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Effect of Oxalic Acid Addition on Hardness and Pore-like surface feature Morphology of AA7075 Oxide Layer via Hard Anodizing

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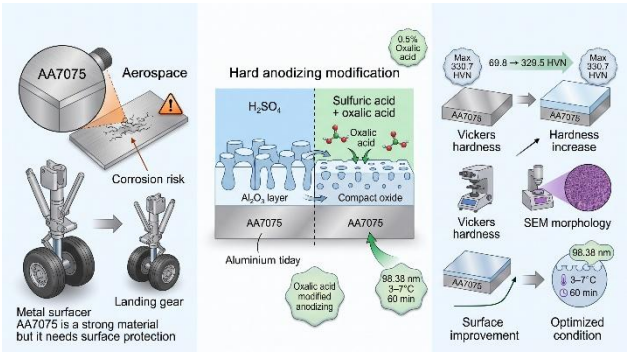
ABSTRACT

AA7075 is a high-strength aluminum alloy widely used in aerospace applications; however, its susceptibility to corrosion and surface degradation may reduce its long-term performance. This preliminary study evaluated the effect of oxalic acid addition to a sulfuric-acid electrolyte during hard anodizing on the measured surface microhardness and surface morphology of an aluminum alloy supplied as AA7075. Hard anodizing was conducted for 60 min at a bath temperature of 3–7°C and a current density of 3 A/dm² using 20% (w/w) sulfuric acid with oxalic-acid additions of 0, 0.5, 1.0, 1.5, and 2.0% (w/w). Surface morphology was characterized using scanning electron microscopy (SEM), while surface microhardness was measured using the Vickers method. The measured surface microhardness increased from 69.8 HVN for the untreated alloy to 296.9–330.7 HVN after anodizing. The highest measured surface microhardness was obtained at 2.0% (w/w) oxalic acid (330.7 HVN). However, the 0.5% (w/w) condition produced a comparable hardness value (329.5 HVN) and the smallest average diameter of selected pore-like surface features (98.38 nm), indicating a favorable combined response within the investigated conditions. The findings demonstrate that oxalic-acid addition modified the measured surface microhardness and surface morphology of the anodized alloy. Further studies involving independent replication, coating thickness, corrosion resistance, wear resistance, and adhesion evaluation are required before aerospace application claims can be established.



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INTRODUCTION

High-strength aluminum alloys are widely used in aerospace and engineering applications because of their excellent mechanical properties, low density, and high strength-to-weight ratio. The 7xxx-series aluminum alloys represent an important group of high-strength materials applied in structural components requiring high load-bearing capability. Aircraft landing gear is one of the most critical components that requires reliable materials because it experiences complex mechanical loads during landing, braking, and ground operations. The landing gear must withstand impact forces, support aircraft weight, and maintain structural integrity during repeated service conditions. Failures associated with landing-gear components may result in serious safety consequences,

making material selection and surface protection important considerations in aerospace engineering [1], [2]. AA7075 aluminum alloy is widely selected for aerospace-related applications because of its high strength and lightweight characteristics. The susceptibility of AA7075 to localized corrosion remains a major limitation because corrosion-induced surface degradation may reduce structural reliability and long-term service performance.

The corrosion behavior of AA7075 is strongly influenced by its alloy composition and microstructural characteristics. AA7075 contains Zn, Mg, and Cu as major alloying elements that contribute to precipitation strengthening and improved mechanical properties. The formation of strengthening precipitates enhances mechanical performance but may also increase corrosion susceptibility because secondary phases can produce electrochemical differences with the aluminum matrix. Grain boundary precipitates and intermetallic compounds may act as preferential regions for corrosion initiation under aggressive environments, particularly those containing chloride ions [3], [4]. Previous studies have demonstrated that corrosion behavior of AA7075 is affected by microstructural conditions, precipitate distribution, and environmental exposure [5], [6]. Corrosion control is therefore an essential requirement for maintaining the reliability of aluminum alloy components, especially for aerospace applications where material degradation may affect operational safety [7].

Surface modification through protective oxide-layer formation has been widely investigated as an approach to improve the corrosion resistance and surface properties of aluminum alloys. Anodizing is an electrochemical surface-treatment process that converts aluminum surfaces into protective aluminum oxide layers. The formation of anodic oxide layers has been reported to improve corrosion resistance and modify surface characteristics of AA7075 alloys [8]. Hard anodizing is a specialized anodizing process designed to generate thicker and harder oxide layers compared with conventional anodizing treatments. The resulting anodic film provides improved surface hardness, wear resistance, and corrosion protection, making it suitable for high-performance aluminum components [9]. The characteristics of anodic films are controlled by several processing parameters, including electrolyte composition, electrolyte temperature, current density, treatment duration, and substrate characteristics [10]. These parameters influence oxide growth, pore formation, film thickness, and mechanical properties of the resulting anodic layer.

