024 alloy. Prior to the anodization process, the specimens underwent a multi-step surface pretreatment: *Degreasing:* Immersed in an aqueous suspension of CaO + MgO to remove organic contaminants. Rinsing: Washed sequentially in cold and warm tap water. *Desmutting:* Immersed for 30 s in an aqueous solution of nitric acid 400 g/L HNO_3 to remove intermetallic smuts. *Final Rinsing:* Thoroughly rinsed in distilled water and dried. Anodization Setup and Electrochemical Modes. The oxide layers were synthesized using custom-designed electrochemical equipment (Fig. 1a). A 20 wt % aqueous solution of sulfuric acid H_2SO_4 prepared with distilled water served as the base electrolyte. To investigate the effect of temperature conditions, the electrolyte temperature in the setup was maintained within the range of -5°C to +5°C with an accuracy of ±0.1°C. The total duration of the anodization process was 60 min for all experiments. Anodization was performed in two distinct electrical modes (Fig. 1b): Steady-State (Direct Current) Mode (II): Conducted at a stabilized current density of 5 A/dm^2 with a constant voltage of V = 60 V. High-Voltage Pulse Mode (I–III): Executed in cycles consisting of three steps:

Step I (Pulse Initiation): High-voltage pulse stage with V = 140 V and an instantaneous current density of I = 1.4 A/dm^2.

Step II (Main Working Period): Base anodization stage with V = 40 V and I = 0.4 A/dm$_2$.

Step III (Pulse Pause): Off-period where both voltage V and current I were reduced to zero.

Troughout the pulse cycling, the effective average current density was maintained at 5 A/dm^2 via a specialized control unit.

Electrolyte Modification and Process Intensification. To accelerate the layer growth rate and enhance the mechanical performance (hardness and wear resistance) of the coatings, the baseline sulfuric acid electrolyte was modified using two approaches: *Chemical modification:* Addition of hydrogen peroxide H_2O_2 in concentrations of 30, 50, 70, and 100 ml/l. Ozone gas sparging: Continuous bubbling of an ozone-air mixture through the electrolyte at a constant ozone flow rate of 5 mg/min/L.

Microstructural and Physical Characterization. The microstructure, surface morphology, and elemental composition of the synthesized oxide layers and deposited coatings were analyzed using: Optical Microscopy Neophot 22 metallographic microscope, Scanning Electron Microscopy (SEM) Carl Zeiss EVO-40XVP system operated in high-vacuum mode. Energy-Dispersive X-ray Spectroscopy (EDS): INCA Energy 350 microanalysis system coupled with the SEM for elemental profiling. X-ray Diffraction (XRD**):** DRON-3 diffractometer to determine the phase composition of the oxide scales.

Tribological Testing. The mechanical and functional performance of the synthesized layers was evaluated through comprehensive tribological testing according to standardized methods. The wear resistance was characterized under the following contact conditions: Three-body abrasive wear: Testing with loose/unfixed abrasive particles. Two-body abrasive wear: Testing against fixed abrasive media. Oscillatory sliding wear

(Fretting/Reciprocating): Evaluated under dry friction (unlubricated) conditions as well as in the presence of various industrial lubricants.

