



STUDY ON Al_2O_3 COATING DEPOSITED ON 2024 Al ALLOYS BY MICRO ARC OXIDATION (MAO) USING TiO_2 NANO ADDITIVES.

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ABSTRACT

In this work, the effect of adding natural additives such as rice husks ash and nano additives such as TiO_2 to the electrolyte on the properties of ceramic oxide coating formed on 2024 Al alloy substrate by micro arc oxidation (MAO). Results proved that the formation of hard ceramic oxide coatings were values (313 to 531.3)HV. Roughness increased with deposition time increasing, thickness of coatings variation with deposition time. Morphology of coated sample for 45min with electrolyte compositions (10g/l KH_2PO_4 , 5g/l $NaCO_3$, 4g/l H_3PO_4 , 6g/l $(NH_3)_2MO_7O_{24}$, 3g/l RHA, and 9g/l KOH, 1g/l nano TiO_2) showed the dense structure and uniform pores distribution, increasing the addition of TiO_2 to 3g/l the results showed that more porous structure. Wear resistance of coatings improved by means of decreasing loss weight from (0.0017g of substrate) to (0.0003-0.0009) of samples at (15min and 60min) respectively, with composition (1g/l nano TiO_2 , 10g/l KH_2PO_4 , 5g/l $NaCO_3$, 4g/l H_3PO_4 , 6g/l $(NH_3)_2MO_7O_{24}$, 3g/l RHA, and 9g/l KOH).

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

دراسة طلاء الالومينا Al_2O_3 المرسبة على الالمنيوم 2024 بواسطة الاكسدة المايكروية باستخدام مضافات ثاني اوكسيد التيتانيوم TiO_2

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الخلاصه

تم في هذا البحث دراسة تأثير أضافة المواد الطبيعيه مثل قشور الرز المحروقه ودقائق TiO_2 النانويه الى المحلول الالكتروليتي على مواصفات الطلاء المرسب على قواعد سبائك الالمنيوم 2024Al بطريقة الأكسدة المايكرويه (MAO). اثبتت النتائج تكوين طلاء سيراميكي يمتاز بصلاده عاليه (313 to 531.3)HV أن خشونة الطلاء تزداد بزيادة زمن الترسيب بينما سمك الطلاء يتغير مع الوقت. البنيه المجهرية للعينات المطلبه بزمن ترسيب 45 دقيقه تتميز ببنيه كثيفه و يوجد مسامات مايكرويه منتظمة التوزيع عند استخدام محلول يتألف من (5g/l $NaCO_3$, 10g/l KH_2PO_4 , 4g/l H_3PO_4 , 6g/l $(NH_3)_2MO_7O_{24}$, 3g/l RHA, 9g/l KOH, and 1g/l nano TiO_2) مع زيادة الدقائق النانويه الى 3 g/l فإن بنية الطلاء تتميز بوجود مسامات بنسبه أعلى. زيادة مقاومة بلى بشكل ملحوظ عند أستخدام (1 g/l) وبزمن ترسيب 15 و 60 دقيقه حيث وجد انخفاض كبير في الوزن المفقود عند مقارنة النتائج مع الماده الاساس من 0.0017g الى 0.0003-00.0003g) وهذا يعود الى الصلاده العاليه للطلاء السيراميكي .
مفاتيح الكلمات: عمليه الأكسده المايكرويه, سبائك الالمنيوم, قشور الرز و أوكسيد التيتانيوم .

INTRODUCTION

Light metals, particularly aluminum and aluminum alloys, are existing increasing implementations in various industries like aerospace and automotive because of their low density, strength to weight ratio compared with steel, ease of recycling possibility and fabrication, these metals require careful finishing to generative surface coatings with sufficient resistance to both corrosion, and wear for they are low hardness and have reactive [V. Dehnaviet. al., 2013]. Lately a number of deposition processes, like gas-flame spray, arc-discharge plasma, high temperature glass enameling and vacuum deposition have been attained to produce layers on light alloys, to increase their corrosion resistance, and wear resistances. Anyhow, these processes require high-energy consumptions to provide sufficient layers adhesion under high osculate loads. So, a novel electrochemical surface treatment method, Micro-Arc oxidation(MAO), was introduced, which could form ceramic oxide layers, with low energy consumption[K. Wang et. al., 2009] . Micro-Arc Oxidation (MAO) is a method utilized to produce relatively thick (up to hundreds of micro-meters) ceramic oxide coatings on metals, basically: Mg, Al and Ti and their alloys. Like of films have good mechanical attributes, thermal and corrosion resistances. Very good mechanical attributes (high wear resistance and micro-hardness) are regarded to the existence of the crystalline structure of the oxide film. In the instance of aluminum basically α -alumina and γ -alumina are discovered in the oxide film. Coatings produced via MAO have different implementations, particularly where high wear resistance is required. The mechanism of coatings production during MAO can be divided into three phases that occur instantaneously, first stage "it is an electrochemical formation of oxide on the metal-oxide interface, thus the electrolyte have to migrate through oxide layer towards substrate", second stage "is a chemical dissolution of oxide to gel form on the oxide-electrolyte interface"[W. Gebarowski, and S. Pietrzyk, 2013] . **Third** stage "is associated with high voltage that is enough to dielectric breakdown of the oxide layer. These breakdowns occur as short-living micro-discharges uniformly distributed on the whole surface of the anode, they cause production of discharge channels, from which molten aluminum is ejected. Ejected aluminum is forthwith oxidized, hydrolysed and precipitated on the anode surface". In addition, due to elevated temperature inward the discharge channel, and comparatively low thermal conductance of the oxide phase transmigrations from amorphous alumina and hydrolysed form to crystalline phase can conversing [W. Gebarowski, and S. Pietrzyk, 2013]. Many researches concentrated on the using different electrical parameter with conventional electrolytes solution such as (silicate, aluminate, and phosphate) solution, so in this work concentrate on the using natural(as a silica source) and nano additives prepared ceramic oxide coatings consisting nano particles with good microhardness and wear resistance. Rice husk is one of the further plentiful via-products formed in the rice domain, the agricultural via-product consists rice straw, rice husk, and plant materials. Anyway rice husk includes more than 95% silica. Therefore, numerous efforts have been made to produce bulk silica ash from the most economical and eco-friendly source rice husk[R. Yuvakkumar et. al., 2014]. In this work, in order to improve the structural and properties of ceramic oxide coatings prepared by MAO method, a new electrolyte based on rice husks ash solution with different concentrations of TiO_2 nano-additive was used to fabricate ceramic oxide coatings on 2024 aluminum alloy.