Sulfuric acid (H_2SO_4) is one of the most commonly used electrolytes for industrial hard anodizing because of its availability, effectiveness, and ability to produce stable anodic films. Sulfuric-acid hard anodizing is generally performed under low-temperature conditions, commonly below 10°C, to minimize excessive dissolution of the oxide layer during film formation [9]. Previous studies have reported that sulfuric-acid electrolytes can produce anodic layers with improved thickness and hardness compared with conventional anodizing processes. The characteristics of

sulfuric-acid anodic films are influenced by electrolyte concentration, operating conditions, and additional electrolyte components. Studies on sulfuric-acid-based anodizing have demonstrated that electrolyte modification can affect anodic alumina formation, pore characteristics, and film growth efficiency. The addition of specific compounds into sulfuric-acid electrolytes has therefore been explored as an approach to modify anodic-film formation and improve surface performance [11].

Oxalic acid is one of the organic additives that has received attention in aluminum anodizing processes because of its ability to influence anodic-film development. Previous investigations have demonstrated that oxalic acid can modify anodic-film morphology, thickness, and electrochemical formation behavior during aluminum anodizing [12], [13]. The addition of oxalic acid may affect pore-like surface feature formation and influence the structural characteristics of the resulting oxide layer. The measured hardness of anodic films is influenced by multiple factors, including oxide thickness, pore structure, total porosity, chemical composition, and substrate contribution. Surface-feature dimensions alone cannot fully explain the measured hardness response because anodic-film properties are determined by the interaction of several structural factors [12]. Evaluation of both surface morphology and mechanical properties is therefore required to understand the influence of electrolyte modification on anodic-film performance. Previous research on additive modification of AA7075 anodizing has also shown that electrolyte composition and processing conditions influence anodic-film characteristics [14].

Previous studies have provided valuable information regarding the influence of organic additives on aluminum anodizing behavior. Research has mainly focused on anodic-film formation mechanisms, corrosion characteristics, and general morphological changes of anodized aluminum alloys [12]–[14]. Studies involving sulfuric-acid-based anodizing have also demonstrated the importance of electrolyte modification in controlling anodic alumina characteristics [15], [16]. Limited information is available regarding the combined evaluation of measured surface microhardness and SEM-observed surface features of AA7075 treated using mixed sulfuric–oxalic acid electrolytes under controlled hard anodizing conditions. The relationship between oxalic-acid concentration, surface morphology, and measured hardness response remains insufficiently described. This research gap is important because optimization of anodic-film properties requires an understanding of how electrolyte additives influence both structural characteristics and mechanical performance.

This study presents a descriptive evaluation of the effect of oxalic-acid concentration on the measured surface microhardness and SEM-observed surface features of AA7075 treated using sulfuric-acid hard anodizing. The novelty of this research lies in the evaluation of oxalic acid as an additive to a 20% (w/w) sulfuric-acid electrolyte and the comparison of its influence on measured hardness and selected pore-like surface features under controlled anodizing condi-

tions. The relationship between surface morphology and measured microhardness was analyzed to provide information regarding the influence of electrolyte modification on anodic-film characteristics. This study aimed to evaluate the effect of oxalic-acid addition on the measured surface microhardness and surface morphology of anodized AA7075.

METHODS

1. Research Design

This study employed an experimental laboratory approach to investigate the effect of oxalic acid addition on the measured surface microhardness and surface morphology of hard-anodized AA7075 aluminum alloy. The independent variable was the concentration of oxalic acid added to the sulfuric-acid electrolyte, while the measured responses were surface microhardness and selected pore-like surface feature diameter of the anodic oxide layer.

The experimental procedure consisted of specimen preparation, surface pretreatment, hard anodizing treatment, and characterization of the untreated and anodized specimens. The overall research stages are presented in the flowchart shown in Figure 1. The experimental arrangement of the hard anodizing process is illustrated in Figure 2.

