



# Deposition of Hard and Hydrophobic Al<sub>2</sub>O<sub>3</sub> Coatings on Al alloys By MAO process

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## Abstract

Surface engineering of Al alloys by deposition of hydrophobic coatings using different techniques to improve the wear and corrosion resistance and self-cleaning properties has gained more attention in the last years. This study is an attempt to use thassos additives in modification of the electrolytes used in the micro-arc oxidation MAO process for the deposition of hard and super hydrophobic Al<sub>2</sub>O<sub>3</sub> ceramic coatings on Al alloys for heavy load-bearing tribological and self-cleaning applications. Thassos rocks in different concentrations (1, 2, 3, 5 and 7 g/L) were used as modification components. Ceramic coatings of Al<sub>2</sub>O<sub>3</sub> were deposited on Al alloy by using a homemade MAO unit with an applied voltage of (320-350) V, current of (0.1-0.3) A and a temperature of (15-25°C), and at different deposition times (10-45min). The coated surfaces were post-treated using fatty acids such as myristic acid (10g/L) and stearic acid (5g/L) for important self-cleaning properties. Various analytical techniques and tests were used to characterize the coatings such as scanning electron microscopy (SEM), X-ray diffraction (XRD), hardness, thickness, and angle of contact measurements and tests. The highest contact angle of 95.120° was achieved when using thassos (3g). This high contact angle indicates that the surfaces are highly hydrophobic, which means that they repel water effectively. The best result for the highest hardness value recorded was 975.9 HV when using thassos (7g) as additives. Results from this research will promote future works in surface engineering of Al alloys by MAO process using more environmentally friendly, readily available and require minimal process additions such as ceramic natural additions of thassos in the deposition of hard and hydrophobic ceramic coatings for wear resistance and self-cleaning applications.

**Keywords:** hydrophobic coating, MAO, natural rocks, Thassos marble.

## 1. Introduction

Hydrophobic coatings have gained significant attention in recent years due to their potential to improve corrosion resistance and provide self-cleaning properties [1-2]. These coatings create a water-repellent surface, preventing the adhesion and spread of water droplets, making them valuable for applications where water contact or moisture can be problematic. Hydrophobic Al alloy surfaces have been generated using different techniques, including anodic oxidation, sol-gel, physical method, etching, electrodeposition, and chemical modification by immersion [3-5]. However, these techniques typically require expensive facilities, extensive materials, and/or present environmental issues [6].

MAO oxidation method is a complicated process of plasma discharge and anodizing oxidation, and the initially of the MAO method is an anodization method [7]. MAO has proven effective in creating a nano scale coral-like structure, resulting in super hydrophobic surfaces.



These surfaces not only demonstrate impressive corrosion resistance and self-cleaning capabilities but also exhibit excellent chemical stability. The surface engineering of Al alloys by means of hardness and self-cleaning aspects using MAO process and modified electrolyte with ceramic natural rock additives [8-9].

Many most researchers have made significant efforts to develop and utilize various artificial additives to modify electrolytes in the Micro arc Oxidation (MAO) process, with the goal of enhancing the quality and properties of the oxide coatings. These additives can include a wide range of substances, such as Carbonates, borates, phosphates, and silicate [10], graphene oxide into the base silicate hypophosphite electrolyte [11], nano-SiO<sub>2</sub> [12], the Al<sub>2</sub>O<sub>3</sub> additive [10] and complexing agents [13-15], each serving in the improvement of corrosion resistance, wear resistance, or biocompatibility of the resulting coatings.

Thassos Marble is renowned for its luxurious appearance and is considered one of the most expensive natural stones on the market due to its scarcity and premium quality [16]. The highest-grade is characterized by pure, clear white coloration. It often appears in the form of pure blocks resembling transparent sugar cubes. Thassos Marble is known for its high degree of purity, with a nearly 100% absence of impurities. This characteristic sets it apart from many other types of marble. Thassos Marble is predominantly composed of dolomite, a mineral that contributes to its unique properties [17]. Dolomite-based marbles are known for their whiteness and special thermal characteristics. In summary, Thassos Marble is a prestigious and sought-after natural stone with a history of use in iconic architectural and artistic creations. Its rarity, purity, and exquisite appearance make it a symbol of luxury and high class in the world of natural stone materials [18].