EXPERIMENTAL WORK

Preparation of Al alloy Substrates

2024AA aluminum alloys specimens with (4x ϕ 25)mm were cut from 2024 aluminum alloy shaft, the microhardness of 2024 aluminum alloy substrate was 157HV and its chemical composition was done in Ministry of Industry and Minerals (State Company for Inspection and Engineering Rehabilitation) as shown in (Table 1). The surface of the coated component should features most important properties, like cleanliness (to free the surfaces from organic contamination , products of chemical reactions, and particles of mechanical contamination) and smoothness surface[S. H. Awad, 2005]. Before to coating method the Al alloy specimens were polished with silicon carbide paper SiC with (400 to 2000)# grit emery, to obtaining surface roughness approximately 0.896 μ m, cleaned with detergent like acetone, then washed with distilled water, to be utilized for MAO method.

Powders preparation

Rice husks ash powder was equipped from rice husks, first an appropriate quantity of rice husks were cleaned ideally with distilled water to eliminate, impurities, and dust. They were desiccating at the ambience and, burning at temperature 700°C for 8hr using the firing furnace. Grinding for 12hours, after grinding the powders were evaluated by particle size analyzer (Type Better Size 2000, Ceramic and Building Materials Department/ College of Materials Engineering / Babylon University); the suspension was prepared by taking little amount of powder and mixed them with 500 ml of distilled water, then the sample mixing by magnetic stirring. Finally, the sample will be ready for the test. The mean particle size was approximately (8 μ m), and the chemical composition was (90.42% SiO₂, 0.50% Al₂O₃, 2.15% CaO, 0.44% SO₃, 0.22% P₂O₅). The TiO₂ nano powder was with particle size of (10nm).

Electrolytes selection

Numerous efforts were carried out in utilizing various composition of the MAO electrolytes consisting rice husks ash as exporter for silica additives, and TiO₂ nano additives and other additives for PH adjustment (H₃PO₄), property modification (KH₂PO₄), performance modification ((NH₃)₂Mo₇O₂₄), electrolyte conductivity increasing (KOH), and assistant additive (Na₂CO₃). Then, the selection of the appropriate electrolytes, and their compositions was done according to the results from the MAO experiments like : common observation, breakdown voltages and production spark related current and voltage, controlling the electrolyte temperature, coatings production, and their uniformity. (Table2) shows the major electrolytic solutions utilized in the MAO method.

MAO process

Deposition of the ceramic coating was done by a home-made (domestic) MAO unit shown in (Fig.1) with voltage of (0-500V), and current of (0-2A). The unit consisted of high voltage DC-AC power supply, a plastic case for the electrolyte prepared with the cathode holder and a sample holder anode, mixing regime, and cooling regime. The positive output of the power supply was connected to the sample immersed in the electrolyte serving as the working electrode anode and the negative output was connected to the stainless steel 316L plate acting as the electrode cathode. During the method electrolytic solution was mixed and cooled to prevent heating over to 25°C, the cooling method was done by a cooling unit connected to the MAO unit, this cooling unit, was prepared with a distilled water in plastic case surrounded the electrolyte plastic case. The deposition process was controlled to form an ceramic film 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 appropriate electrolyte components which mixed in liter of distilled water, then mixed by magnetic stirrer for 3hours before the deposition method, PH of electrolyte solution(7-8).

Apparatuses and characterization

Phase composition of the coatings were tested utilizing an X-ray diffractometer (XRD-6000 SHIMADZU, Japan, Cu $K\alpha$ radiation, 40KV, 30 MA, 6°/min scanning speed, in Ceramic and Building Materials Department/ College of Materials Engineering/ University of Babylon). The observations were complimented with an energy dispersive spectroscopy (EDS, QUANTA 450, in College of Pharmacy Faculty/ Babylon University) for analysis of elemental distributions across the surface. The microstructure of coatings were observed utilizing scanning electron microscope (SEM,TESCAN/ VEGA II Series/ USA, in Al-Razi Institute Tehran, Iran). The micro-hardness of the coatings were studied utilization a Vickers indenter (HVS-1000, Laryee, digital Micro-hardness tester, in the University of Technology) with 9.8N load. The thickness of coatings was measured using (Microprocessor CM-8822, coating thickness meter, (in Department of Chemical Engineering/ Engineering College/ University of Babylon). The roughness of coatings was tested utilizing(SRT-6210,surface roughness test, in the (Department of Chemical Engineering/ Engineering College/ University of Babylon). The wear resistance and coefficient friction of coated samples was studied with(microtest-28021, in Department of Metallic Engineering /College of Materials Engineering/Babylon University).

RESULTS AND DISCUSSION

MAO spark conditions

(Table3)shows the current-voltage values could prove the potential of using natural and nano additives containing electrolytes in the work in production of ceramic oxide coatings by utilizing of moderate current and voltages in the range (0.2-0.4)A and (360-364)V, respectively. It can be noted that, spark discharge could be appeared above 200Vin the electrolyte solution.

XRD and EDS results of samples

(Fig.2), shows the XRD results of the ceramic oxide coatings prepared in nano and natural containing electrolytes particularly sample A₄. Also, (Fig.3), and (Table4) show the EDS results of sample A₄.XRD analysis of MAO layer showed the aluminum peaks (JCPDS No. 004-0787) arriver from the underlying substrate were ditto discovered due to the penetration of X-rays into the Al substrate, γ - Al_2O_3 (JCPDS No. 010-0425) and α - Al_2O_3 (JCPDS No. 046-1212), and the peak intensity at 46° belongs to TiO_2 , which pinpointed that some nano-particles have entered into the equipped ceramic oxide coatings. As well known, continuous and long time discharge results in a elevated temperature in the discharge channels, which is propitious to the deposition of TiO_2 nano-particles. So TiO_2 peaks at 46° can be detected in the coating. Anyhow, titanium oxide compound was not produced during the method since no corresponding diffraction peaks were found this

behavior agreement with [H. xia Li et. al., 2013]. Results of EDS showed the existence of Al and O element in the coatings, thereat, betokened to the production of alumina Al_2O_3 ceramic layers modified with another compounds of Mo, P,C, and Si at various weight of these modification compounds.

