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# Effect of Micro Arc Oxidation Coatings on the Properties of Aluminium Alloys

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Additional information is available at the end of the chapter

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## 1. Introduction

Aluminium and its alloys are one of the most useful substances in industries such as aerospace industry where the demand for lighter, more durable, recyclable and replaceable materials is increasing because of economical and technical considerations. Their good corrosion resistance made them more important in use as a component of internal combustion engines for instance cylinder blocks cylinder heads and pistons [1].

By means of MAO process, which is also known as plasma electrolytic oxidation, spark anodisation or micro-arc discharge oxidation, with the combination of electrochemical oxidation and high voltage spark treatment, the engineering equipments which have excellent adhesive, strength, friction, corrosion, wear, electrical and thermal properties are obtained.

The micro-arc oxidation (MAO) process has been examined on dense metals in a wide range of structures [2], but its application to metallic foams has not been systematically investigated. PEO is a plasma-assisted electrochemical surface treatment that is used to convert surfaces of light metals, e.g. Al, Mg and Ti, into hard and well-adhered oxide layers [3]. The crucial problem in using these components are aluminium component's poor surface hardness and wear resistance which limits its use in applications and reducing the lifetime of the substance.

Previous treatments on aluminium such as anodising and thermal spraying have suffered from the low load support from the underlying material and insufficient adhesion. Also some coated aluminium alloys can achieve high surface hardness but when it comes to practice they often show poor performance under mechanical loading [4]. Furthermore, AA2014 alloy consists mainly of copper, magnesium, manganese, silicon and some other elements which make it usable for conditions where strength hardness and good workability is need-

ed. If we define workability as the amount of deformation that a material can undergo without cracking or reaching undesirable conditions at a given temperature and strain rate, improving workability means to undo these undesirable conditions and increasing the processing ability of the materials and increasing the productivity. This situation can be achieved by optimum homogenization treatment [5].

PEO coatings on aluminium are characterised by complex mixtures of amorphous material and crystalline phases and consist mainly of two regions [6]: a dense and hard inner region which has increased  $\alpha\text{-Al}_2\text{O}_3$  content and a porous outer region containing more  $\gamma\text{-Al}_2\text{O}_3$ , plus alumina-silicates and amorphous phases that incorporate some electrolyte species (e.g. Si).

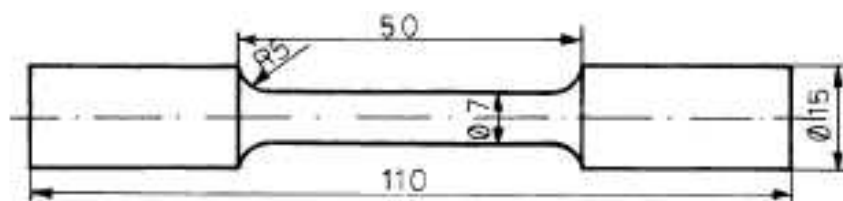
This process combines electrochemical oxidation with a high voltage spark treatment in aqueous electrolytic bath which also contains modifying elements in the form of dissolved salts to be incorporated into resulting alumina coatings which are thick hard oxide ceramic coatings that are fabricated on aluminium alloys [7]. However, ceramic coatings were formed on a pure aluminium substrate in an aqueous solution of phosphate salt, using the micro-arc oxidation method. The result indicated that elements from the electrolyte like P could be incorporated into the film during MAO process [8].

In Aluminium and its alloys, depending on the production method and alloy, by means of some thermal processes and applications, important differences can be achieved on the tribological and surface properties of aluminium alloys by the heat treatment furnaces [9]. With particular attention, providing and controlling the necessity of heating, post phone, cooling process play a crucial role in the hardness and mechanical properties of aluminium and its alloys [10].

In this study, the authors investigated the best properties of AA2014 aluminium components and minimizing the undesirable conditions that occur in components by following the method of homogenization and micro-arc discharge oxidation process along the specimens.

## 2. Experimental details

Before coating and homogenization, the specimens are machined to fatigue test specimens and the dimensions of the specimens are given in Figure 1.