The aim of this work is using MAO technique [19] with modified electrolytes containing natural ceramic additives of thassos rock in depositing of nano-composite ceramic Al<sub>2</sub>O<sub>3</sub> coatings with notable properties of hardness and hydrophobicity together. It is considered a qualitative leap existing body of scientific knowledge by presented for the first time of the process of depositing a hard and hydrophobic alumina layer using thassos rocks.

## 2. Experimental Works

### 2.1 Substrates Preparation

The Al 1105 alloy was selected as the substrate material as shown in table (1). The shaft prepared from this alloy has specific hardness values of 146.7 HV. The substrates were machined to achieve the desired shape and dimension ( $\varnothing 15 \times 5$  mm<sup>2</sup>). After machining, the surfaces of the substrates were polished using silicon carbide (SiC) abrasive paper with various grit sizes (ranging from 280 to 2000), the substrates are polished to obtain a specific roughness value (Ra) of about 0.282  $\mu$ m. After polishing, the substrates were degreased using acetone to remove any contaminants or residual oils from the surface. This step was necessary to ensure a clean and uncontaminated surface for subsequent processing; the substrates were cleaned with distilled water for 10 seconds. The preparation steps described were crucial to ensure the success of the MAO process and the quality of the final product.

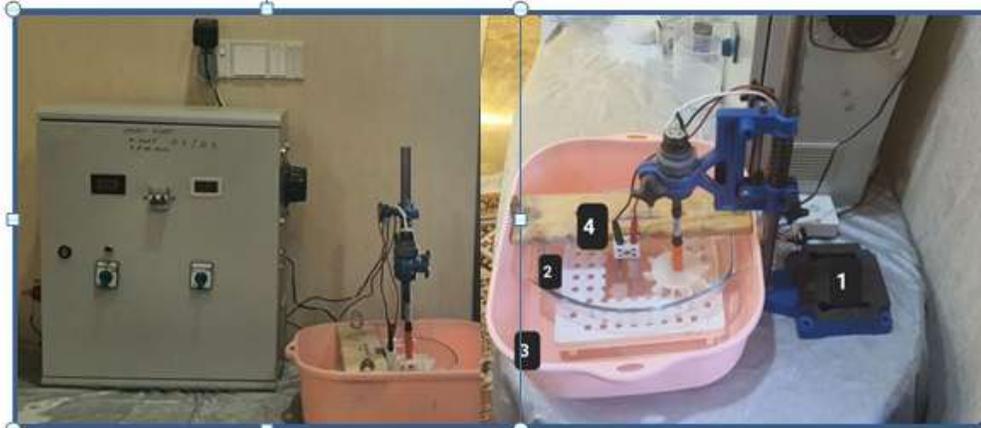
**Table 1: Chemical Composition of 1105-GOST4784 Al alloy**

Element	Content (Wt. %)	Element	Content (Wt. %)
Si	0.266	Ni	0.0021
Fe	0.386	Zn	0.0423
Cu	3.530	Ti	0.0129
Mn	0.890	Pb	0.0072
Mg	0.463	V	0.0054
Cr	0.0634	Al	94.3

## 2.2 MAO Process

MAO a homemade DC-AC unit with a voltage of 500V to deposit ceramic coatings on an aluminum substrate as shown in figure (1). Process performed at a fixed current density of (1-3) A/dm<sup>2</sup> and a voltage of 350V. The electrolyte solution held in a 2-liter glass container. Table (2) contains information about the chemical composition of the electrolyte solution, which includes first additives thassos. This solution is crucial for deposition of Al<sub>2</sub>O<sub>3</sub> coatings on the aluminum substrate. Al substrate acts as the anode during the MAO process, while stainless steel 316L used as the cathode electrode. The anode and cathode play crucial roles in the electrochemical reaction that occurs during the process, sparks occurred that a common observation in the MAO process and indicates the formation of micro-arcs or localized breakdown of the electrolyte due to high voltages and current densities being applied. These sparks contribute to the generation of plasma and localized heating required for the formation of the ceramic coatings on the sample surface. A cooling unit utilizes a combination of ice, salt, and alum to control the temperature of the electrolyte solution of 5°C. After the MAO process is completed, the coated samples washed with distilled water to remove any residue or excess electrolyte solution. Subsequently, the samples dried using a hot air dryer.

**Figure 1: Images of the Coating Unit, (1) Mixer, (2) Coating Container, (3) Cooling Container, (4) Electrodes, (5) Thermometer.**



- Mixing of basic electrolyte based on thassos additions as shown in table (2).