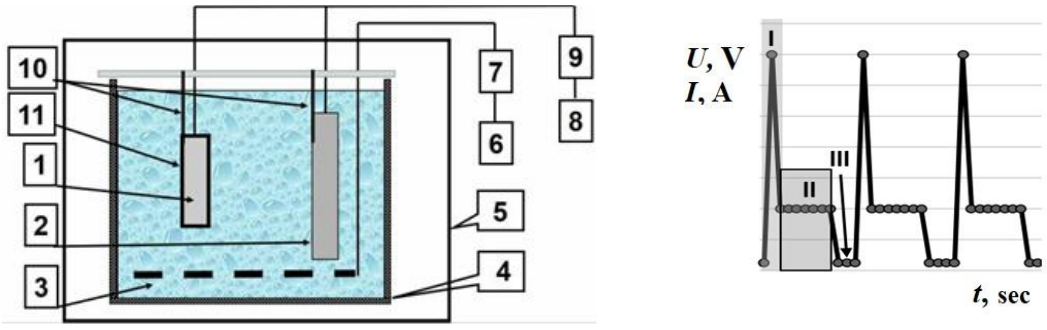

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Fig. 1. Schematic representation of the experimental setup and electrical parameters: (a) Schematic diagram of the custom pulse anodization equipment: *1*—specimen (anode); *2*—counter-electrode (cathode); *3*—gas sparger (bubbler); *4*—electrolyte tank 20 wt.% H_2SO_4 solution); *5*—thermal stabilization chamber (operational range from -4 °C to +8 °C ; *6*—air compressor; *7*—flow control unit for the compressor and sparger; *8*—power supply (maintaining an average current density of 5 A/dm$_2$; *9*—pulse modulation and power control unit; *10*—electrode and specimen fixture; *11*—synthesized anodic oxide layer. (b) Schematic profile of voltage U and current I variations over time t during the high-voltage pulse anodization cycle

Results and Discussion

Microstructure and Morphological Features of the Anodic Layers

The synthesized anodic oxide layers characteristically consist of a thin, dense inner barrier layer 10—20 nm in thickness and a porous outer layer with fine, vertically oriented pores extending to the surface (Fig. 2). The highly packed hexagonal columnar cells, each containing a cylindrical pore channel at its center, are composed of heavily hydrated aluminum oxide $Al_2O_3•nH_2O$. The hydration degree (the number of water molecules, n highly depends on the electrochemical synthesis mode and varies from 1 to 3. Specifically, for the layers formed in the sulfuric acid electrolyte, the structural parameters of the hexagonal cells do not exceed 50 nm, the pore diameters are limited to approximately 25 nm, and the barrier layer thickness ranges within 10-30 nm.

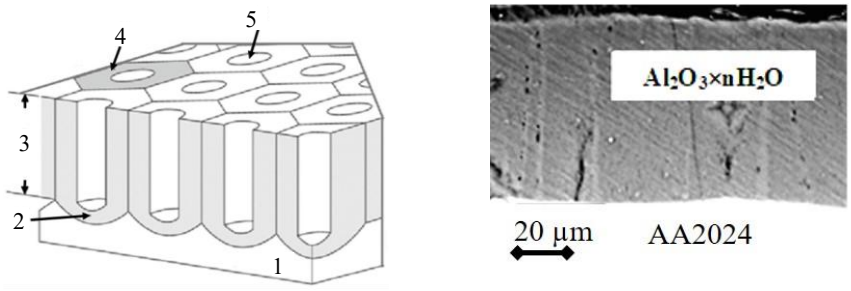

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Fig. 2 Morphological model of the anodic film formation: (a) Schematic diagram of pore development: *1*—aluminum substrate, *2*—barrier layer, *3*—barrier layer thickness, *4*—porous anodic layer, *5*—pore channel; (b) Cross-sectional structural model of the hexagonal cellular oxide matrix

Effect of Electrolyte Composition and Oxidation Mechanisms

Experimental data revealed that the addition of hydrogen peroxide H_2O_2 to the baseline electrolyte, as well as ozone O_3 gas sparging, significantly increases both the total thickness and the microhardness of the synthesized anodic coatings. By increasing the H_2O_2 concentration in the sulfuric acid bath from 0 to 70 mL/L, the coating thickness expanded by 33%, growing from approximately (60 to 100 mum (Fig. 3a). Concurrently, the coating microhardness enhanced by 25%, rising from 400 to 500 $HV_{0.3}$. (Fig. 3b). The most pronounced peak in mechanical performance was achieved at an H_2O_2 content of 30 mL/L, where the abrasive wear resistance of the pulse-anodized layer improved by 23%, as evidenced by the lowest mass loss values (Fig. 3b). The simultaneous growth of coating thickness and microhardness during hard anodization (HA) with H_2O_2 modification or O_3 sparging can be attributed to the altered kinetics of oxygen species transport. The introduction of these powerful oxidizing agents enriches the pore channels with active oxygen ions according to the following decomposition and ionization pathways: : ($H_2O_2 \rightarrow 2H^{+2} + 2O^{-2}$, або $O_3 \rightarrow O_2 + O^-$). The elevated concentration of highly reactive oxygen ions promotes a structural transition within the aluminum hydroxide/oxide matrix. It drives the dehydration process, reducing the number of chemically bound water molecules in the film from three to one $Al_2O_3·3H_2O$ (Gibbsite/Bayerite)→ $Al_2O_3·H_2O$ (Boehmit). This partial dehydration yields a more compact, dense, and

