

Effects of Rice husks ash addition on alumina layers deposited on 2024 aluminum alloys by micro-arc oxidation(MAO).

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ABSTRACT

Aluminum and its alloys are becoming increasingly significant, particularly in the aerospace and automotive manufactures. Anyhow, these substances tend to have poor wear resistance and micro-hardness. So, the aim of this study, formation of ceramic oxide coatings by the micro-arc oxidation (MAO) on 2024 aluminum alloy with rice husks ash containing electrolyte solution. The XRD, EDS, SEM, micro-hardness, thickness, roughness and wear tests were used to evaluate the microstructure, morphology of deposited phases and coating properties. The results proved that the formation of hard ceramic coatings were values (246.8-496.6)HV. Roughness increased with deposition time increasing (2.320-6.837) μm , thickness of coatings variation with deposition time (25-48.8) μm . The structure of coatings was characterized by the porosity, with increasing RHA ratio from (5 to 15)g the structure became less porous particularly with increasing deposition time to 45min. Also, the RHA addition could improve wear resistance by means of decreasing loss weight from 0.0017 to (0.0006-0.0005)g and decreasing friction coefficient from 0.66 to (0.34-0.52) for (15g/l RHA, 5g/l KOH), (10g/l KH_2PO_4 , 4g/l H_3PO_4 , 5g/l NaCO_3 , 6g/l $(\text{NH}_3)_2\text{MO}_7\text{O}_{24}$, 4g/l KOH, 6g/l RHA), respectively. Finally it can be concluded that the properties of ceramic oxide coatings improved with addition of natural additive (rice husks ash).

KEYWORDS: Micro-arc oxidation, aluminum alloys, rice husk.

INTRODUCTION

Alumina coatings are very effective in developing wear resistance surfaces for Al alloys that also exhibit excellent corrosion protection. A number of deposition techniques such as arc-discharge plasma, gas-flame spray, vacuum deposition methods and high temperature glass enameling have been investigated to produce ceramic coatings on Al and its alloys. These techniques require a high substrate temperature to provide adequate coating adhesion under high contact loads [1]. Micro Arc Oxidation (MAO) which sometimes is called Plasma Electrolytic Oxidation (PEO) or Spark anodizing [2]. MAO is a promising novel process to form hard and thick Ceramic-like coatings on valve metals (Al, Mg, Ti, etc.) and their alloys. MAO is a complex process combined with simultaneous partial processes, including oxide film formation, dissolution, and dielectric breakdown [3], valve metal is positioned as the anode in an electrolytic cell, the precursor to spark deposition is anodization, as the voltage is raised, an anodic oxide film is induced to grow. However, above a certain applied potential, film thickness no longer increases uniformly and dielectric breakdown occurs. The breakdown of the initial barrier

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layer is seen in the form of many freely moving sparks. The movement of the sparks is attributed to the deposition of material and localized heating at one spot with subsequent sparking at nearby weak spots in the coating. The applied voltage sufficient to cause dielectric breakdown varies with the anode material, and electrolyte composition and temperature[4]. The literature review shows that the influence of the electrical parameter on the mechanism of the coating formation and its phase composition such as using cathodic currents[5], unipolar mode with AC power supply[6], and DC current[7], also studied the influence of using different additives to basic electrolytic solution (sodium silicate and potassium hydroxide) additives such as Na_3AlF_6 [8], rutile TiO_2 nano-additive[9], KF [10], and Na_2HPO_4 [11] etc.. So in the present study concentrated on the using natural and alternative source for silicate, such as rice husk ash extracted from rice husk.

Rice husk is one of the most abundant by-products produced in the paddy field. The agricultural by-product includes rice husk, rice straw, plant materials and so on. Of all these by-products, rice husk contains more than 95% silica. Hence, several attempts have been made to produce bulk silica from the most eco-friendly and economical source rice husk. Rice husk is an excellent source for the production of high-grade amorphous bulk silica powder [12]. The surface and mechanical properties of the MAO Al_2O_3 coatings with RHA micro-grains were examined.

2. Experimental:

2.1 Powder preparation:

Rice husks ash powder was prepared from rice husks, first a suitable amount of rice husks were cleaned perfectly with distilled water to remove dust and impurities. They were drying at the atmosphere and burning at temperature 700°C for 8hr using the firing furnace. After milling for 12hours, the powders were tested by particle size analyzer. The mean particle size was about ($8\mu\text{m}$), and the chemical composition shown in table(1).

Table1: Chemical composition of rice husks ash.

RHA	SiO_2	Al_2O_3	CaO	SO_3	L.O.I	P_2O_5
Content(wt.%)	90.42	0.50	2.15	0.44	-	0.22

2.2 Preparation of Al alloy Substrates:

2024AA aluminum alloy samples with ($\phi 25\text{mm} \times 4\text{mm}$)mm were cut from aluminum bar, the hardness of Al substrate was 157HV and chemical composition of aluminum substrate shown in table(2). Prior to coating process the 2024AA samples were polished with silicon carbide paper with (400 to 2000)# grit emery, to get surface roughness Ra about $0.896\mu\text{m}$, cleaned with detergent typically acetone, then washed with distilled water, to be used for MAO process.

Table2: Chemical composition of 2024AA aluminum alloy.

2024AA	Si	0.177
	Fe	0.427
	Cu	4.68
	Mn	0.685
	Mg	1.19
	Cr	0.064
Content(wt.%)	Ni	0.005
	Zn	0.013
	Ti	0.009
	Pb	0.017
	V	0.004
	Al	Bal.