SEM Results

(Figs. From 4 to 6) show the SEM results for the surface morphology of the coated samples A_3 , A_7 , and A_{11} at various magnifications. Increasing the nano- TiO_2 addition from 1g of A_1 to 3g of A_{11} resulted in more micro cracks, tunnels, and more porosity with large sizes. The surface of the coated samples A_3 , A_7 , A_{11} had a exemplary morphology, identified by existence of large, connected pores and micro crack existence in the structure of A_3 coated samples, A_7 sample identified by less pores and micro cracks, but A_{11} identified by more tunnels, pores with micro cracks, the micro pores are the residual discharge channels during the plasma spark reaction, but the micro cracks were caused by the thermal stress due to the rapid solidification of molten oxide in the cool electrolyte [X. Wang et.al., 2013]. For the composite oxide coatings, nano- TiO_2 could embed into the micro-arc discharge channels through the diffusion by the incorporation of nano- TiO_2 additive in the electrolyte during MAO. When the TiO_2 nano powders were added into the electrolyte, they were homogeneously distributed under vigorous stirring. During MAO, when the molten Al_2O_3 erupted from the discharge channels, nano- TiO_2 particles would be mixed within the Al_2O_3 coating. Its thought that more and more dispersed nano particles entered into the pores with increasing the nano-additive concentrations, so the coating surface became denser and smoother; by this method, high quality coatings with high micro-hardness, and excellent wear resistance could be synthesized [H. xia Li et. al., 2013].

Thickness, and Roughness Results

Table 5 shows the resulted coatings thickness and roughness after various deposition times. In the present study thickness of coatings heterogeneous with deposition time due to breaking of the weak oxide film during growth by strong spark, that give various value of thickness, it can be showed that the thickness values were (39-67) μm , and the sample A_2 showed the highest thickness among the others (67 μm at 30 min). There were a clear increase in the coatings thickness with increasing deposition time, the coatings become uniform and more thick with few porosity [N. Yu. Dudareva et. al., 2014]. Such behavior is dependent on the electrolyte components, resistance and PH. The production number of oxide is comparatively increased in a unit time, enhancing the growth rate of ceramic coating, while when the thickness increased to a particular limit, the cathodic voltage restricted to break down the coatings [S.Chen et. al., 2016].

It can be observed that the deposited ceramic oxide films displayed surface roughness in the range (1.908-3.358) μm . The samples A_1 and A_4 recorded the lowest and the highest roughness, respectively, among the others. Generally, the coating roughness increased with deposition time increasing. The ceramic films produced in the electrolyte with addition of nano particles has rougher surface, this is because the electrolyte composition was adsorption on ceramic film surface, particularly the dispersant adjusted nano-particles are electronegative, has trend of accretion on anode surface. The uneven distribution of the

adsorption film is the major reason of the increasing value of roughness [S. Xiangbo et. al.,2014].

Micro-hardness Results

Table 6, and Fig. 7 show the micro-hardness results at load of (9.81N) and holding time (15sec) of the coated samples. Commonly during the test, utilizing loads of (100, 200, 300, 400, 500)g could not give any track in the coated sample surface. The hardness of film 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 oxide coatings, so the hardness is rather low in this region [K. Wang et. al., 2009]. It can be observed that the hardness of coatings in the range (313-531.3)HV. The sample A_{10} at deposition time 30min recorded the highest hardness 531.5HV among the others because of incorporation of nano- TiO_2 additives 3g in the electrolyte which could enhance the structure of ceramic films with uniform distribution of pores and high alumina content from EDS analyses.

Wear Results

Table 7, Fig. 8, and Fig. 9 show the wear resistance for aluminum alloy substrate. (Table 8), and (Figs. from 10, to 13) show the wear results for the A_1 to A_4 samples. Table 9, and Fig. 14, and Fig.15 show the wear results for the A_5 and A_8 samples. Generically, the results showed that with loading time increased, the weight losses increasing. In comparison, with 2024 AA substrate, the coated samples could evince the good wear resistance of the ceramic oxide films which yielded in less weigh losses. Moreover, ceramic oxide films produced utilizing the nano- TiO_2 consisting electrolytes gave the best wear resistance (0.00003-0.0009) for (A_1 - A_4), due to improved hardness. For the friction coefficient of the (A_1 - A_4) samples was in the range (0.39-0.67), it can be seen that the friction coefficient of coatings increasing with weight losses decreasing.

CONCLUSION

1-It is possible to deposit hard, dense and antiwear ceramic oxide coatings on Al alloy substrates via (MAO) method utilizing home-made unit and nano additives in the electrolyte like nano- TiO_2 additives and silica natural additives extracted from rice husks ash.

2- XRD results and EDS analyses evidenced the deposition of alumina (Al_2O_3) ceramic with different contents and other compounds by (MAO) utilizing, the electrolytes utilized in this study at controlled temperature.

3- MAO ceramic oxide coatings with thicknesses (39-67) μm could improve the Al alloy substrate hardness from (157HV) to (313-531.311)HV of (3g TiO_2). Using of TiO_2 additives in the MAO electrolyte provided the highest hardness and coating thickness.

4- The coatings roughness increased with the deposition time due to thickness increasing.

5- The MAO ceramic layers could improve the wear resistance of the Al alloy substrates. At 60min deposition time, the additions of TiO_2 reduced weight loss and increased friction coefficient.

Table (1): Chemical composition of 2024AA aluminum alloy.

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

Table (2): Compositions of nano-TiO₂ , basic materials and the RHA containing electrolytes .