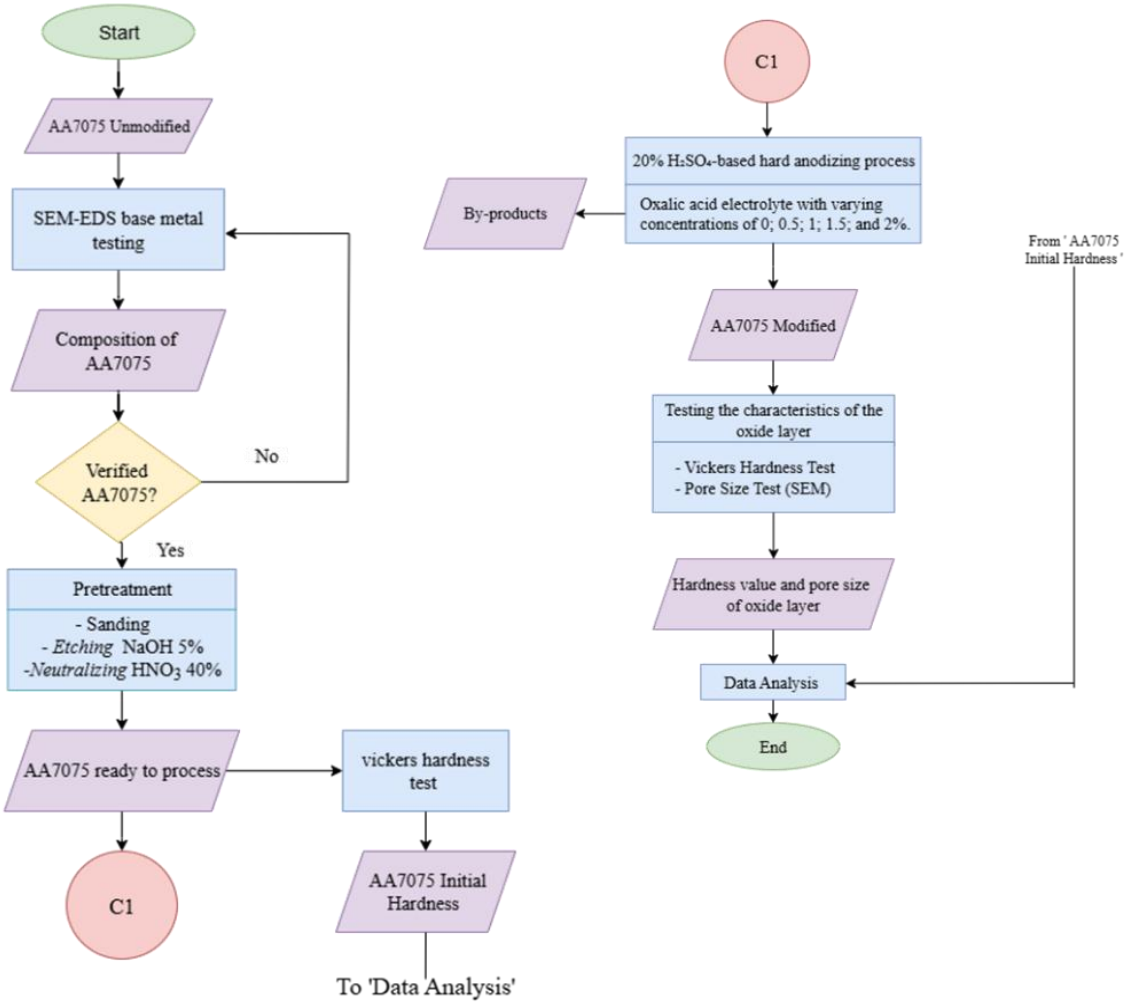


Figure 1. Research Flowchart

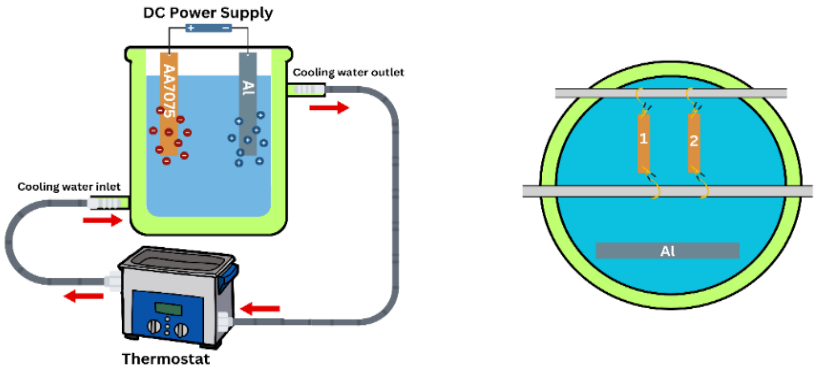


Figure 2. Research Instrumentation Modelling

**Note:*
- *Anode = AA7075 aluminum alloy specimen (orange-colored workpiece), where one specimen was used for SEM observation and pore-like surface feature measurement, while the other was used for Vickers surface microhardness measurement.*
- *Cathode = Pure aluminum*

The AA7075 alloy specimen was used as the anode, while pure aluminum was used as the cathode. Two AA7075 specimens were positioned parallel to each other with a distance of 3 cm during the anodizing process. One specimen was characterized using scanning electron microscopy (SEM) to evaluate surface morphology and selected pore-like surface feature diameter, while the other specimen was used for surface microhardness measurement using the Vickers hardness method.

2. Materials Preparation and Surface Pretreatment

The material used in this study was an aluminum alloy supplied as AA7075. Prior to hard anodizing, the specimens underwent surface pretreatment consisting of mechanical cleaning, alkaline etching, and desmutting.

The specimen surface was initially cleaned using abrasive paper to remove contaminants and surface impurities. Alkaline etching was then performed using 5% (w/w) sodium hydroxide (NaOH) solution at 60°C for 20 s to remove the native oxide layer and modify the initial surface condition. Subsequently, desmutting was performed by immersing the specimens in 40% (w/w) nitric acid solution for 30 s to remove residual products generated during the alkaline etching process [17].