2.3 Micro-arc oxidation process:

Deposition of the ceramic coating was done by a home-made MAO unit shown in fig.(1) with voltage of (0-500V), and current of (0-2A). The unit consists of thermometer for measuring temperature of electrolytic solution during deposition process, a high voltage DC-AC power supply, a plastic container for the electrolyte equipped with a sample holder (anode) and the cathode holder, mixing system, and cooling system. The positive output of the power supply was connected to the sample immersed in the electrolyte serving as the working electrode anode(2024 aluminum alloy) and the negative output was connected to the stainless steel(316L) plate acting as the (cathode). During the process electrolytic solution was mixed and cooled to prevent heating over to 25°C using the cooling unit connected to the micro-arc oxidation coating unit. This cooling unit, was equipped with a distilled water in plastic container surrounded the electrolyte plastic container. The deposition method was controlled to form a ceramic layer of designated conditions, then the coated Al sample was uncoupled from the sample holder, washed with water and dried in warm air. The preparation of electrolytic solutions was done through the selection of suitable electrolyte components which

mixed in liter of distilled water, then mixed by magnetic stirrer for 3hours before the deposition process, PH of solution (7-8). Tables (3) and (4) show the electrolyte compositions and deposition parameters.

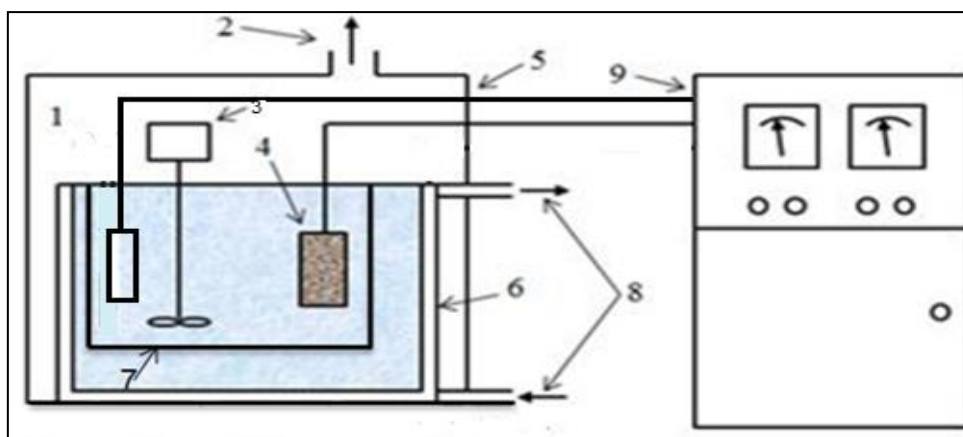


Fig.1:Typical arrangement of the equipment used for MAO coating: (1) stainless steel plate (cathode), (2) exhaust/ventilation system, (3) mixer, (4) Sample(anode), (5) Plastic case, (6) plastic container of cooling bath, (7) plastic container of coating electrolyte, (8) flow circulation via cooling system/filter, (9) power supply unit[13] after disposition.

Table 3: Composition of rice husks ash containing electrolyte(type A).

Composition	Sample code	Deposition time(min)
(5g/l) RHA, (5g/l) KOH	A ₁	15
	A ₂	30
	A ₃	45
	A ₄	60
(5g/l)RHA, (3g/l) KOH	A ₅	15
	A ₆	30
	A ₇	45
	A ₈	60
(10g/l)RHA, (5g/l) KOH	A ₉	15
	A ₁₀	30
	A ₁₁	45
	A ₁₂	60
(15g/l)RHA, (5g/l) KOH	A ₁₃	15
	A ₁₄	30
	A ₁₅	45
	A ₁₆	60

Table4: Composition of basic materials and RHA containing electrolyte(type B).

Composition	Sample Code	Deposition time(min)
(10g/l)KH ₂ PO ₄ ,(4g/l)H ₃ PO ₄ , (5g/l)NaCO ₃ , (6g/l)(NH ₃) ₂ MO ₇ O ₂₄ , (4g/l)KOH, (6g/l)RHA	B ₁	15
	B ₂	30
	B ₃	45
	B ₄	60

2.4 Apparatuses and characterization:

Phase composition of the coatings was studied using an X-ray diffractometer(XRD-6000 SHIMADZU, Japan, Cu K α radiation, 40KV, 30 MA, 6/min scanning speed), the observations were complimented with an energy dispersive x-ray spectroscopy (EDS) for analysis of elemental distributions across the surface, microstructure of coatings were studied using scanning electron microscope (SEM,TESCAN/ VEGA II Series/ USA). Micro-hardness of the coatings were evaluated employing a Vickers indenter(HVS-1000, Laryee, digital Micro-hardness tester) at 9.8N load. Thickness of coatings was measured using Microprocessor CM-8822, coating thickness meter. Roughness of coatings was measured using(SRT-6210,surface roughness test), and coefficient friction and wear resistance of coated samples were tested with(microtest-28021).

RESULTS AND DISCUSSION

3.1 MAO spark conditions:

Tables(5) and (6) show the current-voltage values could prove the possibility of using natural additive containing electrolytes in the work in formation of ceramic oxide coatings by using of moderate current and voltages in the range (0.1-0.2)A and (336-350)V, respectively. It can be noted that, spark could be appeared above 200V in the electrolyte solution.