Electrolyte composition	Sample code	Deposition time min
(1g/l) nanoTiO ₂ , (10g/l) KH ₂ PO ₄ , (3g/l) RHA, (4g/l) H ₃ PO ₄ , (5g/l) NaCO ₃ , (6g/l) (NH ₃) ₂ MO ₇ O ₂₄ , (9g/l) KOH,	A ₁	15
	A ₂	30
	A ₃	45
	A ₄	60
(2g/l) nanoTiO ₂ , (10g/l)KH ₂ PO ₄ , (3g/l) RHA, (4g/l) H ₃ PO ₄ , (5g/l) NaCO ₃ ,(6g/l) (NH ₃) ₂ MO ₇ O ₂₄ , (9g/l) KOH	A ₅	15
	A ₆	30
	A ₇	45
	A ₈	60

(3g/l) nanoTiO ₂ , (10g/l)KH ₂ PO ₄ , (3g/l)RHA, (4g/l) H ₃ PO ₄ , (5g/l) NaCO ₃ ,(6g/l) (NH ₃) ₂ MO ₇ O ₂₄ , (9g/l) KOH	A ₉	15
	A ₁₀	30
	A ₁₁	45
	A ₁₂	60

Table (3): MAO spark condition using TiO₂ and basic materials and RHA containing electrolyte.

Samples	Current (A)	Voltage (V)
A ₁	0.4	360
A ₂	0.3	364
A ₃	0.2	361
A ₄	0.3	363
A ₅	0.3	363
A ₆	0.3	364
A ₇	0.4	360
A ₈	0.2	362
A ₉	0.3	363
A ₁₀	0.2	362
A ₁₁	0.3	364
A ₁₂	0.3	363

Table (4): EDS results of coated sample A₄.

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	46.41	39.20	40.06	5.29
C	6	K-series	37.34	31.54	42.93	4.57
Al	13	K-series	28.01	23.66	14.34	1.36
Si	14	K-series	2.86	2.42	1.41	0.15
P	15	K-series	2.40	2.02	1.07	0.12
Mo	42	L-series	1.37	1.16	0.20	0.08
Total:			118.39	100.00	100.00	

Table (5): Thickness and roughness result of coatings.

Sample	Deposition time (min)	Thickness (μm) at the center	Roughness Ra(μm)
A ₁	15	39	1.908
A ₂	30	67	2.877
A ₃	45	64	3.307
A ₄	60	64	3.358

Table(6): Micro-hardness (HV) results of coated samples.

Sample	Deposition time (min)	Micro-hardness (HV)
A ₁	15	393.7
A ₂	30	486.8
A ₃	45	516.5
A ₄	60	466.7
A ₅	15	328.2
A ₆	30	333.6
A ₇	45	410.8
A ₈	60	413.6
A ₉	15	400.2
A ₁₀	30	531.3
A ₁₁	45	313
A ₁₂	60	456.5

Table(7): Loss weight of Al alloy substrate .

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

Table(8): Loss weight of coated samples (A₁-A₄) .

Sample	Deposition time(min)	Test time(min)	Loss weight(g)	μ
A ₁	15	15	0.0001	~0.39
			0.0002	
			0.0003	
A ₂	30	15	0.0005	~0.67
			0.0009	
			0.0014	
A ₃	45	15	0.0003	~0.63
			0.0006	
			0.0009	
A ₄	60	15	0.0001	~0.45
			0.0005	
			0.0006	

(Table 9): Loss weight of coated samples (A₅, and A₈).

Sample	Deposition time (min)	Test time(min)	Loss weight(g)	μ
A ₅	15	15	0.0002	~0.77
			0.0003	
			0.0006	
A ₈	60	15	0.0004	~0.45
			0.0007	
			0.0010	

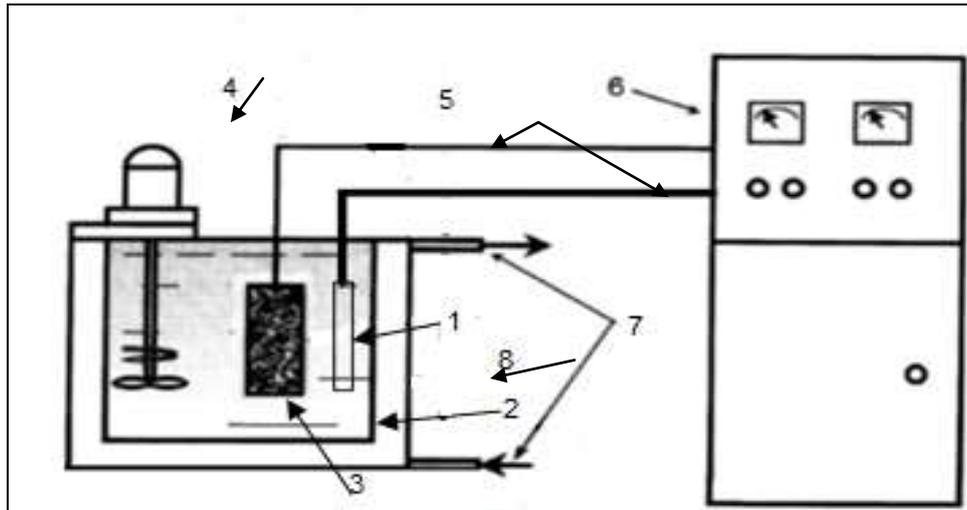


Fig. (1): Prototypical configuration of the equipment utilized for MAO coating: (1) stainless steel 316L plate cathode, (2) plastic case of coating electrolyte, (3) sample anode, (4) mixer, (5) connecting wires, (6) power supply unit, (7), flow circulation via cooling regime (8) plastic case of cooling bath after disposition[S. H. Awad, 2012].