Table (2): Thassos Addition Basic Electrolyte in MAO Process.

<i>Substrate</i>	<i>MC1</i>	<i>MC2</i>	<i>Cth</i>	<i>S</i>	<i>O</i>	<i>THS<sub>1</sub></i>	<i>THS<sub>2</sub></i>
<i>Materials</i>							
<b>KH<sub>2</sub>PO<sub>4</sub></b>	20gm	20gm	20gm	20gm	20gm	20gm	20gm
<b>KOH</b>	2 gm	2gm	2gm	2 gm	2gm	2gm	2 gm
<b>Na<sub>2</sub>SiO<sub>3</sub></b>	—	—	—	10gm	10gm	—	—
<b>(NH<sub>3</sub>)<sub>2</sub>MO<sub>7</sub>O<sub>24</sub></b>	12gm	12gm	12gm	12gm	12gm	12gm	12gm
<b>H<sub>3</sub>PO<sub>4</sub></b>	8 ml	8ml	8ml	8 ml	8ml	8ml	8 ml
<b>Thassos</b>	1gm	2gm	3gm	5gm	7gm	3gm	3gm
<b>MOS<sub>2</sub></b>	—	—	—	—	—	1gm	1gm
<b>PH</b>	4-5						
<b>Temp.( °C )</b>	15-25	15-25	15-25	15-25	15-20	15-20	15-20
<b>Time(min)</b>	45	45	10-45	45	45	15	45

### 2.3 Post-treatment of MAO Coatings

After ensuring, the MAO-coated substrate is clean and free from contaminants:

- Immerse the substrate in a solution of myristic acid dissolved in methanol for 15 min. Myristic acid has long hydrocarbon chains that contribute to hydrophobicity. The carboxylic acid (COOH) groups of myristic acid interact with the substrate's surface. After immersion, the substrate to dried by hot air dryer, which promotes the formation of a self-assembled monolayer (SAM) of myristic acid molecules on the surface.
- Immerse the substrate in a solution of stearic acid dissolved in chloroform many times. Allow the substrate to dry again, facilitating the formation of a SAM of stearic acid molecules on top of the existing myristic acid SAM. During immersion and drying, the fatty acid molecules undergo a self-assembly process.

### 3. Results and Discussion

#### 3.1 XRD Results

The X-ray diffraction (XRD) results for different coatings scanned in diffraction angle ( $2\theta$ ) from  $10^\circ$  to  $90^\circ$  presented in Figure (2). The phases were identified by comparison with standard reference patterns from files (JCPDS cards). Taken altogether, these results affirm that  $\text{Al}_2\text{O}_3$  ceramic layers were successfully deposited onto the Al substrates. Upon analyzing the XRD data, the detection of mean peaks shared across all samples can be attributed to various forms of Al and  $\text{Al}_2\text{O}_3$  including:

- Al peaks verified using (JCPDS card No. 00-002-1109).
- $\gamma$ - $\text{Al}_2\text{O}_3$ , confirmed with (JCPDS card No. 00-010-0425).
- $\text{Al}_2\text{O}_3$  (also known as Corundum), using (JCPDS card No. 01-075-0787).
- $\text{Al}_2\text{O}_3$  (also known as Boehmite), using (JCPDS card No. 96-901-2275).