Table5: MAO spark condition using RHA containing electrolyte.

Composition type(A)	Current (A)	Voltage (V)
A ₁	0.1	337
A ₂	0.1	336
A ₃	0.1	337
A ₄	0.1	337
A ₅	0.1	337
A ₆	0.1	338
A ₇	0.1	338
A ₈	0.2	338
A ₉	0.1	338
A ₁₀	0.1	338
A ₁₁	0.1	338
A ₁₂	0.1	338
A ₁₃	0.1	339
A ₁₄	0.1	337
A ₁₅	0.1	338
A ₁₆	0.2	336

Table 6: MAO spark condition using basic materials and RHA containing electrolyte.

Composition type(B)	Current (A)	Voltage (V)
B ₁	0.2	349
B ₂	0.2	350
B ₃	0.2	348
B ₄	0.2	350

3.2 XRD and EDS results:

Figs.(2) and (3), show the XRD results of the of the coated samples by using rice husks ash containing electrolyte. Also, fig.(4), and table(7) show the EDS results of these samples. XRD analysis of MAO layer showed the aluminum peaks (JCPDS No. 004-0787) coming from the underlying substrate were ditto detected due to the penetration of X-rays into the aluminum alloy substrate, α -Al₂O₃ phase (JCPDS No. 046-1212) and γ -Al₂O₃ (JCPDS No. 010-0425) phase. The formation of α -Al₂O₃ phase in the coating is preferred rather than the meta-stable γ -Al₂O₃ phase due to its higher hardness and higher stability, the dominated peaks of γ -Al₂O₃ were existed at 19.8537° and α -Al₂O₃ at 43.7625°. No silicon-related peak was observed in the XRD spectra of the MAO-RHA sample, possibly due to the low amount of silicon content in the alumina coating, this results agreement with [14]. EDS Analysis proved the existence of Al and O element in the coatings, thereby, denoted to the formation of alumina Al₂O₃ ceramic layers modified with another elements of C, and Si at different weight of these modification elements.

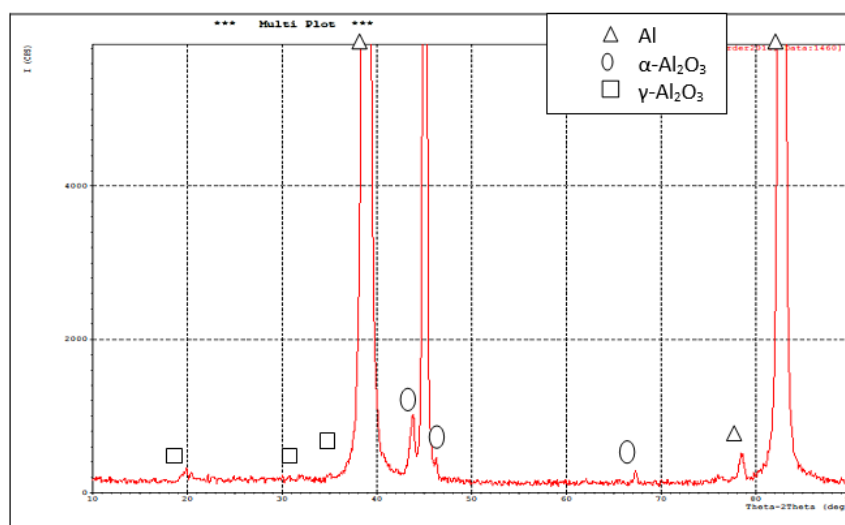


Fig. 2: XRD chart of coated sample(A₁₆).

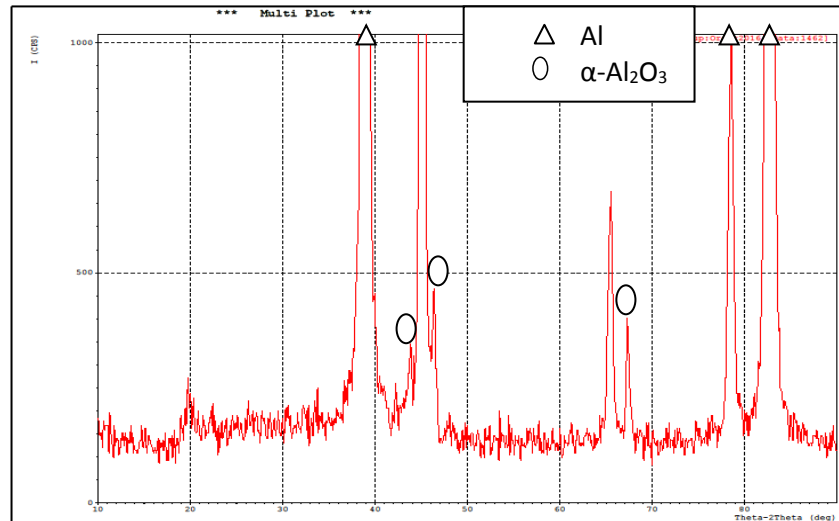


Fig.3:XRD chart of coated sample(B₄).