**Figure 1.** The shape of aluminium specimens for fatigue tests (AA2014)

The chemical composition of the AA2014 aluminium alloy used as specimens is given in the Table 1.

Cu (%)	Si (%)	Mg (%)	Mn (%)	Fe (%)	Cr (%)	Sn (%)
4.821	0.816	0.612	0.572	0.221	0.057	0.041

**Table 1.** The chemical composition of the AA2014 aluminum alloy

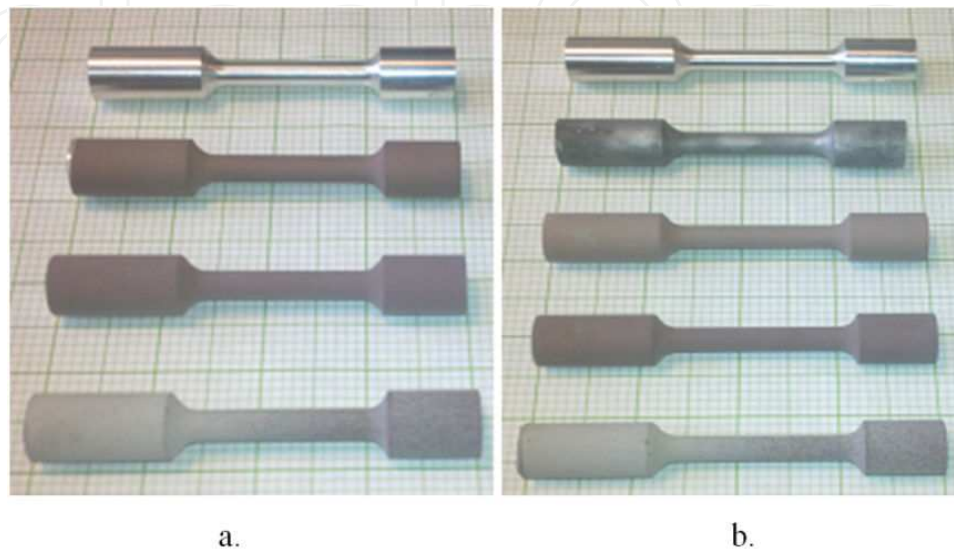
Before coating process, some of the specimens undergo homogenization heat treatment at 524 °C for 6 hours. The specimens are cooled on air after the treatment. The properties and the drawing of the furnace are shown in Figure 2.



**Figure 2.** The Photograph of the furnace that is used for homogenization treatment.

Coatings were formed by the use of micro arc oxidation technique where a voltage pulsed at mains frequency 50 Hz(hertz) was selected in the range 50–500 V(volts) and a predefined current density at the coating surface was maintained by control of the voltage pulses during the process. The electrolyte that is used in coating consists of sodium silicate in distilled

water with other additives [11]. The electrolyte temperature is controlled to remain under  $75^{\circ}\text{C}$  during treatment. 3 treatment times were chosen to produce total coating thicknesses of 20, 60 and 120  $\mu\text{m}$ (micronmeters) respectively as shown in Figures 3.a. and 3.b.



**Figure 3.** Non-homogenized specimens (non-coated, 20, 60, 120  $\mu\text{m}$ (micronmeters) coated respectively), homogenized specimens (non-coated, only homogenised, 20, 60, 120  $\mu\text{m}$ (micronmeters) coated respectively)

The electrolytic solution consists of distilled water with potassium hydroxide and sodium silicate additives. The tank is filled with 200 litres of distilled water with 12 grams of sodium silicate and 2 grams of potassium hydroxide per litre. The required amount of sodium silicate and potassium hydroxide are dissolved in another tank with a stirring device before addition process. The used sodium silicate was in solid state and had 5 water molecules in their formula that is why in total, 4171 grams of sodium silicate was used in process. The Ph of the solution is measured as 12,4.

The MAO process was made with 200 $\mu\text{f}$ (micronfarads) interference because of the geometry of the samples. The voltage values in anode and cathode were measured in every 5 minutes during coating process. The current was also measured. The tables and graphs regarding to coating voltages are given in results section.