crystalline bohmite-like or low-hydrated alumina structure, which directly accounts for the drastic improvement in hardness and abrasive wear resistance.

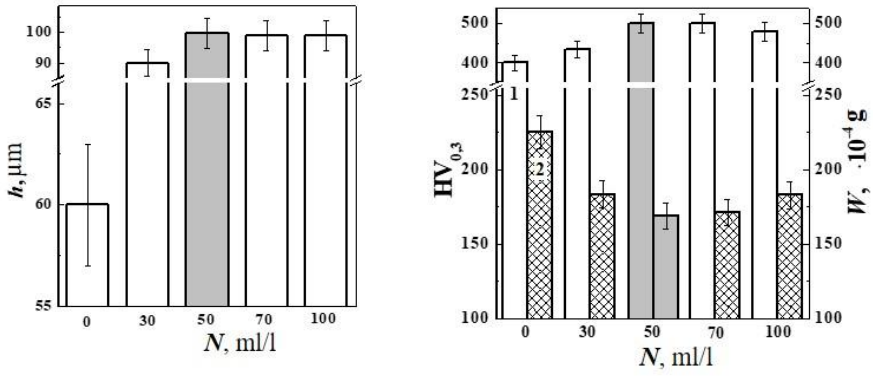

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Fig. 3. Influence of the hydrogen peroxide concentration n in the electrolyte (at a fixed synthesis duration of 60 min on the coating properties: (a) **Anodic layer thickness h;** (b) **Layer microhardness $HV_{0.3}$ (*1*) and mass loss W during standardized abrasive wear resistance tests (*2*)**

Post-Synthetic Thermal Treatment of Anodic Layers

Post-synthetic thermal treatment of the anodized layers induced further dehydration of the oxide matrix, driving a significant reduction in the chemically bound water molecules from three to one n = 3→1. Specifically, as the heat treatment temperature was elevated up to 400 C, the coating microhardness enhanced from the initial 400 HV0.3 and stabilized at approximately 650 $HV_{0.3}$. Concurrently, this structural alteration led to a nearly 2.5-fold increase in abrasive wear resistance (Fig. 4).

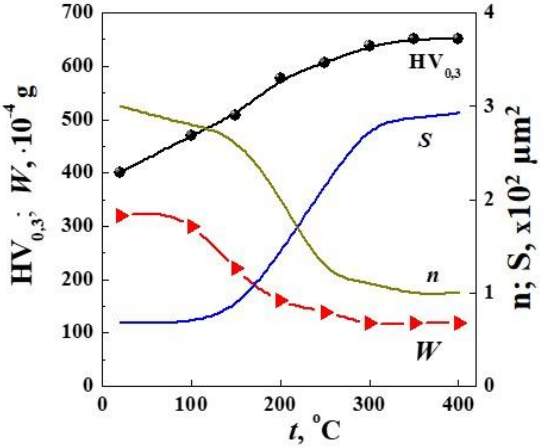

Fig. 4. Influence of the thermal treatment temperature T on the water molecule content n within the aluminum hydroxide matrix, coating microhardness $HV_{0.3}$, mass loss W during standardized abrasive wear tests, and the wear track cross-sectional area S after frictional wear testing.