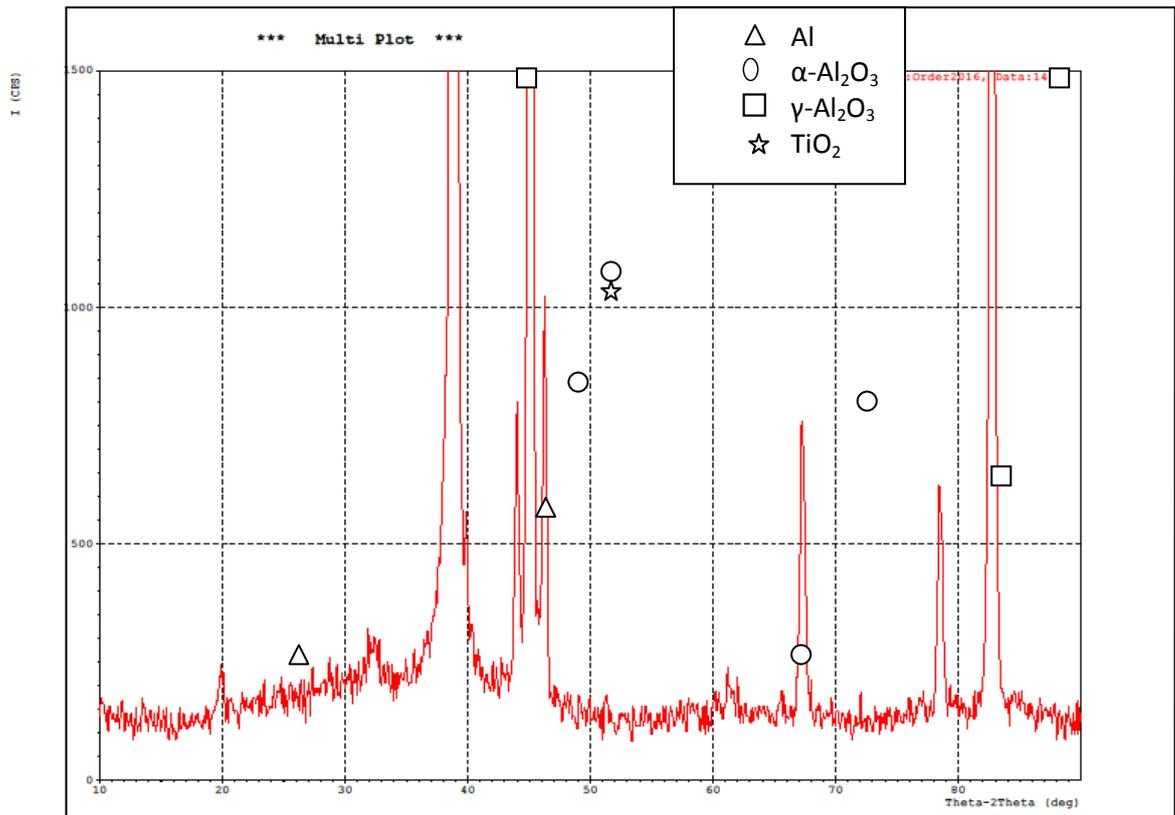
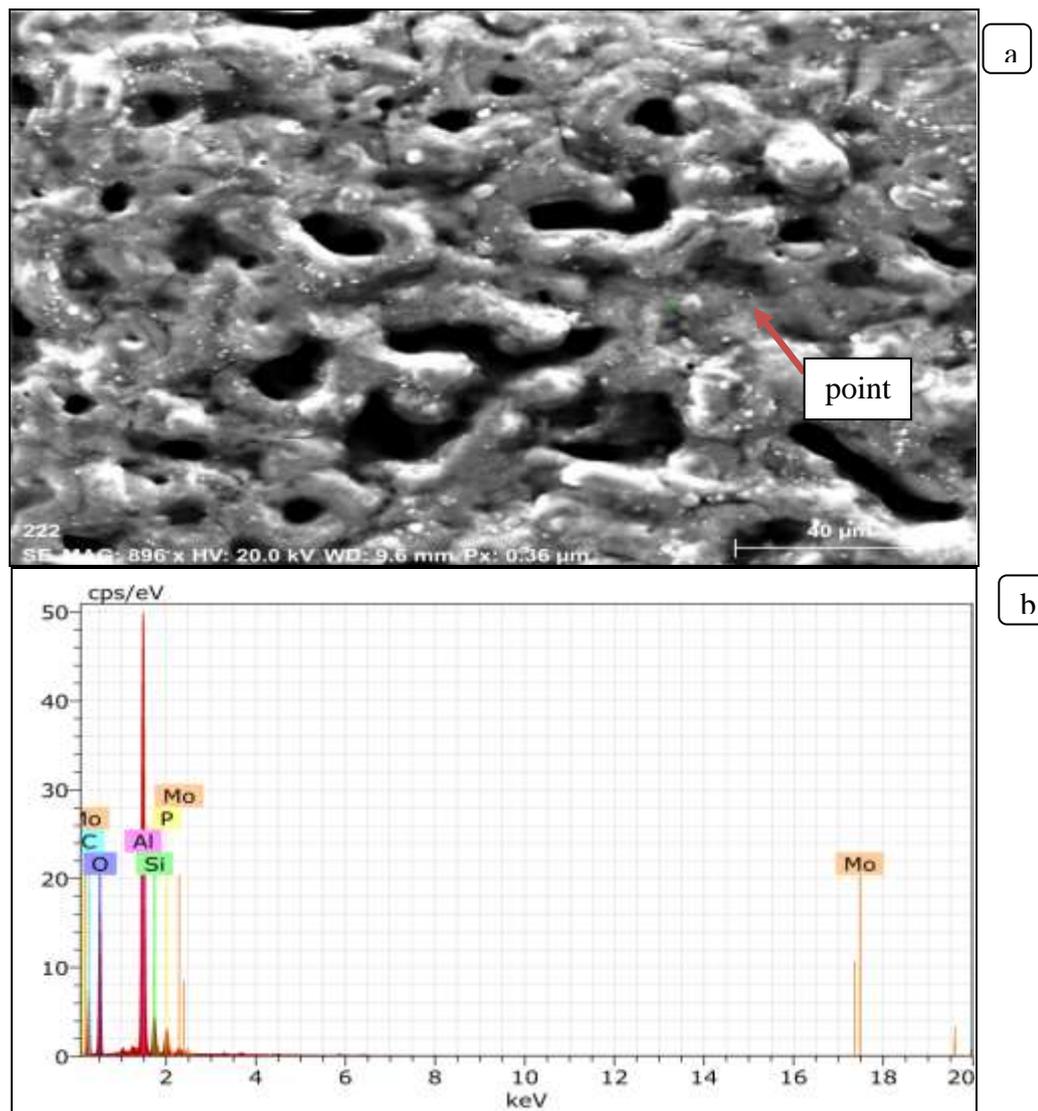


Fig. (2): XRD chart of coated sample A4.