3. Hard Anodizing Treatment

The pretreated AA7075 specimens were subjected to a hard anodizing process using sulfuric acid-based electrolytes with different oxalic acid concentrations. The AA7075 specimen was used as the anode, while pure aluminum was used as the cathode.

The electrolyte consisted of 20% (w/w) sulfuric acid (H_2SO_4) with oxalic acid additions of 0, 0.5, 1.0, 1.5, and 2.0% (w/w). The hard anodizing process was conducted at a bath temperature of 3–7°C, a current density of 3 A/dm², a treatment time of 60 min, and an electrolyte volume of 400 mL.

For each electrolyte composition, one anodizing treatment was performed using two

AA7075 specimens positioned 3 cm apart. One specimen was allocated for SEM observation, while the second specimen was used for Vickers surface microhardness measurement. Therefore, the reported values represent measurements from the investigated specimens and do not represent statistical averages from independently replicated anodizing experiments.

4. Chemical Composition Analysis

SEM–EDS was performed on the untreated material to obtain localized semiquantitative information on its surface elemental composition. The analysis was not used as the sole basis for verifying the bulk alloy grade.

The localized elemental composition of the metal alloy surface was analyzed using scanning electron microscopy equipped with energy-dispersive X-ray spectroscopy (SEM–EDS). The SEM–EDS results were compared descriptively with the nominal composition limits specified for AA7075 in ASTM B209/B209M. However, because SEM–EDS provides localized semiquantitative surface analysis, the results were not considered sufficient to verify the bulk alloy grade. EDS works by analyzing and detecting X-rays emitted by the sample when interacting with an electron beam, followed by the identification and quantification of the chemical elements within it [18]. The analysis was performed using a Hitachi SU3500 scanning electron microscope equipped with an EDAX Octane Pro energy-dispersive X-ray detector.

5. Surface Morphology Analysis of Oxide Layer

The anodized surfaces were observed using SEM, and the diameters of selected pore-like surface features were measured from the resulting micrographs. SEM is important for examining the surface structure of a material in greater detail. SEM micrographs were used to describe the surface morphology of the anodized specimens [19]. The images were acquired at 20,000× magnification and calibrated using the SEM scale bar in [software name]. Only approximately circular or clearly bounded depressions were measured. Irregular cracks, elongated grooves, and poorly defined features were excluded. A total of [number] features were measured from [number] field(s) for each condition. The SEM testing equipment used was a Hitachi SU3500 with EDAX Octane Pro and used a magnification of 20,000x.

6. Surface Microhardness Measurement

Surface microhardness measurements were performed using the Vickers hardness method on both untreated and anodized AA7075 specimens. The untreated alloy was measured to obtain the initial hardness value, which was used as a reference for comparison with the anodized specimens.

The Vickers hardness test was performed using a TMTECK HV-100B Vickers hardness tester (Figure 3) according to ASTM E384. The measurement was conducted using a test load of 0.05 kgf with a specified dwell time. Indentations were performed at different locations on each

specimen to obtain the measured surface microhardness values. Because the thickness of the anodic oxide layer was not measured in this study, the obtained hardness values may include a contribution from the underlying substrate.

Figure 3. Vickers Hardness Tester TMTECK HV-100B

RESULTS AND DISCUSSION

1. Localized Surface Elemental Composition of the Untreated Alloy

The localized surface elemental composition of the untreated alloy is fundamentally based on the alloy composition test results shown in Table 1 , Figure 4 and Figure 5. SEM–EDS detected predominantly Al, with localized Si contents of 0.58 and 0.55% (w/w) at the two analyzed surface areas. These results provide only localized semiquantitative surface-composition information. The principal alloying elements expected for AA7075, particularly Zn, Mg, and Cu, were not reported in the available spectra. Moreover, the measured Si values exceeded the nominal maximum specified for AA7075. Therefore, the SEM–EDS results cannot independently verify the material as AA7075. In the absence of a material certificate or validated bulk-composition analysis, the material is described in this study as an aluminum alloy supplied or nominally specified as AA7075. The measured base-metal hardness of 69.8 HVN was also not used to verify either the alloy grade or temper [21] [8].

The detected elemental characteristics provide additional information regarding the challenges associated with anodizing aluminum alloys in the 7xxx series. The inherent composition of the 7xxx series, particularly the inclusion of Si, Cu, and Zn, makes uniform oxidation notably difficult. Silicon, for instance, remains highly inert and does not dissolve in acidic baths. As the anodic film grows, these undissolved Si particles get trapped inside the Al_2O_3 structure. They essentially act as roadblocks, forcing the pore-like surface features to bend and grow irregularly around them, which creates weak spots or discontinuities in the layer [8], [10].