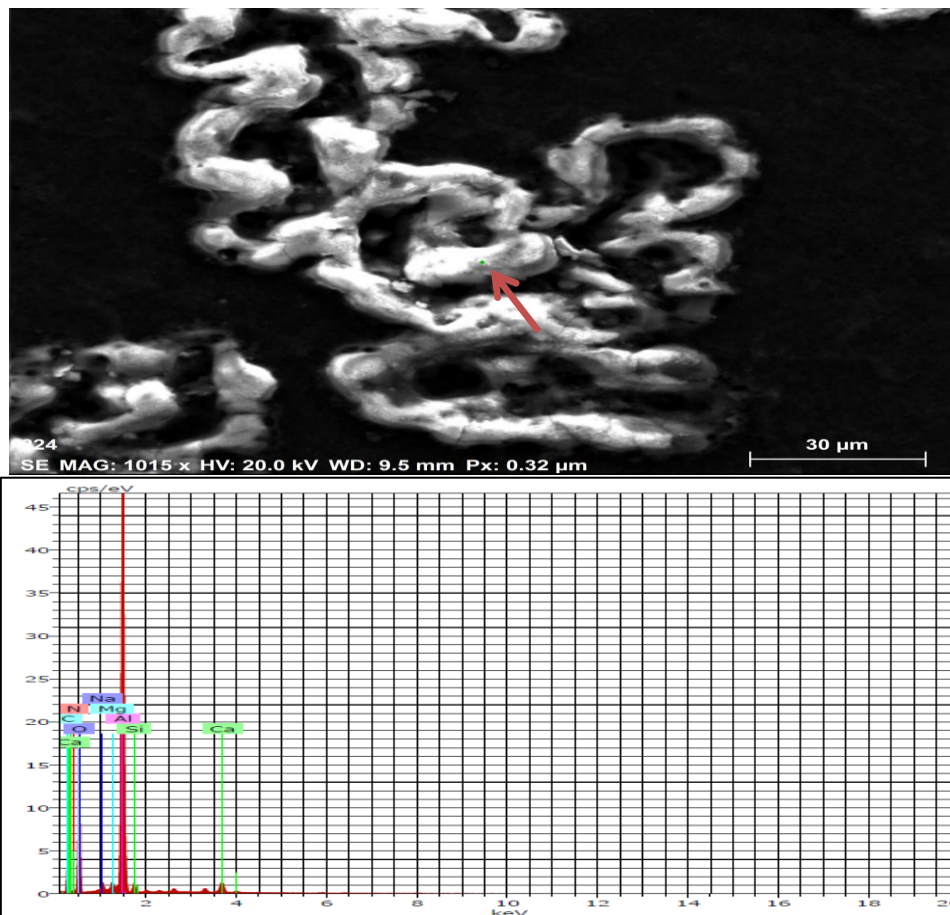


Fig. 4:EDS results for coated samples A₁₆.

Table7: EDS results of coated sample A₁₆.

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	54.73	40.76	43.14	6.25
Al	13	K-series	39.76	29.61	18.59	1.92
C	6	K-series	31.59	23.53	33.18	4.04
N	7	K-series	3.38	2.51	3.04	0.68
Ca	20	K-series	2.11	1.57	0.66	0.09
Na	11	K-series	1.13	0.84	0.62	0.10
Mg	12	K-series	0.84	0.63	0.44	0.07
Si	14	K-series	0.73	0.55	0.33	0.06
Total:			134.28	100.00	100.00	

3.3 SEM results of coated samples:

Figures from(5) to (9) show the SEM results for the surface morphology of the coated samples A₁₃, A₁₄, A₁₅, A₇ and B₃ at different magnifications. It can be observed that the morphology of the coated samples was characterized by pores with different sizes. In general, sample A₇(5g RHA at 45min) showed the highest porosity and sample A₁₅(15g RHA at 45min) showed the lowest porosity among the other. It can be concluded that the increasing of the RHA ratio from (5 to 15)g and deposition time to 45min could decrease the porosity in the structure due to the RHA ratio increasing. Considering the effects of pores in ceramic coatings on the resulted hardness, such low porosity will have its effects on increasing the micro-hardness. Anyhow, samples A₁₅ recorded the lower hardness in comparison with samples A₁₃, A₁₄. As it would be expected, the hardness differences can be also strongly attributed to the non-uniform distribution of pores in sample A₁₅ coatings and the deposited phases of α - and γ -alumina.

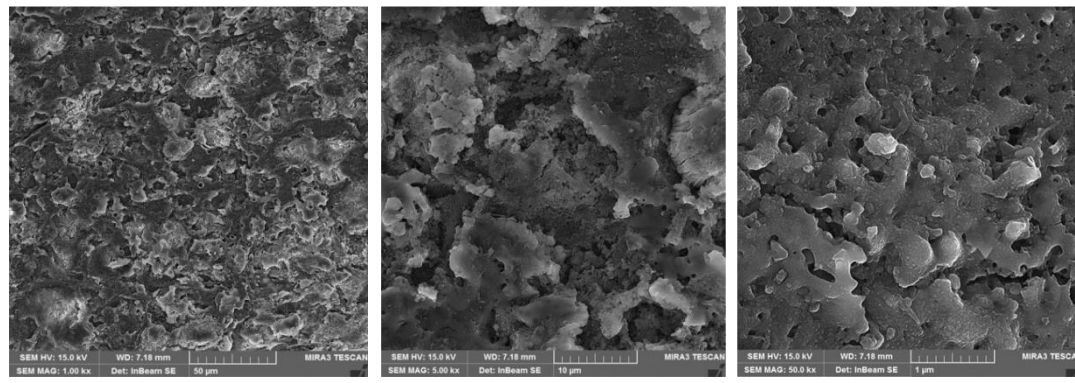


Fig.5: SEM results for the surface morphology of the sample A₁₃ (15min), at different magnifications.