Comparative Analysis of Steady-State and Pulse Hard Anodization Kinetic Features

The kinetics of the anodization process played a decisive role in determining the final coating properties. It was established that regardless of the electrochemical mode applied (steady-state direct current or high-voltage pulse mode), increasing the synthesis duration led to a substantial improvement in both the coating microhardness (Fig. 5a) and the corresponding abrasive wear resistance of the AA2024 (D16) aluminum alloy substrate (Fig. 5b).

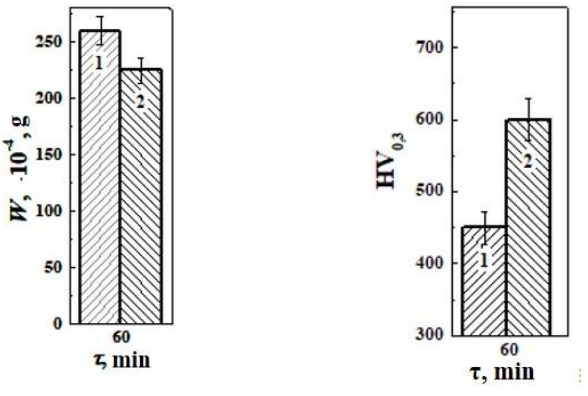

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Fig. 5. Comparative effect of steady-state (1) and high-voltage pulse (2) anodization modes as a function of processing time on: (a) **microhardness $HV_{0.3}$;** (b) **mass loss W of the anodized AA2024 (D16) aluminum alloy specimens during standardized abrasive wear resistance testing.**

Notably, the positive correlation between the synthesis duration and these functional parameters was significantly more pronounced when the high-voltage pulse mode was employed. The pulse modulation effectively mitigated local thermal concentrations within the pore channels, enabling the steady growth of a denser, less defective, and more wear-resistant crystalline oxide structure over extended treatment times compared to the conventional steady-state method.

Effect of Electrolyte Temperature on the Thickness, Phase Composition, and Tribological Behavior of the Coatings

It was established that conducting the anodization process at a lower electrolyte temperature -5ºC promotes the synthesis of oxide layers with enhanced microhardness due to the suppressed rate of chemical dissolution. Conversely, elevating the electrolyte temperature from -5 C to +5 C accelerated the dissolution processes, which led to a simultaneous reduction in both the total coating thickness (Fig. 6a) and its microhardness (Fig. 6b). X-ray diffraction and phase analysis revealed that coatings synthesized at lower temperatures 0 ºC and -5 ºC were dominated by the boehmite phase $Al_2O_3•H_2O$, containing a single chemically bound water molecule. As the electrolyte temperature was increased to +5 ºC, a phase transition phase coexistence was observed, with the dominant phase shifting toward gibbsite $Al_2O_3•3H_2O$, which locks three water molecules into its crystal lattice. For both the steady-state (1) and high-voltage pulse (2) hard anodization modes, decreasing the electrolyte temperature from +5 ºC to -5 ºC yielded an increase in the final coating thickness h (Fig. 6a). Specifically, the coating thickness increased by 12% for the steady-state mode and by 23% for the pulse mode under condition (II). This temperature-dependent behavior of the coating thickness is governed by the competitive kinetics between electrochemical oxide synthesis and chemical dissolution by the acid electrolyte. Theoretically, an increase in electrolyte temperature intensifies ionic transport, which should accelerate oxide growth. However, it simultaneously accelerates the aggressive surface dissolution of the formed anodic layer, leading to its thinning. Therefore, the observed thickness profiles reflect the synergistic interplay between accelerated electrochemical synthesis and the aggressive acid-induced dissolution that becomes dominant at temperatures above 0 ºC. The results demonstrated that the pulse-anodized layer featuring the monohydrate boehmite structure $Al_2O_3•H_2O$, synthesized at -5 ºC, exhibited the highest microhardness $HV_{0.3}$ and the maximum abrasive wear resistance 1/W (Fig. 6b). On the contrary, the coating containing the trihydrate gibbsite structure $Al_2O_3•3H_2O$, synthesized at +5 ºC, demonstrated the highest frictional wear resistance 1/S during dry sliding interaction against a steel ball counterface. Notably, an identical performance trend was maintained when evaluating the tribological pair with a ceramic ball counterface, proving that the chemical structure of the oxide layer dictates its tribomechanical response under different wear modes.