Table 1. Results of AA7075 alloy composition testing using SEM-EDS at two test points on the sample surface (base metal)

Element	% (w/w)Weight		
	Area Point 1	Area Point 2	Average
Al	99.42	99.45	99.44
Si	0.58	0.55	0.565

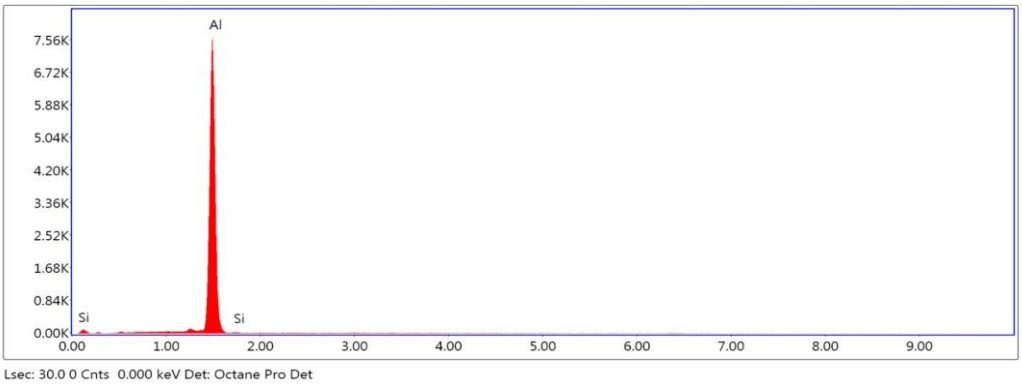


Figure 4. Base Layer Selected Area 1

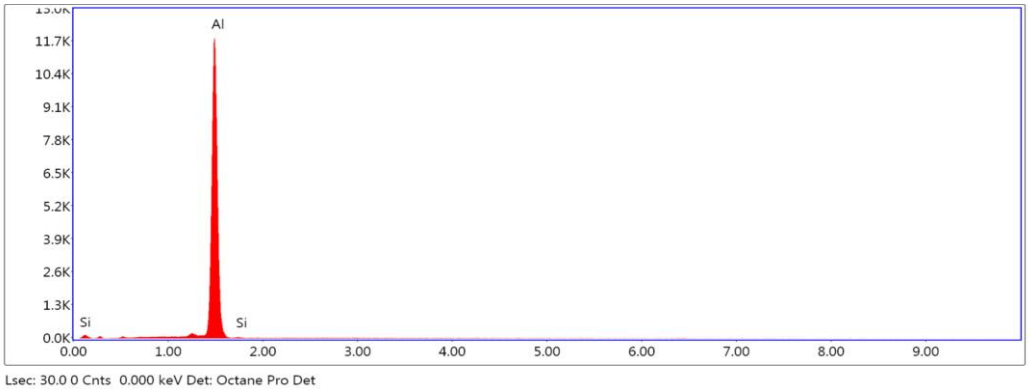


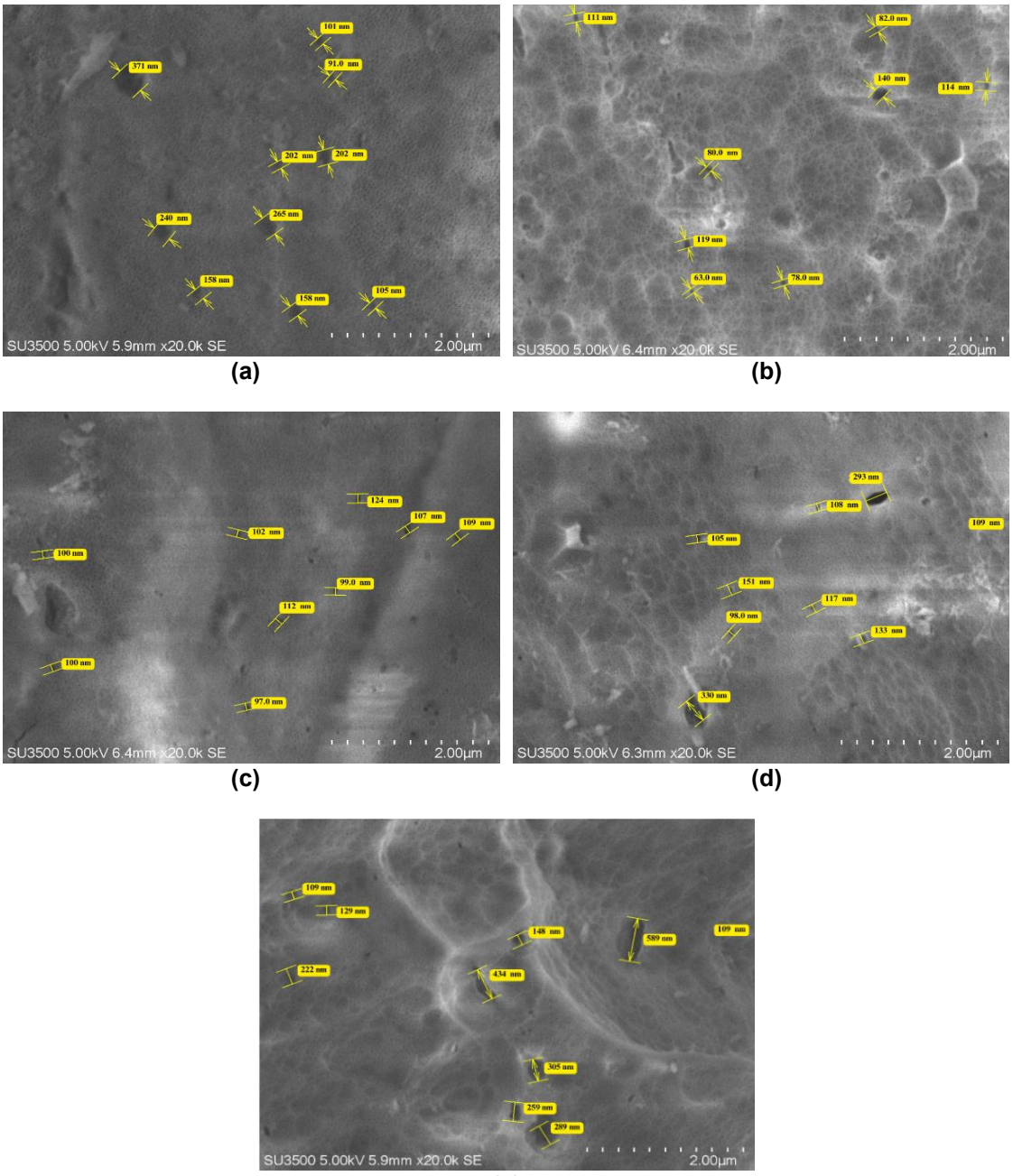
Figure 5. Base Layer Selected Area 2

The behavior of alloying elements such as Cu and Zn during anodizing may also affect the stability of oxide formation. Copper- and zinc-containing precipitates, including $MgZn_2$-related phases, may undergo preferential dissolution compared with the surroundding aluminum matrix. This localized dissolution can promote oxygen evolution at the anode surface and contribute to defects or degradation of the anodic oxide layer [8], [22]. The use of sulfuric acid electrolyte alone may not sufficiently regulate these localized dissolution processes or minimize microstructural effects during oxide growth. The incorporation of organic acid additives, such as oxalic acid, has been reported to modify anodic-film formation behavior by influencing electrolyte–surface interactions and controlling oxide-layer development [6]. The addition of oxalic acid in the present study was therefore evaluated as an approach to improve anodic-film formation on the aluminum alloy supplied as AA7075.

