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A Case Study on Ionic Compounds on the Aluminium 6061 Surface during Etching and Passivating Processes

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ABSTRACT

The surface treatment process for aluminium is extensive, encompassing many methods and techniques. This study utilises chemical etching and passivation chemicals as surface treatment processes for chemical cleaning. The corrosion-controlled procedure involves the application of a powerful etchant solution to eliminate undesirable layers of material. Additionally, passivation chemicals are utilised as coatings to enhance the corrosion resistance of aluminium surfaces. Following the chemical cleaning process, ion chromatography testing was conducted to verify the ionic compounds on the surface of the aluminium metal subsequent to its reaction with etching and passivation chemicals. Different approaches were employed to address the ionic compound deposition problem on the surface of aluminium 6061 metal. Results revealed that replacing existing chemicals with new ones is the best solution to reduce the reading below the specified limit. However, one economically viable alternative for the chemical cleaning process is implementing an aeration process during rinsing, which offers greater economic efficiency compared to the substitution of chemicals entirely. Besides that, chemical cleaning via aeration procedure can mitigate the accumulation of ionic compounds on the material's surface.

1. Introduction

Aluminium alloys are metals with the highest aluminium content, often used in engineering structures and components due to their low weight and resistance to corrosion [1-3]. They are classified by the American National Standard Institute (ANSI) numbering system or names like DIN and ISO. The choice of an alloy depends on factors like tensile strength, density, ductility, formability, workability, weldability, and corrosion resistance [1,4]. It is also lightweight, with a strength-to-weight ratio surpassing steel. It is widely used in various applications, including foils and conductor cables. Recent technological advancements, such as aluminium cutting/profiling techniques and pre-machining plate and bar materials, have made aluminium more efficient and cost-effective [2,5].

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Aluminium's properties include softness, ductility, corrosion resistance, high strength [6] and high electrical conductivity. In addition, alloys used in aluminium alloys have a superior strength-to-density ratio compared to conventional steel alloys, allowing for weight reduction without compromising structural stiffness or crash safety [7-9]. These alloys have higher strength than pure aluminium due to the presence of various alloyed materials [4,10,11]. It is also a popular material for power transmission lines and household wiring due to its exceptional thermal and electrical conductivity [12].

Chemical etching process can be used to improve the corrosion resistance of aluminium. Aluminium immersed in caustic soda solutions during etching will significantly affect surface modification [13]. The process is governed by mixed electrochemical reactions, with a strong emphasis on the cathodic reaction. Iron as an essential constituent provides cathodic sites and plays a pivotal role in aluminium dissolution [14]. The concentration of free caustic soda fluctuated around a mean value, influenced by temperature, usage rate, and rest periods. The primary objective of chemical etching was to reduce surface gloss and achieve a uniformly matte or satin finish [15]. Moreover, alloys exhibited varying etching rates but could be broadly categorised into two groups based on their iron content [16,17]. Adding a small amount of cobalt to a 2% caustic soda solution significantly impacted the etching rate, particularly for 99.5% purity aluminium and 99.99% purity aluminium [18]. Çakır [19] studied etchants for the chemical machining of aluminium and its alloys, where ferric chloride ($FeCl_3$) as an etchant was implemented. $FeCl_3$ is cost-effective, easy to control, and has numerous industrial applications. According to Çakır *et al.,* [20], high etching temperatures are selected for higher etching depth and better surface finish quality, making it a viable choice for various etching applications. Further research regarding etchant concentrations and some chemical additives, as well as examining other etchants, is needed to ensure this etching process is environmentally friendly and supports the economic regeneration of waste etchant and recovery of etch material [21].

Passivation is the process after etching to create a protective layer on the aluminium surface [22,23]. This reduces susceptibility to environmental factors like corrosion or degradation. Even though metals like aluminium, chromium, zinc, titanium, and silicon develop a passivation layer upon exposure to air, which significantly retards oxidation and corrosion, forming a more protective passive film on its surface can exhibit superior performance [24]. One of the passivation solutions usually used for aluminium and its alloys was nitric acid, and nitric acid solutions contained chloride.

The anodic oxide layer on aluminium exhibits near-perfect dielectric properties, while the film formed on aluminium alloy deviates from the ideal behaviour of a capacitor. The progressive thickening of the oxide layer over time is influenced by factors such as oxide volume ratio, oxygen diffusion, and the chemical potential of the oxide. Certain corrosion inhibitors facilitate the development of passivation layers on metal surfaces, while others create films with poor solubility and non-reactivity [25].