Phases of aluminum oxide, specifically  $\gamma$   $\text{Al}_2\text{O}_3$  and  $\alpha$   $\text{Al}_2\text{O}_3$  presence have indeed distinct mechanical properties impact the hardness of the coatings produced through the Micro-Arc Oxidation (MAO) process, which will be discussed later in hardness. ( $\gamma$ - $\text{Al}_2\text{O}_3$ ) is known for its relatively high surface area, which is due to its fine particle size and porous structure. It can exhibit good mechanical properties, including hardness, but it is often less hard compared to  $\alpha$   $\text{Al}_2\text{O}_3$ . The hardness of  $\gamma$ - $\text{Al}_2\text{O}_3$  can vary depending on factors like crystallite size and processing conditions. On the other hand, ( $\alpha$ - $\text{Al}_2\text{O}_3$ ) is typically harder and more wear-resistant than gamma alumina. It has a denser crystal structure and larger crystallite size, contributing to its higher hardness. It coatings are often sought after for applications where hardness and wear resistance are critical. Boehmite exhibits moderate hardness compared to some other alumina phases like corundum ( $\alpha$ - $\text{Al}_2\text{O}_3$ ). Coatings containing boehmite alumina have moderate hardness but can possess other advantageous properties, such as good adhesion, surface energy, and catalytic activity. It is often used in applications where other mechanical, chemical, or surface properties are more critical. All alumina phases mentioned came from rock additions to electrolyte used in MAO process contained various compounds and additives, these compounds influenced the composition of the coatings. During the MAO process, when a high-voltage electrical discharge or spark occurs between substrate and the electrolyte solution, extremely high temperatures generated momentarily at the spark impact point. These high temperatures can lead to the rapid dissociation and ionization of aluminum ions from the electrolyte solution. As the aluminum ions are released at the point of the spark, they can react with oxygen and hydroxyl ions from the solution to form different alumina phases. High temperatures generated by the spark provide the necessary energy for the formation of various  $\text{Al}_2\text{O}_3$  phases, it form at different temperatures due to the kinetics of phase formation vary. Moreover, the rapid cooling that follows the spark impacts the phase composition by trapping certain phases in the coating. This cooling process can influence the final composition of  $\text{Al}_2\text{O}_3$  phases present. The appearance of Al peaks is most likely due to two factors, the thickness of the film coated on the substrate and the penetration depth of the X-rays into Al alloy substrate.

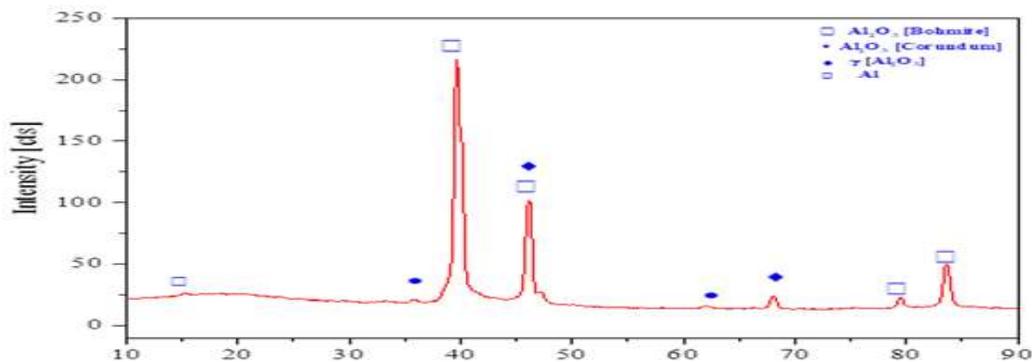


Figure 2: Results of XRD for MC2 Sample.

### 3.2 SEM Results

Figures from (3) to (4) display SEM results show the surface morphology of different coatings at varying magnifications. Observed drawn from these images are the porous structure of the coating layer and the particle distribution of for the samples with thassos added, in figure (3) of sample MC2 it can be observed that the porosity increases with the addition but the shape of pores became more uniform and dense. As well as in figure (4) of sample O, we observed that the porosity decreased with the addition of (7) g to the electrolyte, due to incorporation of thassos components into the micro-pores, which leads to reduce the amount of pores in the structure.