After the coating process, the substrates were polished with Si-C 800 mesh abrasive papers of decreasing grit size. A scanning electron microscope (Apollo 300) (SEM) was employed for observation of the microstructure and surface and fracture cross – section morphology of the coatings before and after the tests. The crystallographic characteristics of the coatings were investigated using an X-ray diffractometer (Philips PANalytical) ( $\text{CuK}\alpha$  radiation) with the film attachment, at grazing angles of  $0.5^{\circ}$  and  $2.0^{\circ}$ . The X-ray generator settings were 60 kV(kilovolts) and 55 mA(milliamperes), the step size  $0.02^{\circ}$ , step time 10 sec(seconds) and the scans were acquired from  $20$  to  $90^{\circ}$  (in  $2\Theta$ ). The fatigue tests were performed on a 50

kN(kilonewtons) capacity of servo-hydraulic material testing machine (Instron 8051) having a frequency range of 0.5-100 Hz(hertz). [13].

### 3. Results and discussion

The specimens that are machined to fatigue test specimens are put into the furnace pre heated to 524°C and after the specimens are put into the furnace, the temperature is remained at 524°C [13]. The specimens are treated in the furnace for 6 hours and after that, they are cooled in air.

Regarding to the coating process, the current-voltage characteristics and the coating thicknesses of the specimens are given in Table 2 and Figures 4, 5 and 6.

normal specimens 20 µm(micronmeters) coated				homojenized specimens 20 µm(micronmeters) coated			
Time (minute)	kathode voltage V(voltage)	anode voltage V(voltage)	Current A(ampere)	Time (minute)	kathode voltage V(voltage)	anode voltage V(voltage)	Current A(ampere)
0	80	400	8,10	0	300	150	11,75
5	75	490	9,18	5	60	440	9,81
10	80	495	8,92	10	70	460	9,15
15	80	495	8,93	15	70	460	9,04

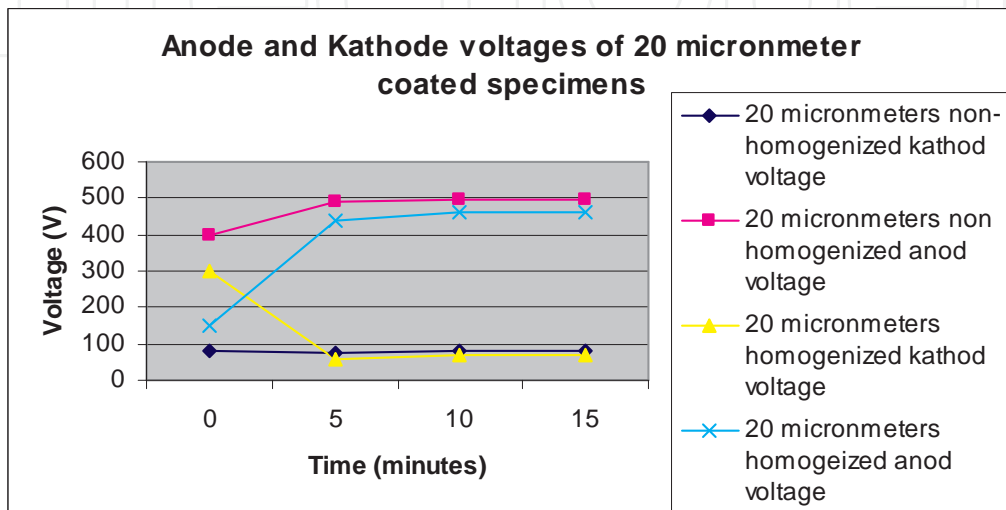
normal specimens 60 µm(micronmeters) coated				homojenized specimens 60 µm(micronmeters) coated			
Time (minute)	kathode voltage V(voltage)	anode voltage V(voltage)	Current A(ampere)	Time (minute)	kathode voltage V(voltage)	anode voltage V(voltage)	Current A(ampere)
0	40	200	11,88	0	160	100	11,90
5	60	445	9,54	5	62	450	9,58
10	65	455	9,28	10	65	460	9,32
15	70	475	9,01	15	70	475	9,29
20	80	480	8,79	20	80	490	9,12
25	80	500	8,83	25	80	500	9,08
30	80	500	8,64	30	80	500	9,01
35	85	500	8,58	35	82	500	8,92
40	90	505	8,38	40	85	502	8,78
45	100	505	8,27	45	92	505	8,65
50	105	505	8,25	50	100	505	8,36