Aeration or aerification refers to air circulation, mixing, or dissolution in a liquid or other fluid-like material, such as dirt. Aeration techniques enhance the mixture's surface area, facilitating more extensive chemical or suspension reactions [26]. Aeration procedures are extensively employed in the wastewater treatment sector to regulate the levels of Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD) during water treatment.

Besides, cleaning, etching, and passivation of aluminium alloys can cause several issues. Chemical etching involves removing metal from a sheet using corrosive chemicals, while passivation is a corrosion-prevention technique that creates an outer layer of shield material. Chemically treating aluminium surfaces involves etching to remove unwanted elements and passivation to protect them from corrosion. However, these processes can cause problems on the surface of the material.

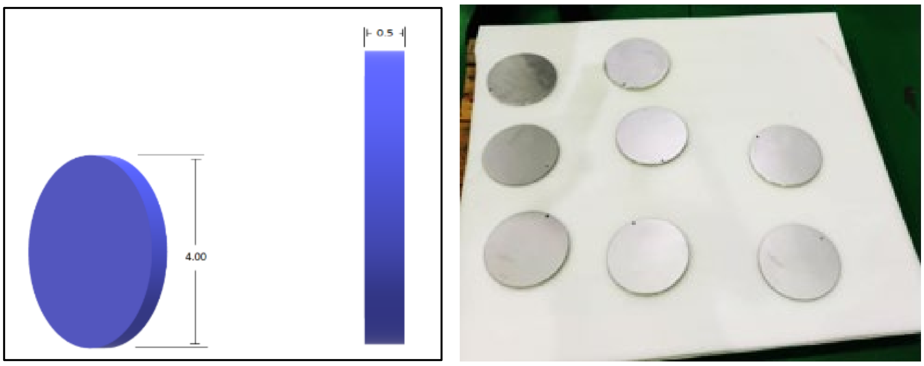
Therefore, this study focuses on studying the ionic compounds on the surface of the Aluminium 6061 after exposure to etching and passivation processes. Analysing factors affecting the accumulation of unwanted ionic compounds and methods to mitigate and decrease the concentration of undesirable ions to adhere to United States Environmental Protection Agency (US EPA) standards will also be investigated.

2. Methodology

In this study, different cleaning parameters were used in the chemical cleaning process for Aluminium 6061, such as various uses of rinsing water, adding an aeration process, and complete changes in chemical etching and passivation. These parameters were set to monitor the results of the ionic compound formation on the surface of the aluminium material after chemical cleaning processes.

2.1 Sample Preparation

The dimensions of coupon testing material used in this research are Aluminium 6061 with a dimension Ø 4 x 0.5-inch, as illustrated in Figure 1(a). This coupon was machined using the CNC machine model Mazak Vortex I-630V, as can be seen in Figure 1(b). Then, coupons need to be buffed and polished as a surface enhancement process to remove metal from the surface, creating a coarse line pattern [27,28].



(a) (b)

Fig. 1. (a) Desired dimensions of coupon testing material (b) Al6061 coupons

2.2 Cleaning Process

After polishing, the Aluminium 6061 coupon material will undergo chemical cleaning as represented in Figure 2. The cleaning process started with a degreasing process with a degreasing chemical that consisted of 10% isoprep and 90% water, and the rinsing process was continued. Next, the coupon part was submerged into the tank with etching chemicals of 20% nitric acid (HNO_3), 0.5% hydrofluoric acid (HF), and 79.5% water) for 45 seconds, followed by a rinsing process with water, and then the chemical is submerged again in a passivation chemical (20% nitric acid (HNO_3) and 80% water). It was used to remove iron from the surface of corrosion-resistant steel parts, thus enhancing corrosion resistance. The passivation process is prepared for 1 minute and followed by another rinse. The sample was immersed in a hot-rinse and ultrasonic tank, and cavitation bubbles induced by high-frequency pressure (sound) waves were utilised to agitate a liquid. The ultrasound frequency is set between 30 to 70 kHz. The accumulative effect of the millions of imploding bubbles is what causes the cleaning effect of ultrasonic cleaning. The coupons were then dried with nitrogen gas to ensure the part was fully dry and there was no residue of chemicals and liquids left that may affect the appearance of the part.