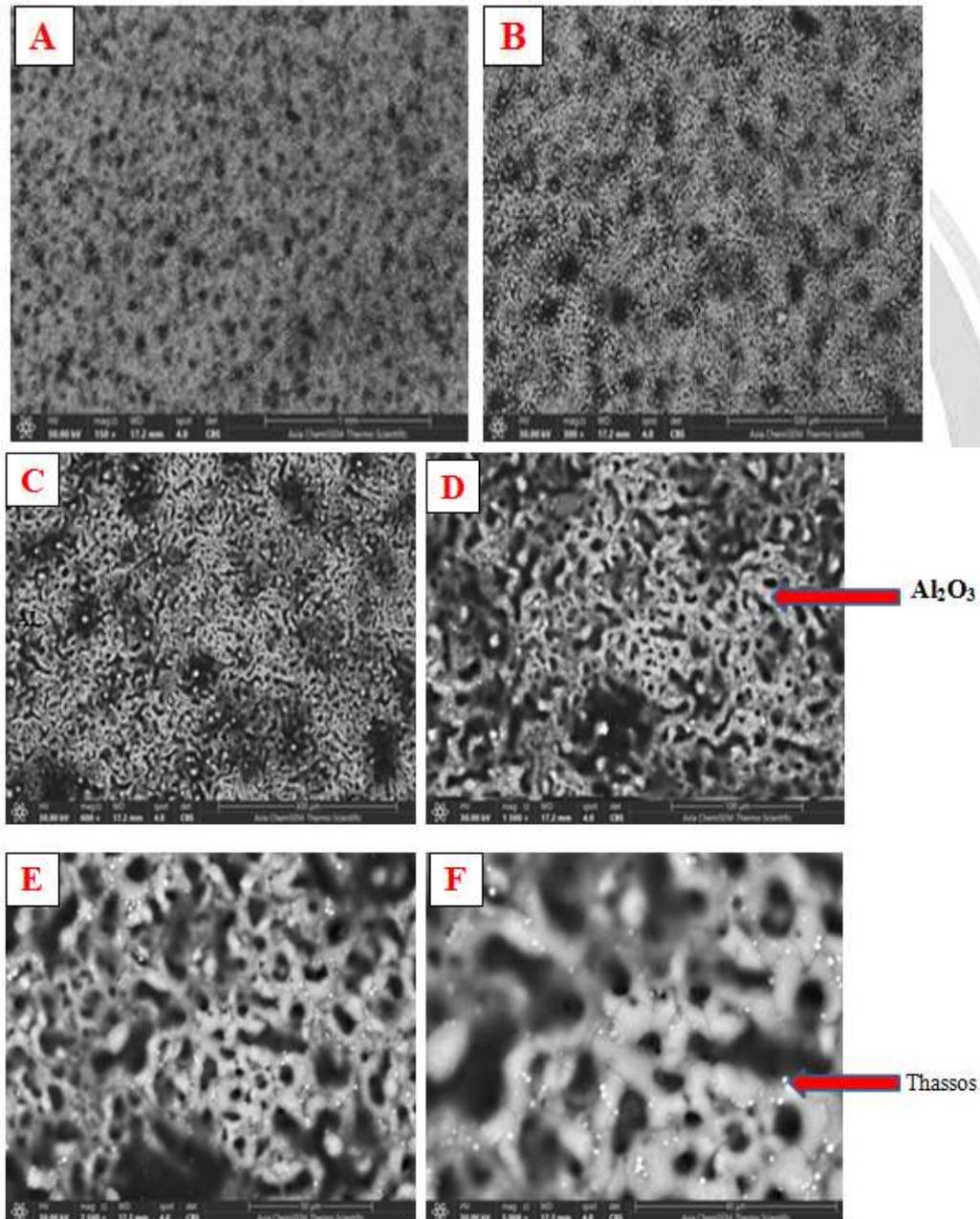


Figure 3: SEM Results for MC2.