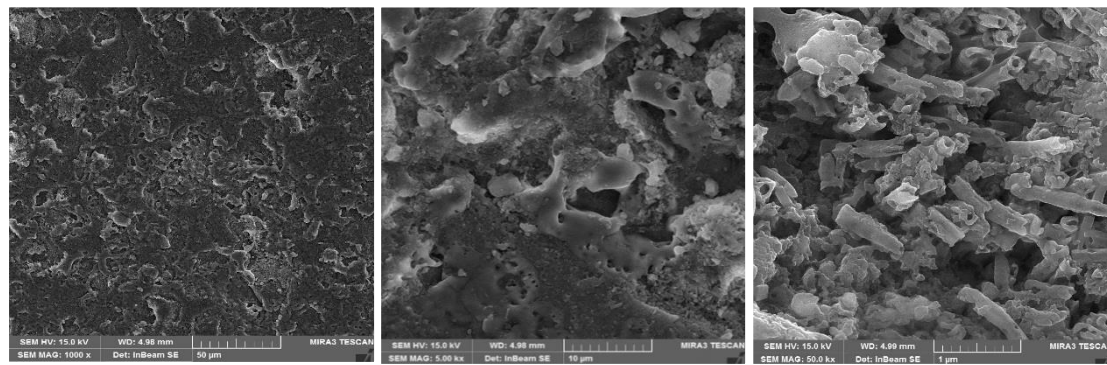


Fig.6: SEM results for the surface morphology of the sample A₁₄ (30min), at different magnifications.

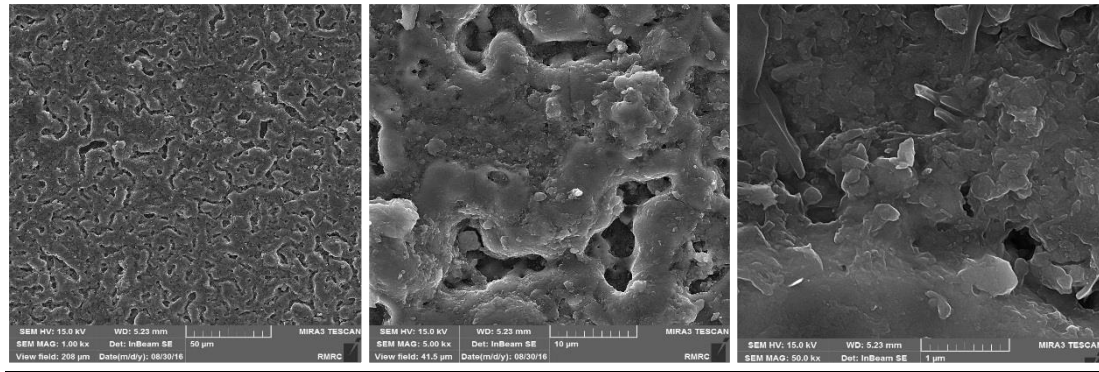


Fig.7: SEM results for surface morphology of the sample A₁₅ (45min), at different magnifications.

Morphology of coated samples A₇ is characterized by distribution of pores different size in the structure of coating, with high roughness lead to less micro-hardness than (A₁₃, A₁₄, and A₁₅).

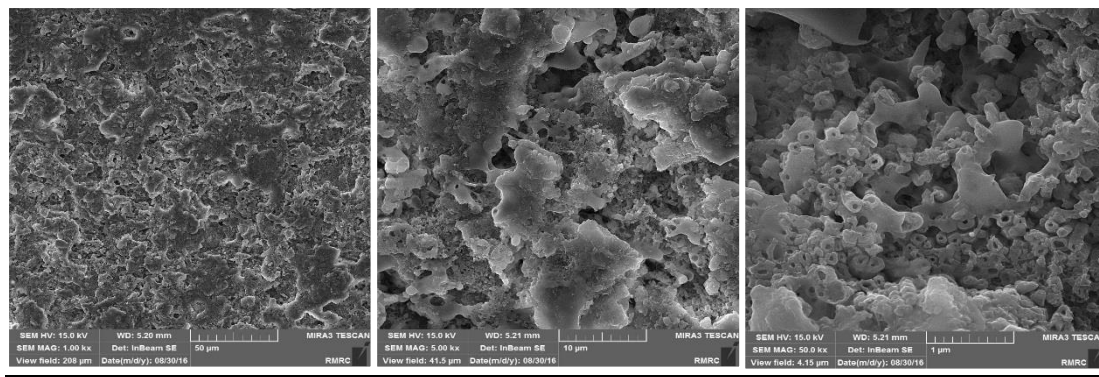


Fig.8: SEM results for surface morphology of the sample A₇ for (45min), at different magnifications.

The surface of the coated samples B had a typical morphology, characterized by cellular pores distribution in the structure, which can enhance of roughness of coatings.