Fig. 2. Chemical cleaning tank

2.3 Material Characterisation

Using the ion chromatography system (ICS) machine, the coupon dried using nitrogen gas was characterised using an ion chromatography (or ion-exchange chromatography) process. This process is implemented to detect the ions attached to the coupon. Controlling the ions to follow the United States Environmental Protection Agency (US EPA) is essential. The issue is that specific cleaning process chemicals exceed the specification's ion value.

3. Results

3.1 Ion Chromatography Analysis

Table 1 presents the findings of the ion chromatography examination conducted on component material (coupon) subjected to chemical etching and passivation procedures. The US EPA standard was utilised to verify 13 ionic elements. Two initial coupons underwent standard chemical cleaning procedures and were determined to have elevated levels of ammonium ions beyond the limit established by industry regulations. Additional testing was conducted utilising identical etching and passivation chemicals, except the rinsing tank, which was meticulously cleansed before commencing the chemical cleaning procedure and replacing the rinsing water. The results of the ion chromatography test, as depicted in Table 2, continue to demonstrate the presence of elevated levels of ammonium ions. However, no alterations were made to any other settings for the procedures. According to Table 1 and Table 2 results, high concentrations of ammonium ions were detected, exceeding the US EPA standard limits of 100 (10^{12} molecules/cm^2). Results for coupon 2 show a slight decrease in the ammonium concentration compared to coupon 1 after an additional cleaning process was added. This might be due to the buildup of ammonium ions accumulated during the reaction of aluminium with etching and passivation chemicals that had not been appropriately cleared during the rinsing process. In addition, hydrogen molecules from water react with nitrogen ions contained in etching and passivate chemicals to produce ammonia which further reacts with the acidic properties of the chemicals to produce additional ammonium ions. This statement agrees with

Christian [29], who believes improper rinsing can leave behind residues that might affect corrosion resistance.

Table 1
Ion chromatography analysis without cleaning process for coupon 1

Element	US EPA	Coupon		Results comparison with US EPA
		Sample 1A	Sample 1B	
	Process	20 min rinse tank		
LI+	10	<9.55	<9.55	Pass
Na+	200	23.15	37.47	Pass
NH4+	100	284.51	386.43	Fail
K+	15	20.42	30.51	Pass
Mg+2	440	49.26	54.52	Pass
Ca+2	200	29.88	66.14	Pass
F-	300	<5.25	<5.23	Pass
Cl-	240	18.92	101.82	Pass
NO2-	53	11.43	<5.04	Pass
Br-	10	<1.92	<1.91	Pass
NO3-	150	40.74	113.43	Pass
PO4-3	10	<1.12	<1.12	Pass
SO4-2	100	<1.04	<1.03	Pass

Table 2
Ion chromatography analysis additional cleaning process for coupon 2

Element	US EPA	Coupon	Results comparison with US EPA
		Sample 2	
	Process	Clean tank and 20 min rinse tank	
LI+	10	<9.24	Pass
Na+	200	<13.66	Pass
NH4+	100	309.75	Fail
K+	15	<1.80	Pass
Mg+2	440	55.38	Pass
Ca+2	200	<4.64	Pass
F-	300	84.36	Pass
Cl-	240	7.23	Pass
NO2-	53	18.12	Pass
Br-	10	<1.85	Pass
NO3-	150	42.39	Pass
PO4-3	10	<1.08	Pass
SO4-2	100	26.03	Pass

3.2 Experimenting with Changes in Parameters of Rinsing Water

The rinsing process was done with deionised water (DI Water), which was changed to direct tap water and filtered using the mixed bed as represented in Table 3. Coupon 3 was tested with an unchanged rinsing time. The results of ammonium ion content still but showed drops in the number of ammonium ions on the surface material of the coupon at 270.58 (10^{12} molecules/cm^2) compared to the previously tested coupon at 309.75 (10^{12} molecules/cm^2). This slight drop in ammonium ions concentration may be attributed to using a mixed bed as a filter for the rinsing water used during the chemical cleaning process.