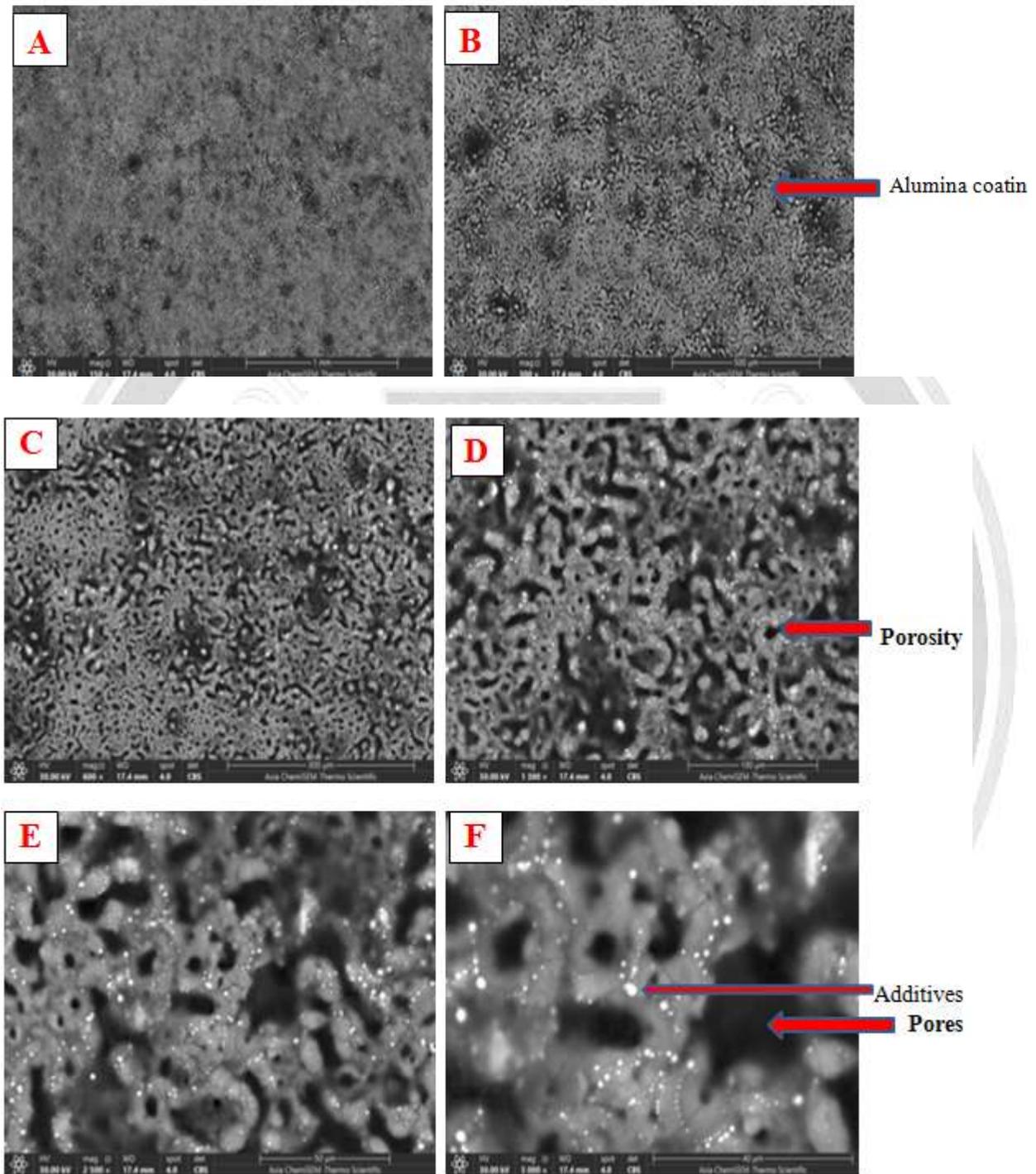


Figure 4: SEM Results for O



### 3.3 Hardness and Thickness Results

Table (3) and Figure (5) presents the results from hardness and thickness to a coating material at thassos particles additives, it is observed that the coatings hardness values were in the range (246.9- 975.9 HV) and hardness results show an increase after coating, explaining the effects of increasing the addition of thassos particles in different ratios and extending the deposition time resulted in an increase hardness. The sample labeled "O" reached a remarkable hardness of 975.9 HV compared to other samples. This increase in hardness attributed to the alteration of the coating's microstructure due to the successful incorporation of Thassos particles into the coating. This, in turn, contributes to the overall improvement in hardness. Natural additives containing MAO electrolytes could improve the Al substrates, proves the combined effect of these on the hardness of the MAO coatings. It is interested to mention that, only the micro-hardness test at 4.9 N and holding time (15sec) could evaluated the coating hardness. It can be concluded that the thickness values were (7.13-19.23)  $\mu\text{m}$ , and the sample S exhibited the highest value (19.23  $\mu\text{m}$ ) at 45 min for thassos additives in comparison with the other samples. The variation of coatings thickness with deposition time can be attributed to the broken of weak oxide layer by strong spark during growth, thereby, forms different thickness values. Generally, the porous nature of oxidized surface makes it possible to impregnate such pores with the number of discrete short-lived micro discharges moving across the Al surface during MAO process.

**Table 3: Hardness and Thickness Results of Thassos Substrates.**

No	Sample	Time (min)	Hardness (HV)	Thickness ( $\mu\text{m}$ )
1	MC1	45	547.4	9.56
2	MC2	45	433.7	14.7
3	Cth1	10	246.9	7.13
4	Cth2	15	347.6	11.1
5	Cth3	30	382.4	12.56
6	Cth4	45	669.2	12.03
7	S	45	724.56	19.23
8	O	45	975.9	14.16
9	THS1	15	750.8	13.06
10	THS2	45	577.9	11.2