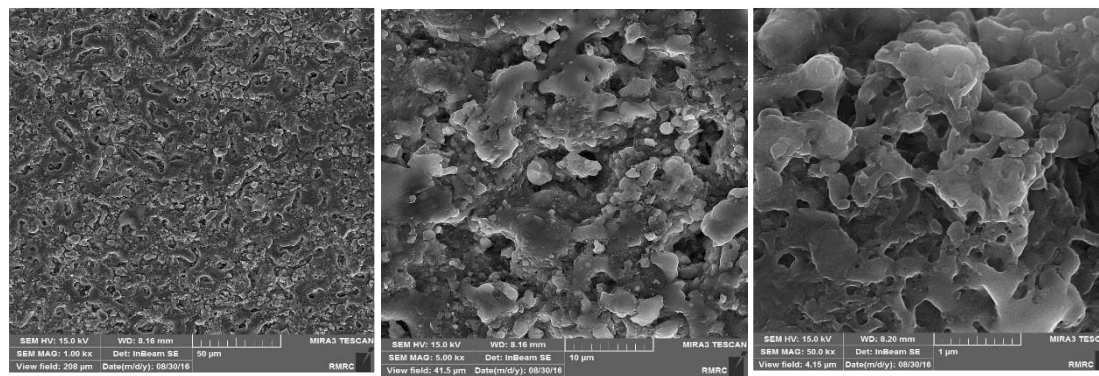


Fig.9: SEM results for surface morphology of the sample B₃ (45min), at different magnifications.

3.4 Thickness, Hardness and Roughness results of coatings:

Table(8) shows the resulted coatings thickness, hardness and roughness after different deposition times. In the present study thickness of coatings varied with deposition time due to broken of weak oxide layer during growth by strong spark, that give different value of thickness. It can be observed that the thickness value were (25-48.8) µm, the sample coated A₁₆ recorded the highest thickness among the others(48.8 µm at 60 min), while A₁₃ recorded the lowest thickness(25µm at 15min). Generally, it can be observed that the coating thickness increased with increasing deposition time using electrolytes, containing rice husks ash . The formation number

of oxide is relatively increased in a unit time, promoting the growth rate of ceramic coating, but when the thickness increase to a certain limit, the cathodic voltage constrained to break down the coatings[15].

It is interested to mention that, only the micro-hardness test at load of (9.81N) and holding time (15sec) could evaluated the coating hardness. In general the using loads of (100, 200, 300, 400, 500)g could not give any track in the coated sample surface. The hardness of dense layer is relatively high, while hardness of pores layer is rather low; there is a transition zone between the coatings and near the substrate interface with its own substrate material and ceramic coatings material, therefore the hardness is rather low in this region, which further promotes metallurgical bonding between ceramic coatings and substrate[16]. For samples A₁₃-A₁₆, it can be observed that the samples A₁₆ (5g KOH, 15g RHA at 60 min) could give the highest hardness (496.6HV) at high thickness (48.8 μm) due to their ceramic oxide content and less pores structure observed in SEM results with increasing RHA additives. Such results prove the effects of using RHA additives in providing the ceramic oxide layers with high hardness(496.6HV). The deposited ceramic oxide layers exhibited surface roughness in the range(2.320-6.837)μm. The samples A₁₃ and A₁₆ recorded the lowest and the highest roughness, respectively, among the others. Generally, the coating roughness increased with deposition time increasing. There was a clear increasing in coatings roughness with deposition time increasing due to coatings thickness increasing, this results agreement with the surface roughness increases significantly on the thicker coatings[17].

Table8: Thickness, micro-hardness and roughness results.

Samples	Time (min)	Thickness (μm) at the center	Micro-hardness(HV)	Roughness Ra (μm)
A				
A ₁₃	15	25	435.7	2.320
A ₁₄	30	32.2	390.6	2.671
A ₁₅	45	29	311.7	3.536
A ₁₆	60	48.8	496.6	6.837
B				
B ₁	15	36	293.9	-
B ₂	30	25.4	285.2	-
B ₃	45	43	276.9	-
B ₄	60	31.5	246.8	-

3.4 Wear results:

Table (9), and fig.(10), and fig.(11) show the wear results for aluminum alloy substrate. Table(10),figs. from (12), to (15) show the wear results for the A₁₃ to A₁₆ samples. Table(11) shows the wear results for the B₁ and B₄ samples. In general, the results showed that the weight losses increased with loading time increasing. In comparison, with Al alloy substrate, the coated samples could prove the good wear resistance of the ceramic oxide layers which resulted in less weigh losses. Furthermore, ceramic oxide layers deposited using the RHA containing electrolyte gave the best wear resistance (0.0006g for sample A₁₃), due to improved hardness, and loss weight of B₁ (0.0005g) . For the friction coefficient of coatings (A) derived from the wear test, it can be identified that the (A) for coatings was in the range (0.34-0.94),the reduction in the friction coefficient arises from the improved surface hardness and reduced porosity of the

MAO Al₂O₃ coatings[17], the lower friction coefficient of the MAO coatings and its higher coating hardness are responsible for its higher wear resistance[14].

Table9: Loss weight of Al alloy substrate.