Table 3
Ion chromatography results after changing rinsing water

Element	US EPA	Coupon	Results comparison with US EPA
		Sample 3	
	Process	In cleanroom, direct SAJ in beaker	
LI+	10	<9.24	Pass
Na+	200	<13.66	Pass
NH4+	100	270.58	Fail
K+	15	<1.80	Pass
Mg+2	440	65.93	Pass
Ca+2	200	8.00	Pass
F-	300	50.61	Pass
Cl-	240	<1.99	Pass
NO2-	53	<4.88	Pass
Br-	10	<1.85	Pass
NO3-	150	39.29	Pass
PO4-3	10	<1.08	Pass
SO4-2	100	26.03	Pass

The duration of rinsing was subsequently extended from the conventional 20-minute interval to 60 minutes (equivalent to 1 hour) in order to evaluate the impact of rinsing time on the ionic concentration present on the material's surface throughout the process of chemical cleaning. The results of the ion chromatography testing indicate a significant drop in the ammonium ion content on the surface of two materials in Coupon 4 as presented in Table 4. However, coupon sample 4 still exhibited greater ammonium ion levels than the permissible industrial criteria. Furthermore, higher rinsing time may be attributed to the sizable drop in ammonium ions concentration on the aluminium coupon material. The influence of the ion concentration plays a role when liquid-solid interfaces charge up in the presence of flow [30]. A longer rinse time might cause a longer reaction of water molecules with ammonium ions, causing the ion to disintegrate into another compound and decreasing the concentration of the ammonium ion detected during the ion chromatography test.

Table 4
Increasing rinsing time during chemical cleaning

Element	US EPA	Coupon	Results comparison with US EPA
		Sample 4	
	Process	In cleanroom, 1 hour rinse in beaker	
LI+	10	<9.24	Pass
Na+	200	<13.66	Pass
NH4+	100	103.23	Fail
K+	15	<1.80	Pass
Mg+2	440	52.73	Pass
Ca+2	200	<4.64	Pass
F-	300	<5.06	Pass
Cl-	240	<1.99	Pass
NO2-	53	16.38	Pass
Br-	10	<1.85	Pass
NO3-	150	17.74	Pass
PO4-3	10	<1.08	Pass
SO4-2	100	<1.00	Pass

3.3 Water Aeration Process

The chemical cleaning process is applied to Coupon 5, accompanied by an aeration process, including injecting nitrogen gas into the cleaning tank as shown in Table 5. Aeration, also known as aerification or aeration, is the procedure of circulating, mixing, or dissolving air in a liquid or other fluid substance. The result of the ion chromatography test for coupon 5 reveals that the concentration of ammonium ions is within the limit of US EPA standards. These drops may be because of the aerobic digestion process [31] as the aeration process induces the motion of water in contact with the surface of the sample material, hence increasing the contact of the surface of the sample material with the rinsing water causing more reaction of ammonium ion and the water molecules to reduces the total concentration of the ammonium ions.

Table 5
Ion chromatography analysis after the addition of aeration process

Element	US EPA	Coupon	Results comparison with US EPA
		Sample 5	
	Process	1 hour rinse in tank (purge N2 in chemical)	
LI+	10	<9.24	Pass
Na+	200	<13.66	Pass
NH4+	100	99.67	Pass
K+	15	<1.80	Pass
Mg+2	440	55.37	Pass
Ca+2	200	<4.64	Pass
F-	300	<5.06	Pass
Cl-	240	<1.99	Pass
NO2-	53	23.39	Pass
Br-	10	<1.85	Pass
NO3-	150	15.71	Pass
PO4-3	10	<1.08	Pass
SO4-2	100	<1.00	Pass

3.4 Testing Fresh Etching and Passivation Chemicals

Coupons 6 and 7 underwent a cleaning procedure involving utilising a comprehensive array of new chemicals, water rinsing, and incorporating aeration, as seen in Table 6. The findings of the ion chromatography tests indicate that coupon 6A exhibits the lowest recorded ammonium ion concentrations at 21.36 (10^{12} molecules/cm^{2}), which is significantly below the specified limit. Coupons 7 and 6B follow suit, with all ionic compounds falling under the specified range set by the industries. Coupon 6A employed new chemicals in conjunction with a rinsing duration of 1 hour, whereas sample 7 underwent cleaning utilising new chemicals but with a rinse duration of 20 minutes. Finally, coupon 6B is subjected to chemical cleaning utilising a novel chemical compound, accompanied by an additional aeration process.

All the coupons presented results that complied with the US EPA standards for all ionic compounds exhibited on the surface of the aluminium 6061 coupon. This can be said due to the pure concentration of ions in new chemicals causing less side reactions from existing ions to react to the aluminium surface that caused the formation of ammonium ions. Impurities from chemicals that have been used alter the pathways that cause the occurrence of side reactions. The essential part that connects all the results is that new chemicals were used in the chemical cleaning process. All the data on ammonium ion concentrations were gathered and graphed together, as shown in Figure 3.