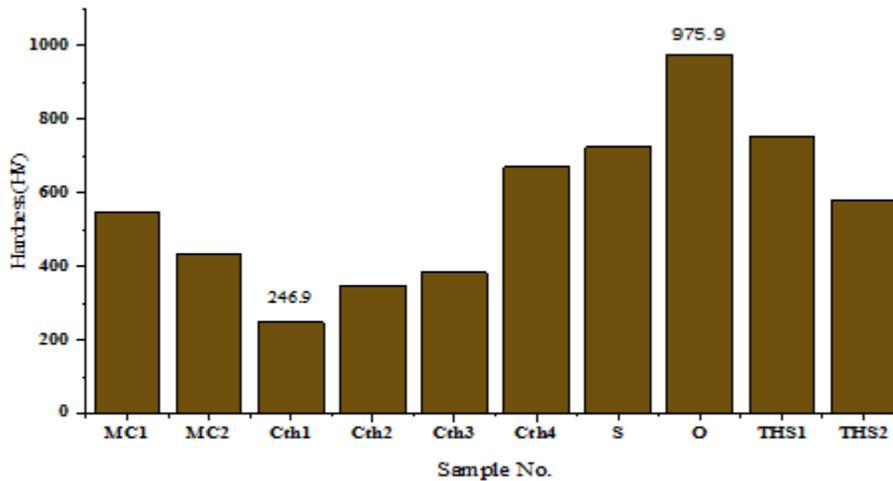


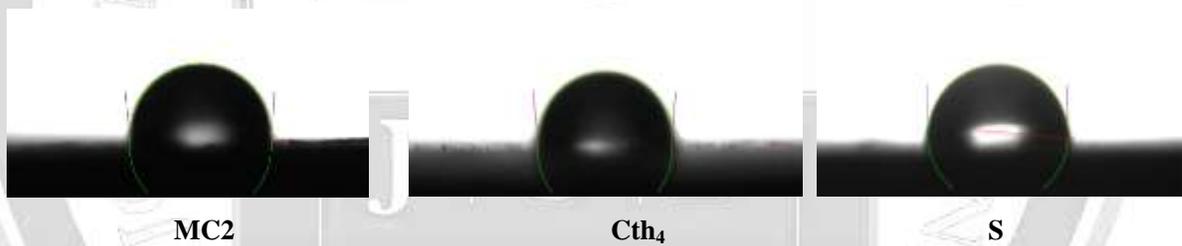
Figure 5: Hardness Results of Thassos Substrates.

### 3.2 Contact Angles (CA) Results

As shown in Figure (6) and Table (4) show contact angle results for coated specimens by thassos rocks. The higher contact angle was associated with hydrophobic properties given by mixing with the addition of 2g of Thassos during a 45-minute deposition time, you observed an increase in the contact angle to  $94.773^\circ$  in sample MC2. This increase in contact angle suggests that the surface became more hydrophobic. A higher contact angle indicates that the surface has become less wettable by water. When 3g of Thassos was added to the coating, you noticed a slight increase in the contact angle to  $95.120^\circ$  in sample Cth4. This slight increase in contact angle is likely attributed to the topography of the surface of the Thassos particles. The surface properties and roughness of these particles can contribute to the hydrophobicity of the coating. In both cases, the addition of Thassos particles seems to have led to an increase in the contact angle, making the surface of the coating more hydrophobic. All alloy substrates after coating own CA of (0.000), due to the coating formed by MAO is usually porous and rough, which cannot effectively prevent the penetration into the coating matrix.

**Table 4: Contact Angle Results for *Thassos* Substrate**

No	Sample	Contact angle (°)
1	MC1	88.596
2	MC2	94.773
3	Cth1	80.764
4	Cth2	90.278
5	Cth3	87.69
6	Cth4	95.120
7	S	90.326
8	O	88.901
9	THS1	88.417
10	THS2	88.698