Sample	Test time (min)	Loss weight(g)	μ
2024(AA)	15	0.0005	~0.66
		0.0010	
		0.0017	

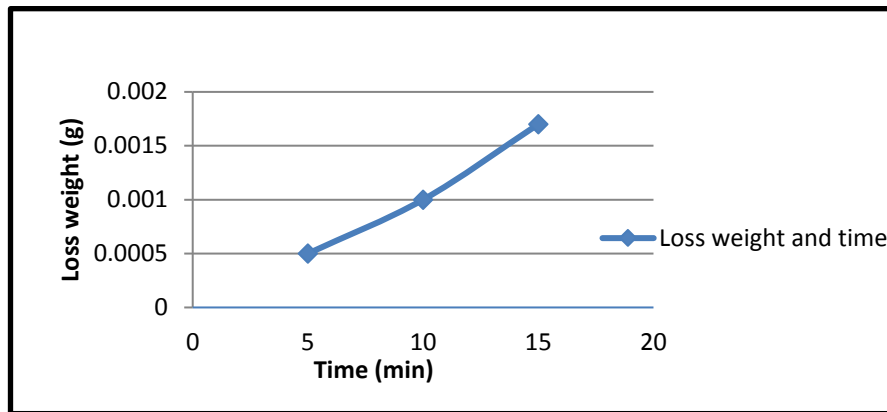


Fig.10: Relationship between loss weight and test time for Al alloy substrate.

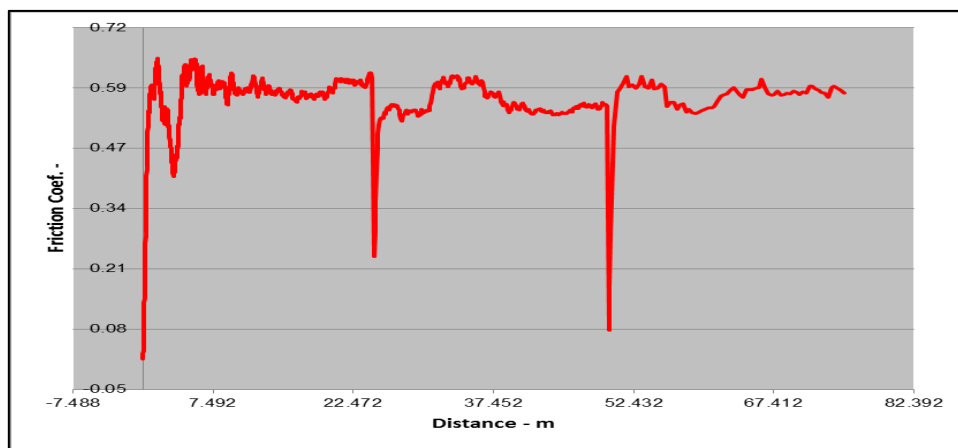


Fig.11: Friction coefficient results for Al alloy substrate.

Table10: Loss weight of coated samples (A₁₃-A₁₆) .

Samples	Test time (min)	Loss weight(g)	μ
A ₁₃	15	0.0002	~0.34
		0.0004	
		0.0006	
A ₁₄	15	0.0002	~0.87
		0.0007	
		0.0009	
A ₁₅	15	0.0003	~0.94
		0.0007	
		0.0012	
A ₁₆	15	0.0002	~0.74
		0.0005	
		0.0008	

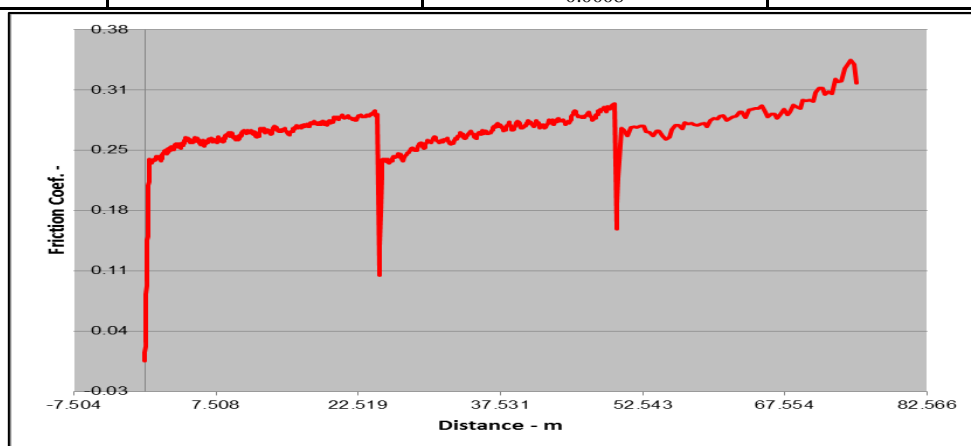


Fig.12: Friction coefficient results for A₁₃ (15min).

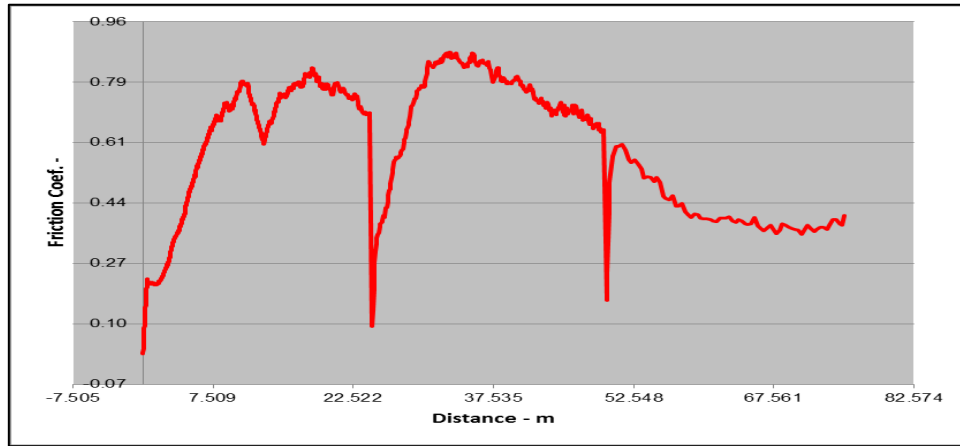


Fig.13: Friction coefficient results for A₁₄ (30min).