**Fig. (3): EDS results of coated sample A₄:
(a) surface morphology, (b) EDS results of the point.**

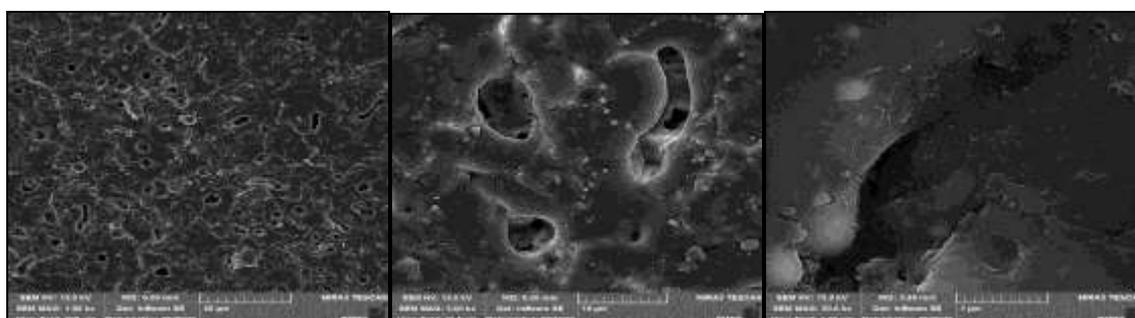


Fig. (4): SEM for surface morphology of the sample A₃ for deposition time (45min), at various magnifications (50, 10, 1) μm, respectively.

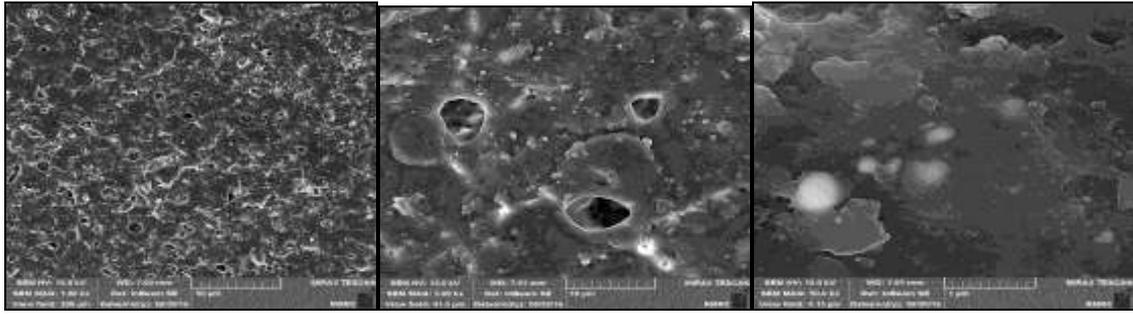


Fig. (5): SEM for surface morphology of the sample A₇ for deposition time (45min), at various magnification (50, 10, 1) μm, respectively.

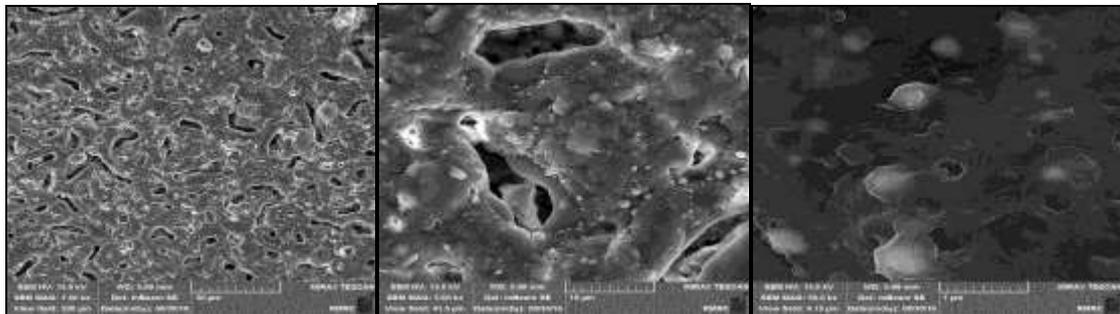
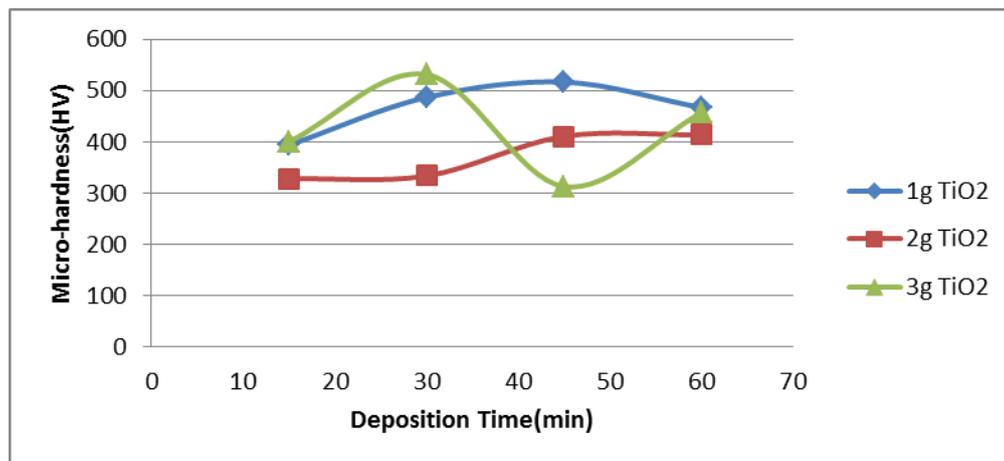


Fig. (6): SEM for surface morphology of the sample A₁₁ for deposition time (45min), at various magnifications (50, 10, 1) μm, respectively.



(Fig. 7): Relationship between the micro-hardness and deposition time of coated samples with nano TiO₂ additives.