**Figure 6: Contact Angle Results for *Thassos* Substrates.**

**Conclusion**

1. The success of the present work in using MAO technique with modified electrolytes containing Iraqi natural ceramic additives of thassos and post treatments using fatty acids, specifically (myristic and stearic), as well as chloroform and methanol in depositing of nano-composite ceramic Al<sub>2</sub>O<sub>3</sub> coatings with notable properties of hardness and hydrophobicity together.
2. The results showed that porous and uniform self-cleaning ceramic composite coatings with several noteworthy properties have thickness (7.13-19.23 μm), hydrophobic and self-cleaning in terms of contact angle (90.278°-95.120°), hardness (246.9- 975.9 HV) values could be obtained.
3. The addition (3g) of thassos led to an enhancement in the contact angle of the coating to (95.120°), which indicated the greater hydrophobicity.
4. The higher finding for contact angle was found at a deposition time of 45 min, whereas the greatest results for hardness were found at a coating deposition time of 45 min.
5. The morphology of the coated surfaces showed a typical crater-like porous structure characterized by a clear and uniform distribution of thassos particles evenly dispersed throughout the coating material. Overall, the porous morphology and distribution of particles in the coated surfaces suggest that the coatings have unique microstructures that can influence their performance characteristics in self-cleaning.
6. XRD results proved the deposition of γ- Al<sub>2</sub>O<sub>3</sub> with different contents and other modification elements.
7. The results showed that the research was successful in protecting the surfaces of Al alloy with self-cleaning composite coatings of high hardness, which will help to maintain environmental cleanliness and aesthetics while also protecting them from scratching and external weather conditions.
8. Results from the present research will promote the future works in surface engineering of Al alloys by MAO process using more environmentally friendly, readily available, require minimal processing additives such as Iraqi ceramic natural additives of calcite, bauxite and thassos in deposition of hard and superhydrophobic coatings.



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## ترسيب طلاءات Al<sub>2</sub>O<sub>3</sub> الصلبة والكارهة للماء على سبائك الالمنيوم بواسطة عملية MAO

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جامعة بابل كلية هندسة المواد قسم السيراميك ومواد البناء

### الخلاصة

الهندسة السطحية لسبائك الالمنيوم عن طريق ترسيب الطلاءات الكارهة للماء باستخدام تقنيات مختلفة لتحسين مقاومة التآكل والبلا وخصائص التنظيف الذاتي قد اكتسبت المزيد من الاهتمام في السنوات الأخيرة. هذه الدراسة هي محاولة لاستخدام إضافات ثاسوس في تعديل الإلكتروليتات المستخدمة في عملية أكسدة القوس الصغير MAO لترسيب الطلاءات السيراميكية Al<sub>2</sub>O<sub>3</sub> الصلبة والفائقة الكارهة للماء على سبائك الالمنيوم للتطبيقات التنظيف الذاتي ذات الأحمال الثقيلة. تم استخدام صخور الثاسوس بتركيز مختلفة (1، 2، 3، 5، 7 غم/لتر) كمكونات تعديل. تم ترسيب الطلاءات السيراميكية Al<sub>2</sub>O<sub>3</sub> على سبيكة Al باستخدام وحدة MAO محلية الصنع بجهد مطبق (320-350) فولت، تيار (0.1-0.3) أمبير ودرجة حرارة (15-25 درجة مئوية)، وفي درجات حرارة مختلفة. أوقات الترسيب (10-45 دقيقة). تمت معالجة الأسطح المطلوبة لاحقاً باستخدام الأحماض الدهنية مثل حمض الميريستيك (10 جم / لتر) وحمض دهني (5 جم / لتر) للحصول على خصائص تنظيف ذاتي مهمة. تم استخدام تقنيات واختبارات تحليلية مختلفة لتوصيف الطلاءات مثل المجهر الإلكتروني الماسح (SEM)، وحيود الأشعة السينية (XRD)، والصلابة، والسمك، وقياسات واختبارات زاوية التلامس. تم تحقيق أعلى زاوية اتصال تبلغ 95.120 درجة عند استخدام ثاسوس (3 جرام). تشير زاوية التلامس العالية هذه إلى أن الأسطح شديدة الكارهة للماء، مما يعني أنها تطرد الماء بشكل فعال. أفضل نتيجة لأعلى قيمة صلادة مسجلة كانت 975.9 HV عند استخدام الثاسوس (7 جم) كمضافات. ستعمل نتائج هذا البحث على تعزيز الأعمال المستقبلية في هندسة الأسطح لسبائك الالمنيوم من خلال عملية MAO باستخدام أكثر صديقة للبيئة ومتاحة بسهولة وتتطلب الحد الأدنى من إضافات العملية مثل الإضافات الطبيعية السيراميكية للثاسوس في ترسيب الطلاءات السيراميكية الصلبة والكارهة للماء لمقاومة التآكل وتطبيقات ذاتية التنظيف.

الكلمات الدالة: الطلاء الكاره للماء، MAO، الصخور الطبيعية، رخام ثاسوس.