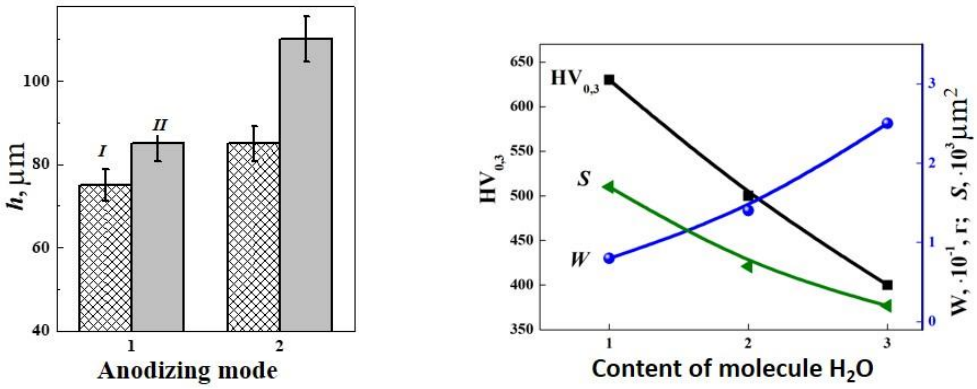

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Fig. 6. Effect of electrolyte temperature during a 60-min hard anodization process under steady-state (1) and high-voltage pulse (2) modes: (a) coating thickness h obtained at +5 oC (I) and -5 oC (II); (b) correlation between the water molecule content (n) within the anodic matrix and its microhardness HV0.3, abrasive wear resistance 1/W, and frictional wear resistance 1/S

Tribological Behavior under Boundary Lubrication and the Tribo-Transfer Mechanism

The distinct variations in the microhardness and abrasive wear resistance of the pulse hard anodized layers (PHAL) as a function of the chemically bound water content are closely related to their dehydration profiles and structural transitions. To evaluate this performance, the sliding wear tracks of the anodic layers synthesized via steady-state and high-voltage pulse modes at an electrolyte temperature of -5 ºC were compared against a steel ball counterface under diverse lubrication regimes.

The experimental data revealed that under all tested conditions—including dry sliding, boundary lubrication with mineral oil (I-20), and lubrication with synthetic oil (EDGE 5W-30)—the PHAL specimens consistently exhibited the lowest volumetric wear (Fig. 7). Depending on the specific friction pair and the operational parameters of the oscillatory sliding, the wear resistance of the coatings synthesized in the pulse mode increased by a factor of 2 to 19 compared to the untreated AA2024 aluminum alloy substrate, and by 1.4 to 5.8 times relative to the coatings formed under conventional steady-state direct current. Surface morphology analysis

indicated that the steel counterface caused negligible abrasive wear to the anodized layer. Instead, it primarily induced the smoothing of initial machining asperities (Fig. 8). During this process, the outer hydrated oxide layer $Al_2O_3 \cdot nH_2O$ was mechanically sheared and transferred, acting as a solid lubricant film deposited onto the contacting surfaces of either the steel or ceramic ball counterfaces.