2. Analysis The Effect of Oxalic Acid Addition on Oxide Layer Hardness

The characterization of the anodized specimens consisted of surface microhardness measurement using the Vickers method and surface morphology observation using scanning electron microscopy (SEM). The SEM analysis was used to evaluate the selected pore-like surface feature diameter of the oxide layer rather than to determine total porosity. The obtained SEM micrographs for each anodizing condition are presented in

Figure 6a-e, while the measured surface microhardness values and average selected pore-like surface feature diameters are summarized in Table 2. The relationship between oxalic acid concentration, measured surface microhardness, and selected pore-like surface feature diameter is presented in Figure 7.



(a) **(b)** **(c)** **(d)** **(e)**

Figure 6. Pore-like surface feature Size Values and Microstructure of oxide layer with **(a)** S20 variation, **(b)** S20OK0.5 variation, **(c)** S20OK1 variation, **(d)** S20OK1.5 variation, **(e)** S20 variation

Table 2. Data from testing the hardness and pore-like surface feature size of the AA7075 oxide layer

Variation	Sample Code	Hardness (HVN)	Pore-like surface feature Size Average (nm)
1	S20	296.9	179.30
2	S20OK0.5	329.5	98.38
3	S20OK1	308.0	105.56
4	S20OK01.5	312.9	166.88
5	S20OK2	330.7	276.00