normal specimens 120 μm(micronmeters) coated				homojenized specimens 120 μm(micronmeters) coated			
Time (minute)	kathode voltage V(voltage)	anode voltage V(voltage)	Current A(ampere)	Time (minute)	kathode voltage V(voltage)	anode voltage V(voltage)	Current A(ampere)
0	180	120	12,14	0	40	150	12,05
5	62	448	9,72	5	60	445	9,61
10	65	455	9,49	10	62	452	9,43
15	75	490	9,36	15	70	475	9,19
20	80	498	9,07	20	78	495	8,96
25	80	500	8,96	25	80	500	8,98
30	80	500	8,89	30	80	500	8,92
35	80	500	8,80	35	82	502	8,75
40	90	505	8,83	40	90	505	8,71
45	100	505	8,55	45	100	505	8,59
50	102	505	8,42	50	112	505	8,38
55	120	505	8,10	55	130	505	8,18
60	140	515	8,06	60	140	515	8,22
65	145	525	8,03	65	140	525	8,19
70	155	525	8,13	70	145	530	8,22
75	160	540	7,97	75	160	525	8,28
80	160	540	8,15	80	170	530	8,22
85	160	540	7,89	85	180	550	8,20
90	180	550	7,85	90	160	525	8,34
95	182	550	7,79	95	220	530	8,13
100	220	550	7,59	100	180	530	8,18
103	210	555	7,81	103	190	540	8,04

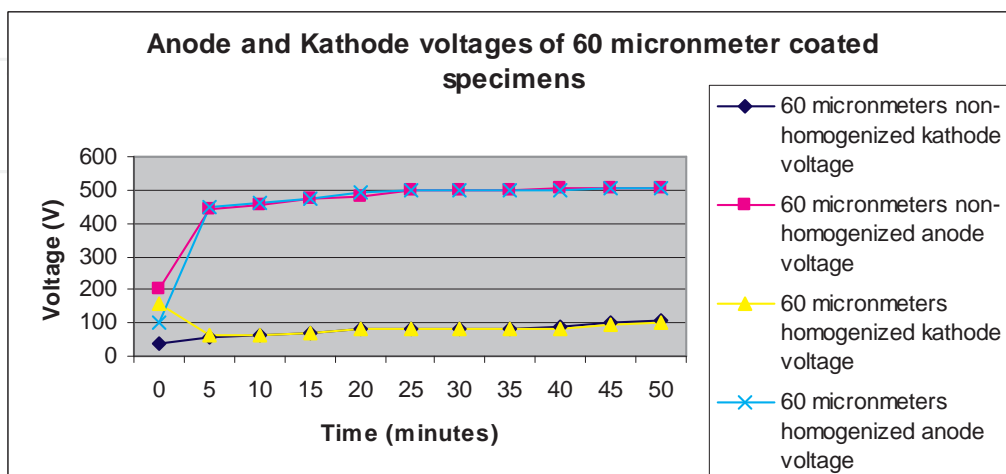
Table 2. Voltage characteristics of coated specimens

According to diagram it is observed that both homogenized and non- homogenized specimens have standart values of voltages in different coating thicknesses, from these results, it is noted that the homogenization treatment has nearly no effect on the voltage values during operation. The coating thicknesses are measured by Mitutoyo device used to measure coating thickness and it is seen that at the same coating time interval the coating thicknesses are