Table 6
Ion chromatography analysis after using fresh chemicals

Element	US EPA	Coupon		Results comparison with US EPA	Coupon	Results comparison with US EPA
		Sample 6A	Sample 6B		Sample 7	
	Process	New chemical, 1 hour rinse in tank	New chemical, 1 hour rinse in tank, chemical purge with N2		New chemical, 20 min rinse in tank	
LI+	10	<9.23	<9.23	Pass	<9.23	Pass
Na+	200	<13.66	<13.66	Pass	<13.66	Pass
NH4+	100	21.36	60.51	Pass	56.95	Pass
K+	15	<1.80	<1.80	Pass	<1.80	Pass
Mg+2	440	390.23	34.28	Pass	39.55	Pass
Ca+2	200	<4.64	<4.64	Pass	<4.64	Pass
F-	300	<5.06	<5.06	Pass	<5.06	Pass
Cl-	240	<1.99	<1.99	Pass	<1.99	Pass
NO2-	53	19.88	30.28	Pass	29.66	Pass
Br-	10	<1.85	<1.85	Pass	<1.85	Pass
NO3-	150	<2.58	69.19	Pass	35.66	Pass
PO4-3	10	<1.08	<1.08	Pass	<1.08	Pass
SO4-2	100	<1.00	<1.00	Pass	<1.00	Pass

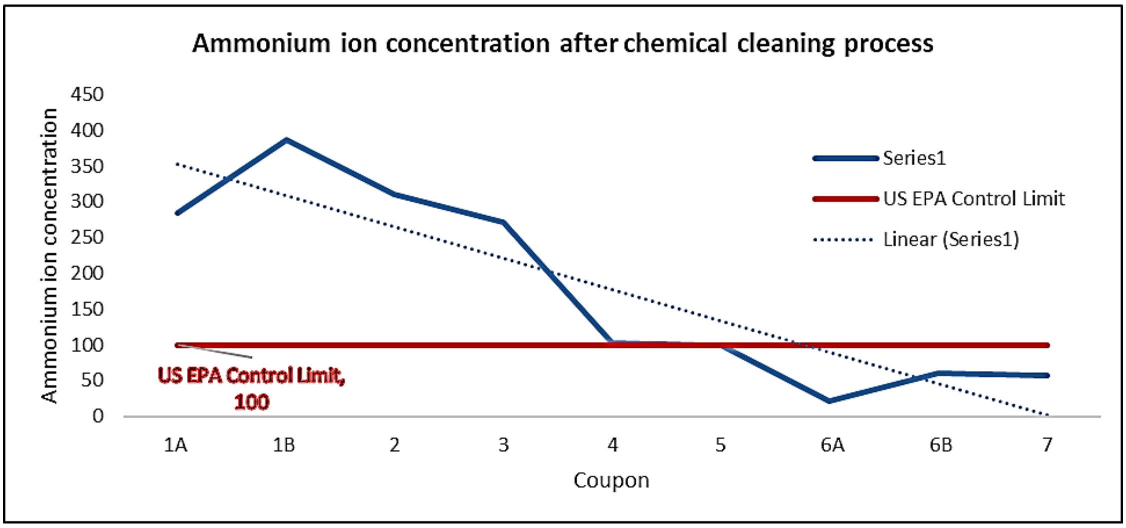


Fig. 3. Comparison of ammonium ion content for all coupon tested with US EPA standard limit

4. Conclusions

The study of ionic compounds on the surface of the Aluminium 606 was implemented and complied with established US EPA standards and specifications, notably for ion concentration. Additionally, the study attempted to address the problem of ionic compound accumulation on surface materials. From observation, replacing existing chemicals with a new chemical reduces the likelihood of undesired element accumulation due to fewer side reactions. However, replacing chemicals for each chemical cleaning step is time-consuming and increases the cost. Therefore, alternative approaches are also being investigated to enhance the efficacy of the chemical cleaning procedure while minimising the financial resources allocated to it. Furthermore, using an aeration procedure in conjunction with chemical cleaning mitigates the accumulation of ionic compounds on the material's surface. Thus, it is said that a reduction in the ionic compound buildup on aluminium 6061 is achieved.

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