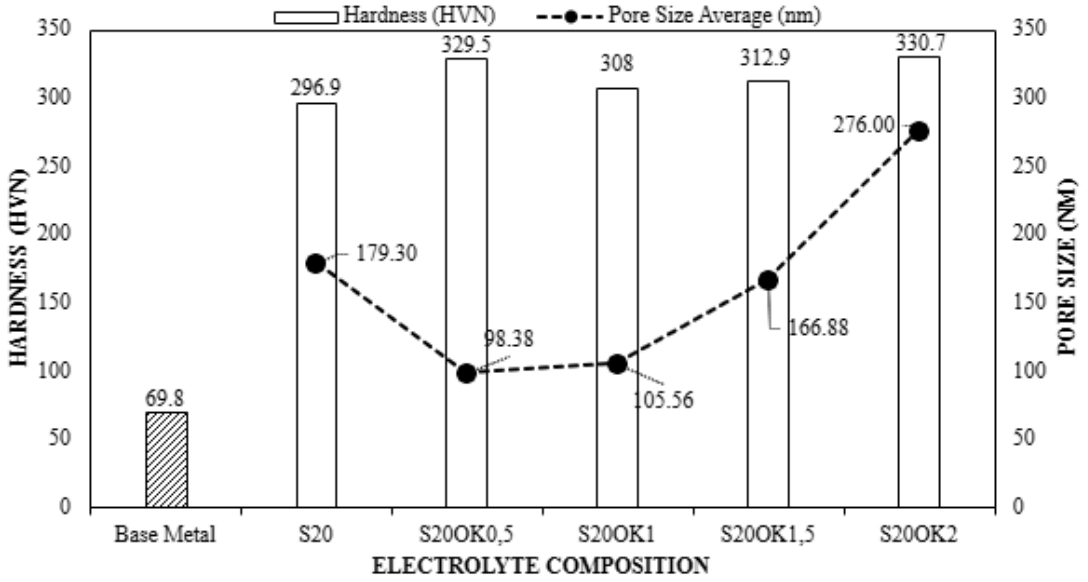


Figure 7. The effect of oxalic acid concentration on the hardness and pore-like surface feature size of the oxide layer

The hard anodizing treatment increased the measured surface micro-hardness of the aluminum alloy supplied as AA7075 under all investigated electrolyte conditions. The untreated alloy exhibited an initial hardness value of 69.8 HVN, whereas the sulfuric-acid-only electrolyte (20% (w/w) H_2SO_4) increased the measured hardness to 296.9 HVN. The addition of oxalic acid further increased the measured surface micro-hardness, producing values ranging from 308.0 to 330.7 HVN depending on the additive concentration. The highest measured value was obtained at 2.0% (w/w) oxalic acid, reaching 330.7 HVN. These results indicate that oxalic acid addition influenced the formation characteristics of the anodic oxide layer under the investigated laboratory conditions. However, the measured surface microhardness values alone cannot establish compliance with aerospace material specifications or confirm suitability for primary landing-gear applications because coating thickness, wear resistance, corrosion resistance, adhesion, fatigue behavior, and long-term durability were not evaluated.

The increase in measured surface microhardness with oxalic acid addition is associated with modifications in oxide-layer

formation during anodizing. Oxalic acid has been reported to influence anodic-film growth, morphology, and electrochemical behavior by modifying the balance between oxide formation and chemical dissolution processes [23]. During hard anodizing, aluminum oxidation occurs simultaneously with localized dissolution of the oxide layer, producing pore-like surface features within the anodic film. The addition of oxalic acid can regulate this competitive process by controlling ion migration and stabilizing oxide formation at the electrolyte–oxide interface [23], [24]. more controlled oxide-growth process may produce a denser oxide structure, which contributes to increased measured surface microhardness.

The proposed mechanism of oxide-layer formation under the investigated electrolyte conditions is illustrated in Figure 8. The sulfuric-acid-only electrolyte promotes relatively aggressive chemical dissolution of the oxide layer, which may result in wider and less uniform pore-like surface features. The addition of 0.5% (w/w) oxalic acid modifies this process by balancing oxide formation and dissolution reactions. The controlled migration of Al^{3+} ions and oxygen-containing species promotes a more compact oxide matrix with smaller selected pore-like surface features. This mechanism explains the high measured surface microhardness obtained from the S20OK0.5 condition.

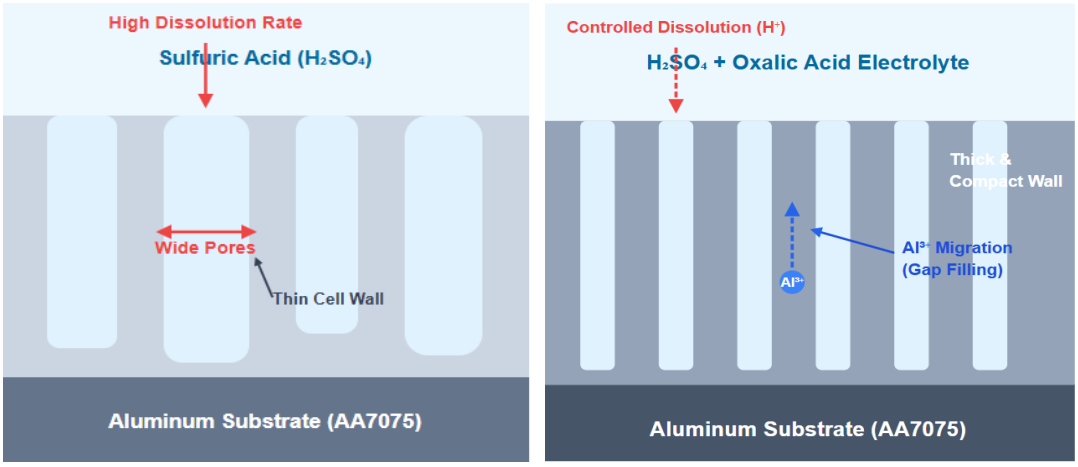


Figure 8. **(a)** Pure Sulfuric Acid Electrolyte. Aggressive chemical dissolution leads to irregular pore-like surface feature widening and less compact structure. **(b)** Sulfuric Acid + 0.5% (w/w) Oxalic Acid. Competitive Process: Balanced dissolution and Al^{3+} migration yields a highly dense and regular oxide matrix.