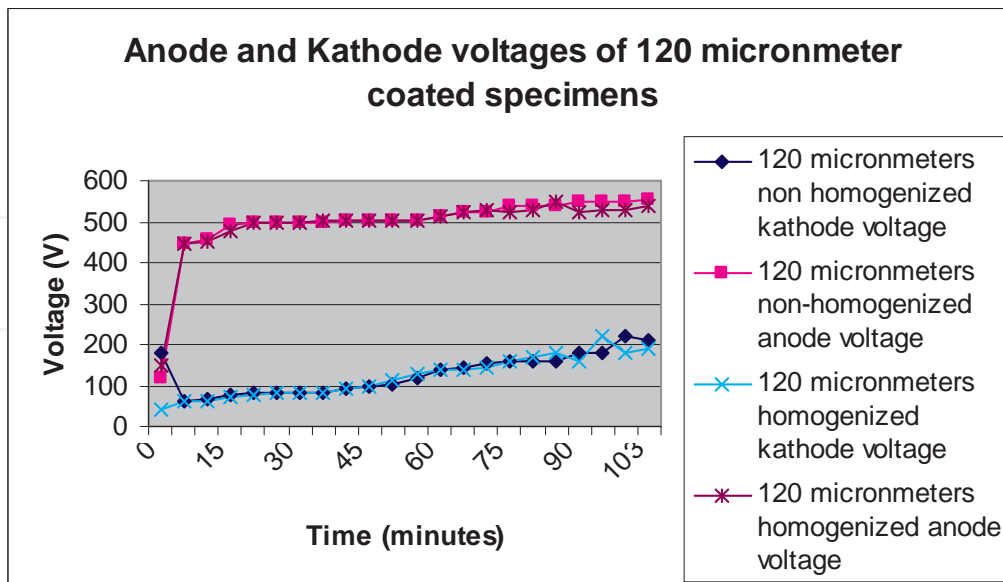
seem to be the same. After the cooling operation and coating non- homogenized, homogenized, coated and both heat treated and coated specimens are put into fatigue tests.



**Figure 4.** Anode and Kathode voltage graphs of 20 micronmeter coated specimens



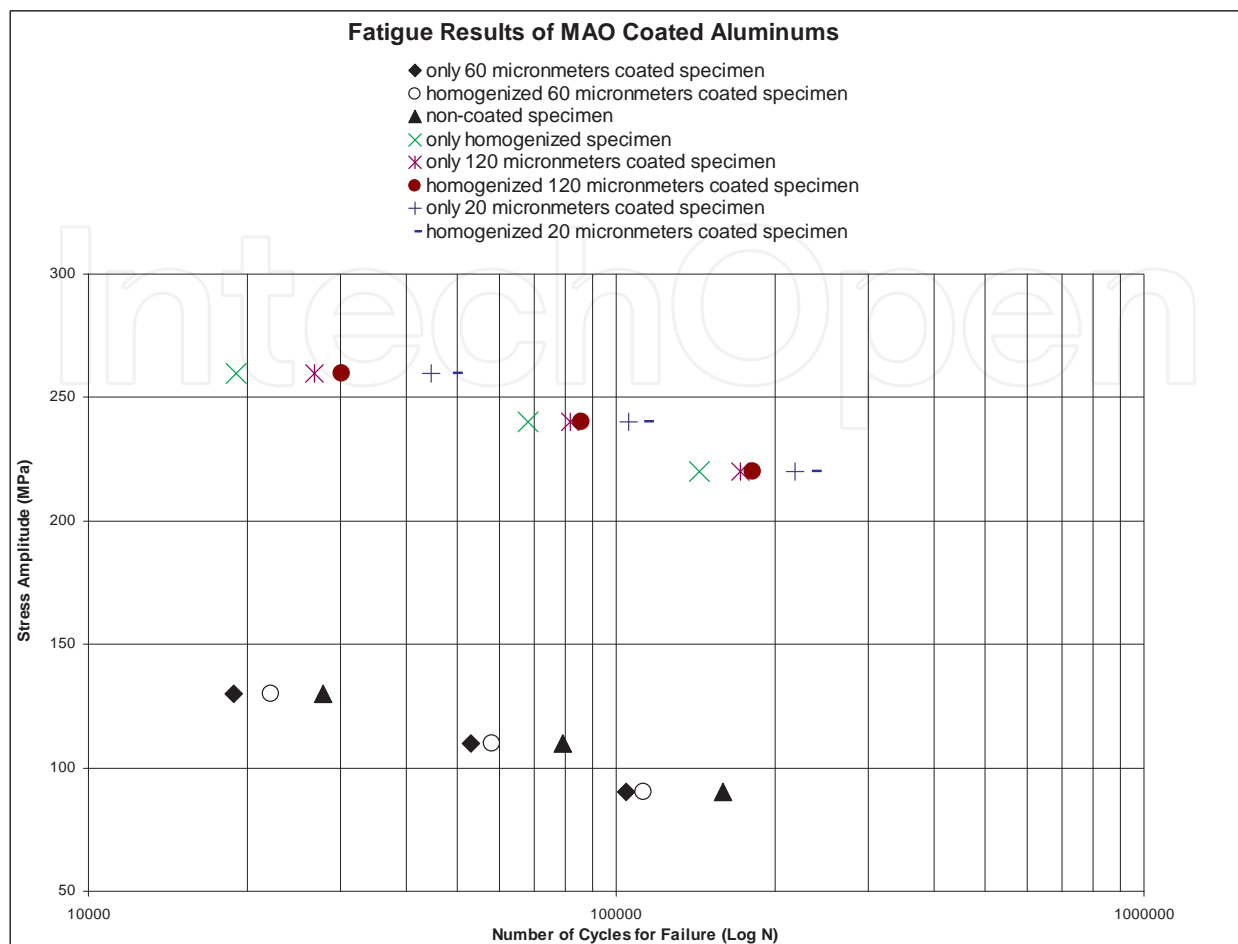
**Figure 5.** Anode and Kathode voltage graphs of 60 micronmeter coated specimens