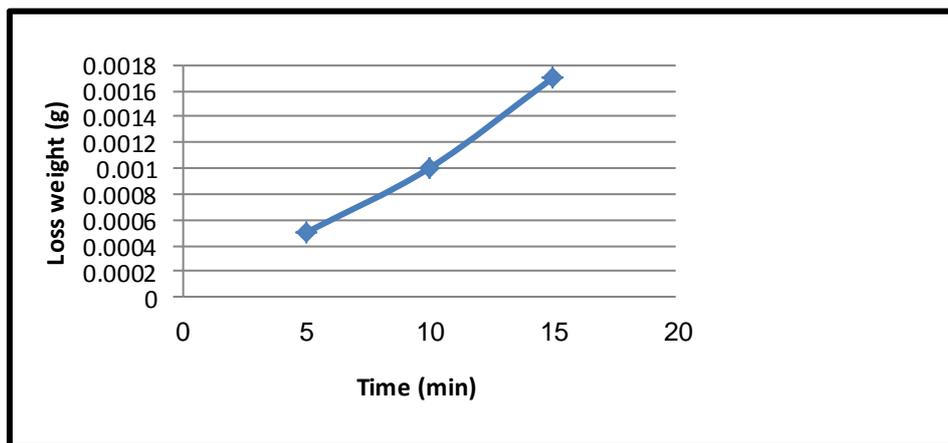


Fig.(8): Relationship between loss weight and test time for Al alloy substrate.

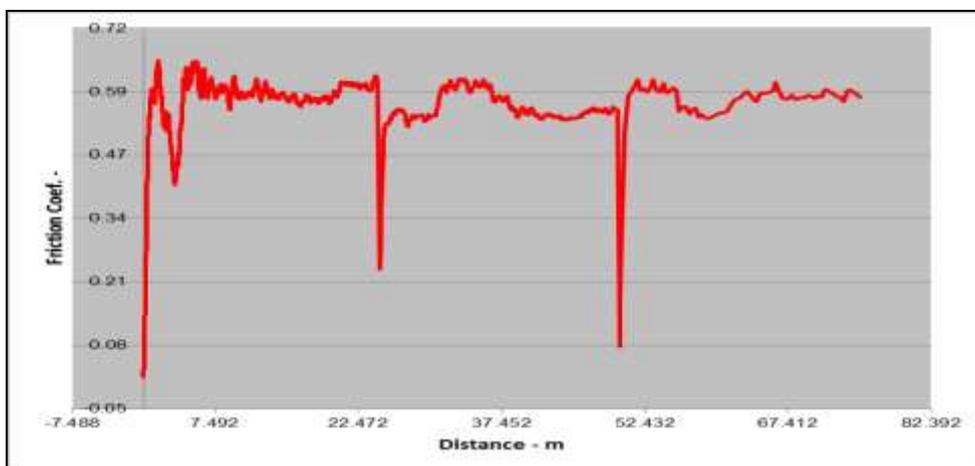


Fig.(9): Friction coefficient results for Al alloy substrate.

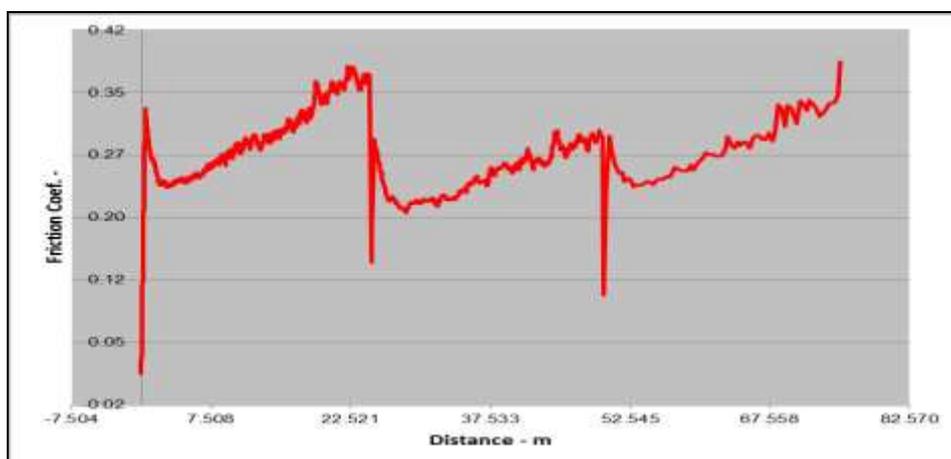


Fig.(10): Friction coefficient results for A_1 at deposition time (15min).

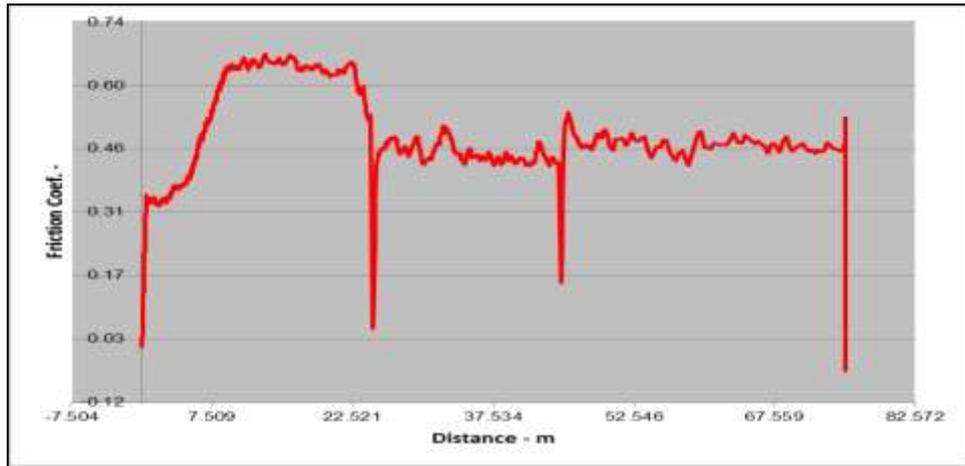


Fig.(11): Friction coefficient results for A_2 at deposition time (30min).