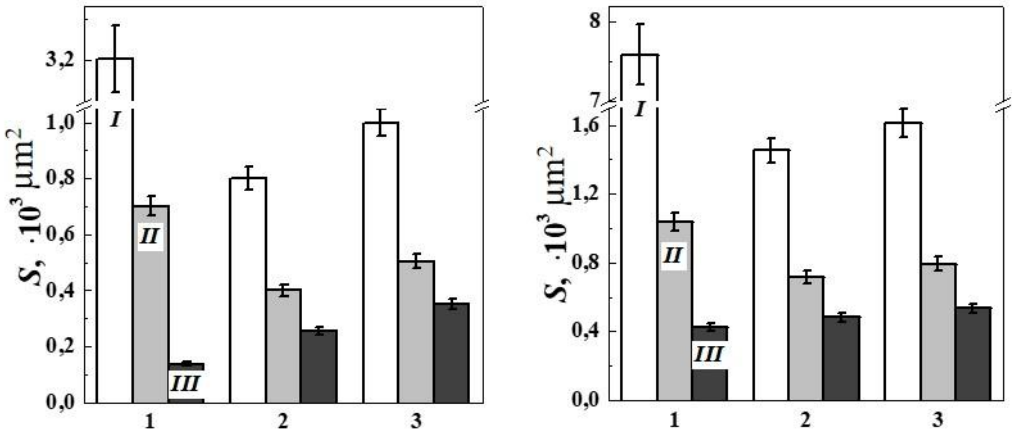

Fig. 7. Frictional wear profiles of the AA2024 alloy substrate without coating (I), with a steady-state anodic coating (II), and with a high-voltage pulse anodic coating (III) under oscillatory sliding conditions against steel (a) and ceramic (b) ball counterfaces tested: (1) without lubrication (dry sliding); (2) under mineral oil (I-20) lubrication; (3) under synthetic oil (EDGE 5W-30) lubrication.

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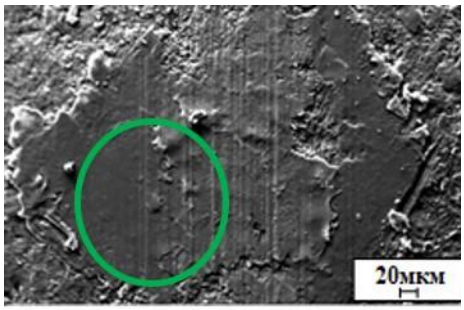

Element	Weight %	Atomic %
O	1.22	4.12
Al	0.43	0.87
Cr	1.74	1.80
Fe	96.61	93.22
Total	100.00	

Fig. 8. Tribotransfer gibbsite film and its corresponding energy-dispersive X-ray spectroscopy (EDS) elemental analysis on the wear scar surface of the ceramic ball counterface

Discussion of the Structural-Tribological Synthesis Mechanisms

Energy-dispersive X-ray spectroscopy (EDS) confirmed the formation of a continuous gibbsite-rich tribo-film on the friction surfaces of both the steel and ceramic counterfaces. This evidence suggests that a dynamic, self-lubricating interfacial film forms directly within the contact zone. Instead of undergoing work-hardening or severe delamination during cyclic sliding, this film undergoes continuous tribochemical renewal and replenishment.

In summary, the abrasive wear resistance and overall tribological behavior of the anodized layers are heavily dictated by the specific hydration state (the number of chemically bound water molecules) within the oxide matrix:

- The boehmite phase $Al_2O_3 \cdot H_2O$ provides the coatings with high microhardness and superior resistance to harsh abrasive scratching.
- The gibbsite phase $Al_2O_3 \cdot 3H_2O$ provides excellent self-lubricating capability, optimizing the friction coefficient and mitigating wear during sliding contact.

Crucially, the concentration of chemically bound water molecules in the aluminum hydroxide matrix $Al_2O_3 \cdot nH_2O$ can be precisely tailored from n = 1 (boehmite) to n = 3 (gibbsite) via three independent processing routes:

- Chemical modification**:** Modulating the electrolyte composition by introducing H_2O_2 in concentrations ranging from 10 to 50 mL/L.
- Thermal control during synthesis**:** Adjusting the electrolyte operational temperature within the range of -5 ºC to +5 ºC.
- Post-synthetic thermal treatment**:** Subjecting the synthesized anodic coatings to controlled heat treatment within a temperature range of 100 ºC to 300 ºC.