**Figure 6.** Anode and Kathode voltage graphs of 120 micronmeter coated specimens

The fatigue tests are taken into action in 8 groups. Non- homogenized and non- coated samples, only homogenized samples, non- homogenized but coated samples (20 $\mu$ m(micronmeters), 60 $\mu$ m(micronmeters) and 120 $\mu$ m(micronmeters) respectively) and homogenized and coated samples 20 $\mu$ m(micronmeters), 60 $\mu$ m(micronmeters) and 120 $\mu$ m(micronmeters) respectively). After several fatigue tests on these specimens we can find these results;

1. Homogenized and 20 $\mu$ m(micronmeters) coated specimens have the best results in fatigue tests.
2. Non homogenized and 20 $\mu$ m(micronmeters) coated sample comes after the first one. Considering this, it can be understood that thin coating in micro arc oxidation has the biggest positive effect on the surface properties of aluminum.
3. Homogenized and 120 $\mu$ m(micronmeters) coated specimens are coming after these 20 $\mu$ m(micronmeters) coated samples
4. Non homogenized and 120 $\mu$ m(micronmeters) coated specimens are coming after 3rd one.
5. Only homogenized specimens have nearly the same fatigue results as 3rd and 4th one,
6. Non homogenized and non coated specimens have the fatigue results that are known.
7. 60 $\mu$ m(micronmeters) coated specimens have the worse fatigue test results no matter how homogenized or non homogenized they are.