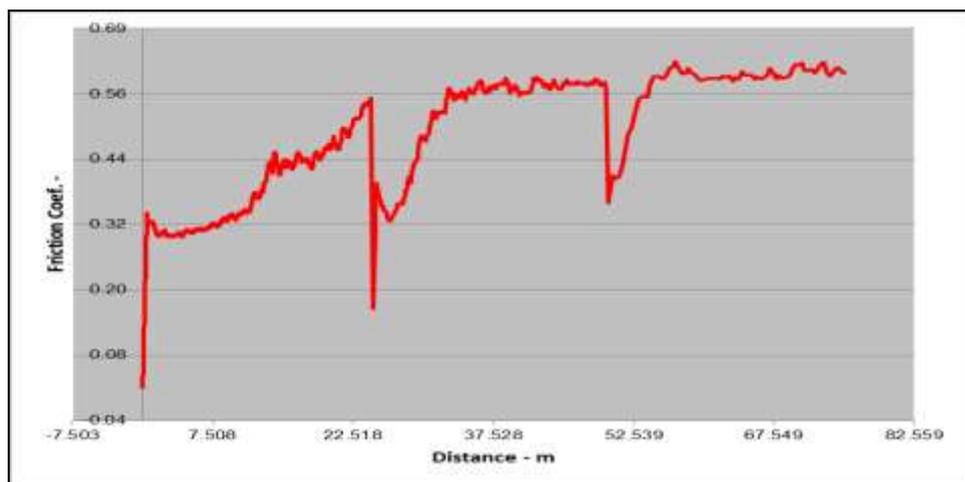


Fig.(12): Friction coefficient results for A_3 at deposition time (45min).

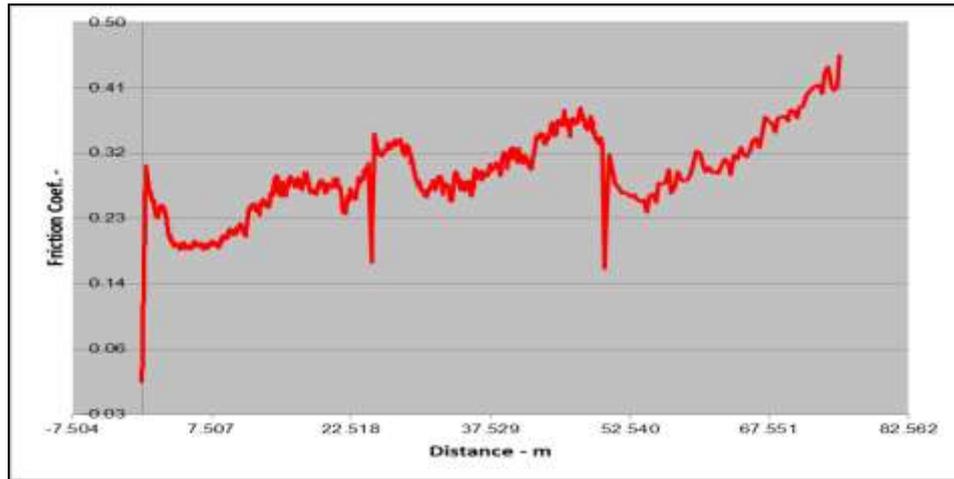


Fig. (13): Friction coefficient results for A₄ at deposition time (60min).

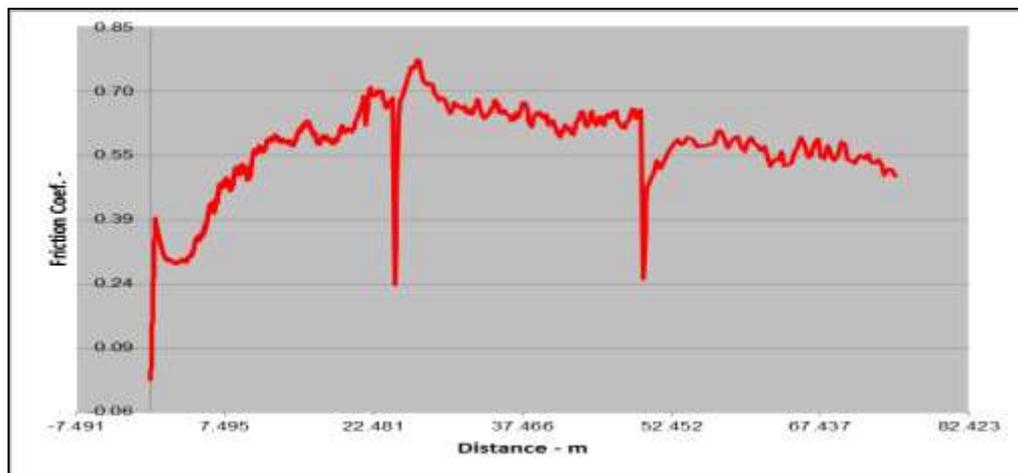


Fig.(14): Friction coefficient results for A₅ at deposition time (15min) .

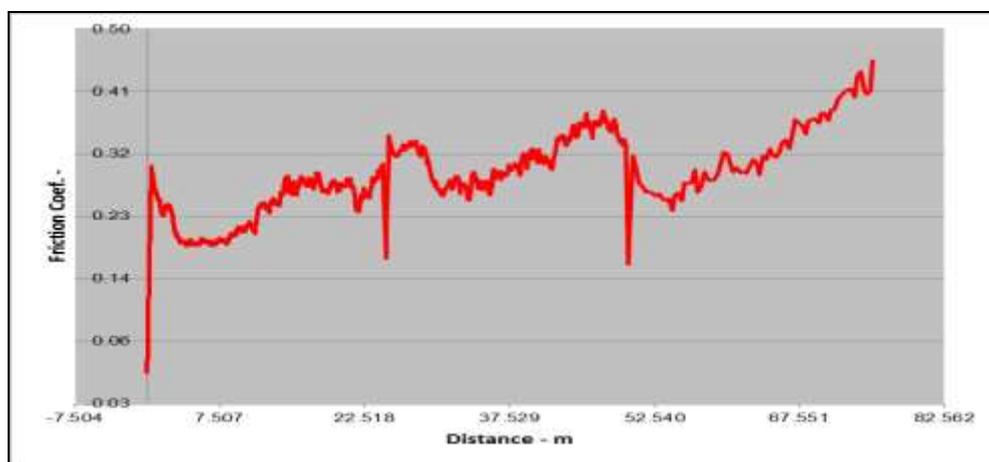


Fig.(15): Friction coefficient results for A₈ at deposition time (60min) .

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