Conclusions

A novel electrolyte modification method for synthesizing hard anodic coatings (HACs) on commercially pure aluminum **(**AA2024**)** was developed. The approach involves introducing 3–5% hydrogen peroxide H_2O_2 into a base electrolyte of a 20% aqueous sulfuric acid H_2SO_4 solution or systematically purging the bath with ozone O_3. Implementing this oxidizing method effectively enhanced the coating growth kinetics and structural density, resulting in a 50–60% increase in total coating thickness**, a** 40–50% enhancement in microhardness**, a** 15% improvement in abrasive wear resistance**,** and a 30% reduction in wear volume when operating within a sliding friction pair against a bearing steel ball counterface.

A comprehensive post-synthetic thermal treatment protocol for the developed anodic layers was proposed. The process consists of controlled thermal exposure within a 100–400 °C range, followed by subsequent sealing via hydrothermal boiling in distilled water. This specific post-treatment sequence stimulates target dehydration and phase transition, driving the formation of a dense boehmite phase $Al_2O_3•H_2O$ within the oxide matrix. Consequently, this phase stabilization doubled the abrasive wear resistance of the coatings and provided a 5- to 10-fold increase in wear resistance compared to the untreated AA2024 (D16) aluminum alloy benchmarks.

A high-voltage pulse hard anodizing (PHA) technique was developed to actively regulate the crystal structure of the anodic matrix, enabling the precise synthesis of either a single-phase oxide or a multi-phase structural mixture. The phase distribution is dynamically controlled by adjusting the electrolyte operating temperature within a -5 °C to +10 °C range**.** Within this system:

The monohydrated boehmite phase yields maximum coating microhardness and superior resistance to abrasive scratching.

The trihydrated gibbsite phase $Al_2O_3•3H_2O$ provides exceptional self-lubricating tribological behavior. This is due to the dynamic formation and continuous replenishment of a highly stable gibbsite-rich tribo-transfer film directly within the contact zone when sliding against steel or ceramic counterfaces.

The coatings synthesized via this PHA method demonstrated a 15–20% higher thickness and a 1.5- to 3-fold higher wear resistance than those produced via conventional steady-state direct current modes, while outperforming the untreated AA2024 (D16) substrate by 2.5 to 8 times**.**

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Гвоздецький В., Яремчук Д. Трибологічні характеристики імпульсно-анодованих шарів на алюмінії

У дослідженні розроблено передові електрохімічні методи синтезу високоефективних твердих анодних покриттів на підкладках з комерційно чистого алюмінію (AA1050/AD0) та сплаву Al-Cu-Mg (AA2024/D16). Систематично встановлено синергетичний вплив хімічної модифікації електроліту шляхом додавання перекису водню або озону, режимів високовольтного імпульсного твердого анодування (РНА) та постсинтетичної термічної обробки на кінетику покриття та фазовий склад. Встановлено, що зниження температури електроліту до -5 °C пригнічує індуковане кислотою хімічне розчинення поверхні, сприяючи стабільному росту щільної моногідратованої фази беміту ($Al_2O_3 \cdot H_2O$) зі значно підвищеною мікротвердістю. Навпаки, підвищення температури синтезу вище 0 °C індукує структурний фазовий перехід до тригідратованої фази гібситу ($Al_2O_3 \cdot 3H_2O$). Трибологічні дослідження в умовах сухого тертя та граничного змащування (мінеральні та синтетичні оливи) виявили унікальний механізм самозмащування: фаза гібситу з низькою твердістю зазнає механічного зсуву під час тертя та утворює безперервну, самовідновлювальну плівку твердого мастила, що транспортується між трибоелементами як на сталевих, так і на керамічних контактних поверхнях. Крім того, постсинтетична термічна обробка при 100–400°C з подальшим гідротермальним кип'ятінням викликає дегідратацію цільової матриці, подвоюючи зносостійкість покриття та забезпечуючи 5-10-кратне зниження зносу порівняно з необробленою алюмінієвою підкладкою AA2024. Імпульсна модуляція ефективно зменшує локалізовані теплові концентрації в порах, забезпечуючи оптимальні трибомеханічні характеристики за різних умов досліджень.

Ключові слова: тверде анодування; алюмінієві сплави; беміт; гібсит; термічна обробка; триботрансферна плівка; граничне змащення, тертя.