The 0.5% (w/w) oxalic acid condition produced a measured surface micro-hardness of 329.5 HVN, which was approximately 10.9% higher than the sulfuric-acid-only condition. The value obtained at 0.5% (w/w) oxalic acid was slightly lower than the maximum hardness obtained at 2.0% (w/w) oxalic acid. The difference between these two conditions indicates that increasing oxalic acid concentration does not continuously produce proportional improvement in measured surface microhardness.

The morphology results presented in Table 2 and Figure 7 show that the S20OK0.5

condition produced the smallest average selected pore-like surface feature diameter, namely 98.38 nm. The sulfuric-acid-only condition produced a larger average diameter of 179.30 nm. Previous studies reported that anodic-film morphology and pore structure characteristics may influence the mechanical response of anodized aluminum alloys [14]. Therefore, the 0.5% (w/w) oxalic acid condition can be considered a preferred combined-response condition within the investigated range because it provided a high measured surface microhardness together with the smallest selected pore-like surface feature diameter. This interpretation does not represent a statistically confirmed optimum because independent experimental replication was not performed.

The increase of oxalic acid concentration beyond 0.5% (w/w) did not consistently improve the surface morphology. The 1.0% and 1.5% (w/w) oxalic acid condi-tions produced measured surface microhardness values of 308.0 and 312.9 HVN, respectively, which were lower than the 0.5% condition. The reduction in measured hardness cannot be conclusively attributed to changes in electrolyte viscosity because viscosity measurements were not performed. Although electrolyte transport properties may influence ion mobility during anodizing, the contribution of viscosity-related effects remains unverified under the present experi-mental conditions [24].

The 2.0% (w/w) oxalic acid condition produced the highest measured surface microhardness value of 330.7 HVN but also resulted in the largest average selected pore-like surface feature diameter of 276.00 nm. This condition demonstrates that surface-feature diameter alone cannot be used as the only parameter to predict oxide-layer hardness. The mechanical properties of anodic films may also depend on oxide-layer thickness, composition, porosity distribution, internal structure, and substrate contribution [29], [30]. The larger selected pore-like surface features observed at higher oxalic acid concentration may be associated with changes in oxide-growth and dissolution balance during anodizing [27], [28].

The SEM observations obtained in this study cannot determine whether the larger surface features observed at 2.0% (w/w) oxalic acid resulted from oxygen evolution, localized dissolution, cracking, or other oxide-formation phenomena. Previous studies have reported that second-phase particles in aluminum alloys may influence oxide-layer formation and promote localized defects during anodizing [22]. The coexistence of the highest measured surface microhardness and the largest selected pore-like surface feature diameter at 2.0% (w/w) oxalic acid confirms that the relationship between morphology and hardness is not governed by a single parameter. The 2.0% (w/w) condition can therefore only be described as producing the highest measured surface microhardness within the investigated range, while further evaluation of coating thickness, wear resistance, corrosion resistance, and adhesion is required to determine its practical performance.

CONCLUSION

The hard anodizing treatment increased the measured surface micro-hardness of the aluminum alloy supplied as AA7075 from 69.8 HVN (untreated alloy) to values ranging from 296.9 to 330.7 HVN under the investigated conditions. The addition of 0.5% (w/w) oxalic acid (S20OK0.5) produced a high measured surface microhardness of 329.5 HVN with the smallest average selected pore-like surface feature diameter of 98.38 nm, indicating a favorable combined response between hardness improvement and surface morphology modification. The highest measured surface microhardness was obtained at 2.0% (w/w) oxalic acid (330.7 HVN); however, this condition also produced the largest selected pore-like surface feature diameter. These findings demonstrate that oxalic-acid addition modifies the anodic oxide-layer characteristics under the investigated laboratory conditions. The present results should be considered preliminary because independent anodizing replication, coating-thickness measurement, wear resistance, corrosion resistance, adhesion, fatigue performance, and long-term durability were not evaluated. Further investigations are required to determine the suitability of the anodized alloy for aerospace-related applications.

Author Contributions: R. P. Sihombing contributed to the conceptualization, methodology development, experimental investigation, data analysis, visualization, and manuscript preparation. M. Hulupi contributed to the methodology, validation, experimental support, and manuscript review. N. Puspita contributed to the investigation, data validation, and manuscript review. J. P. Alamsari contributed to supervision, data analysis, manuscript review, and editing. All authors have read and approved the final version of the manuscript.

Conflict of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this manuscript.

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