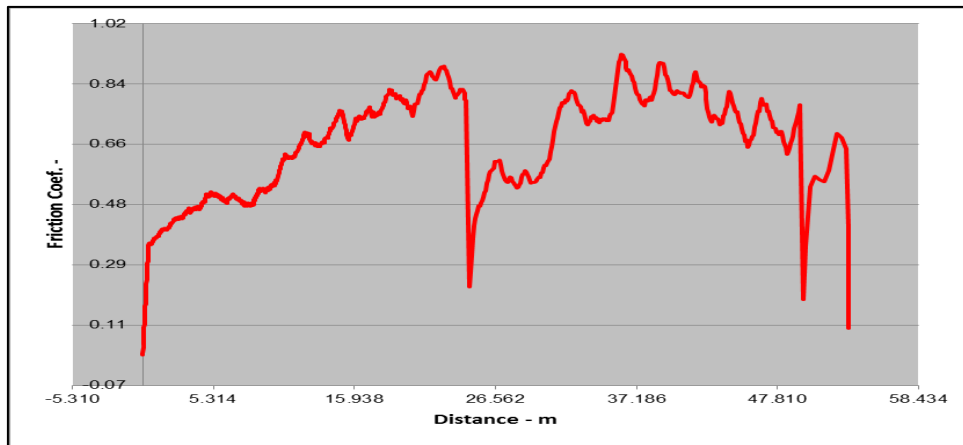


Fig.14: Friction coefficient results for A₁₅ (45min).

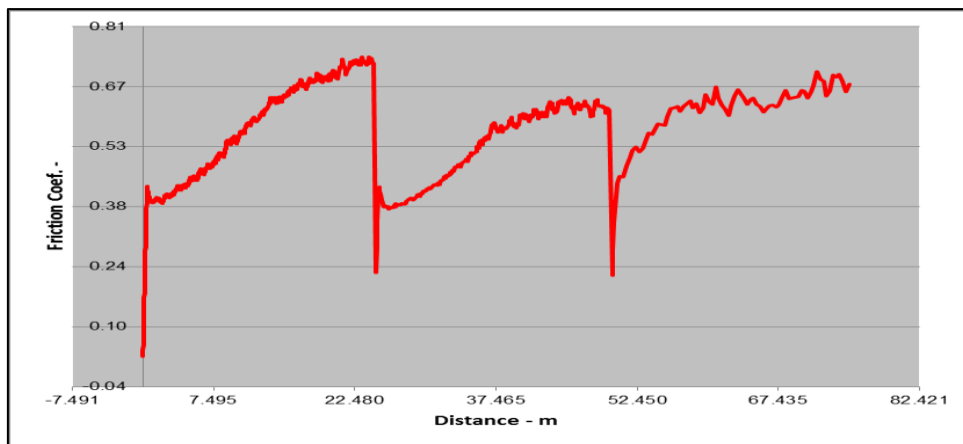


Fig.15: Friction coefficient results for A₁₆ (60min).

Table11: Loss weight of coated samples(B₁, and B₄).

Sample	Test time (min)	Loss weight(g)	μ
B ₁	15	0.0002	~0.52
		0.0004	
		0.0005	
B ₄	15	0.0011	~0.97
		0.0026	
		0.0031	

Conclusion:

The most important facts that may be useful to be mentioned in this study are as follows:

- 1- It is possible to deposit thick, hard and anti-wear ceramic coatings on 2024 Al alloy substrates by micro-arc oxidation process by using natural additive in the electrolytic solution such as rice husks ash (RHA).
- 2- Natural additives such as RHA could be extracted from rice husk with particle sizes in the range (8μm).
- 3- XRD results proved that, the deposition of ceramic oxide coatings on aluminum substrate, coating contain of alumina with two phases : harder and stable phase (alpha alumina) and meta stable phase (gamma alumina). EDS analysis showed that, the ceramic oxide coatings with different contents and other modified elements like (Si, C, Ca etc.) by (MAO) using the electrolytic solution used in this study at voltage (336-350)V, current (0.1-0.2)A, controlled temperature (5-25)°C and various deposition time (15, 30, 45 and 60) minute.
- 4- The MAO ceramic oxide layers with thicknesses (25-48.8)μm could improve the Al alloy substrate hardness from (157HV) to (246.8-496.6)HV. Hardness differences is strongly affected by the non-uniform distribution of pores despite the resulted coating thickness.
- 5- The coatings roughness (2.320-6.837)μm increased with the deposition time due to thickness increasing. Roughness of coating is very important property because it is effect on the tribological properties such as friction increase with high value of roughness.
- 6- Less loss weight and friction coefficient of coated samples of A₁₃(15g/l RHA, 5g/l KOH, and B₁(10g/l KH₂PO₄, 4g/l H₃PO₄, 5g/l NaCO₃, 6g/l (NH₃)₂Mo₇O₂₄, 4g/l KOH, 6g/l RHA) due to high hardness of coatings.
- 7- The results indicate the success of the study in the use of natural materials in the production of ceramic oxide coatings with excellent specifications material on aluminum alloy substrate via micro-arc oxidation.

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