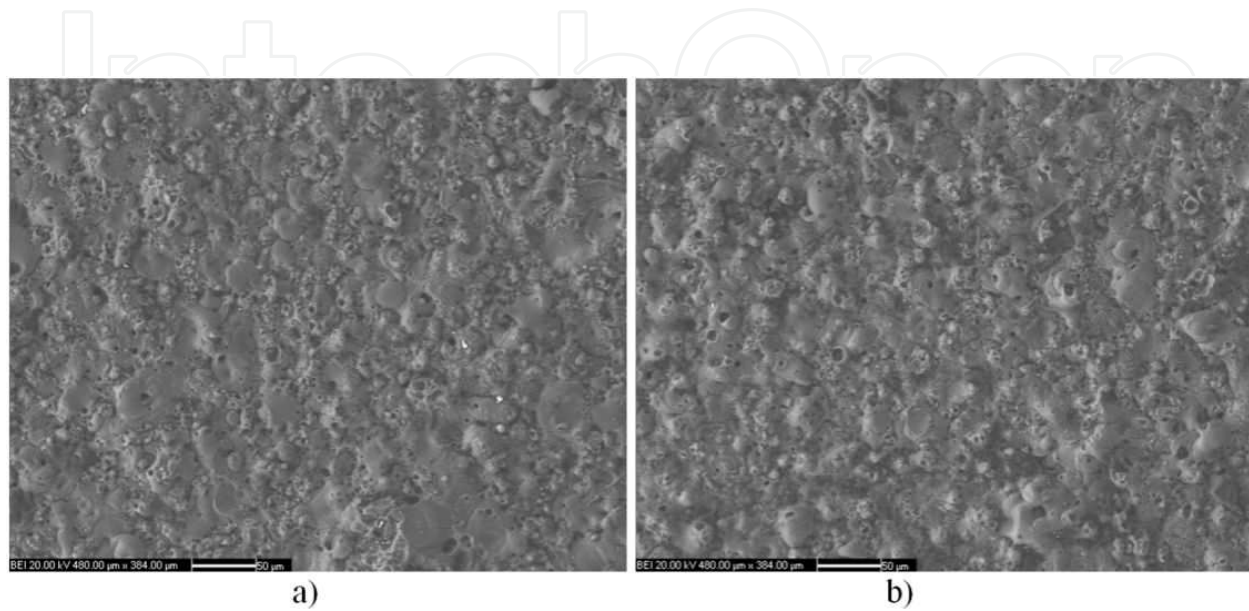


**Figure 7.** Fatigue test results of the specimens.

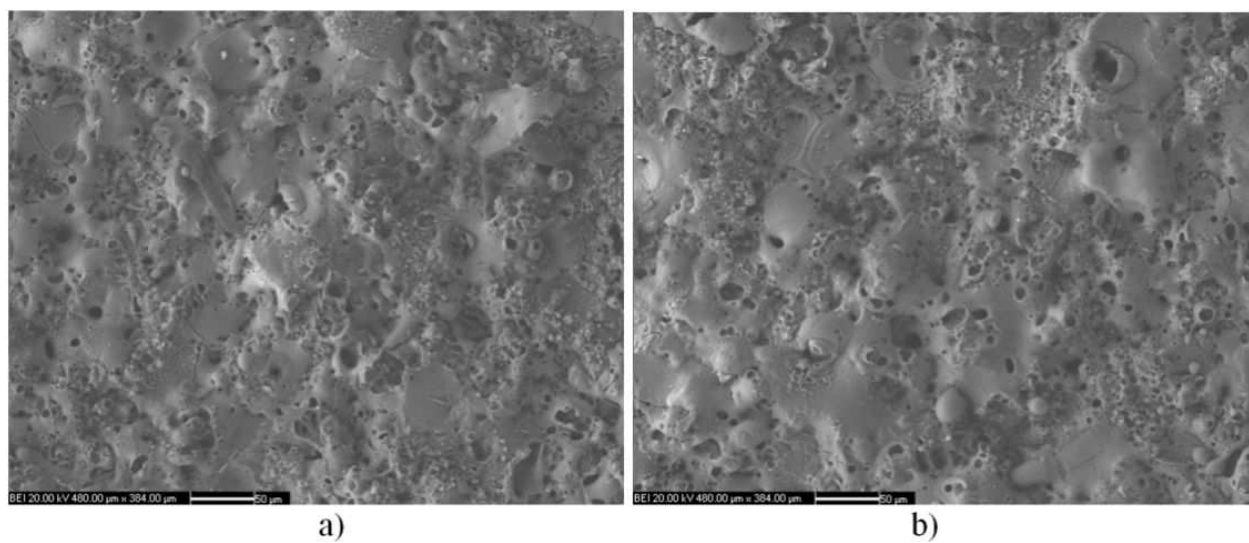
The results according to these tests are given in Figure 7. From Figure 7, we can understand that micro arc oxidation process has better effect on the surface properties of AA 2014 Aluminum alloys than homogenization, furthermore it can also be obtained that for surface properties, thin coatings are better than thick coatings in micro arc oxidation and the middle coatings are the worse coatings in micro-arc oxidation.

The SEM analyses give us an idea about the surface of the coating (Figure 8, 9 and 10.). According to the coating time the thickness of the coating on the material will increase but in MAO process, at first the quality of the coating increases with the increasing coating time but after the derigueur time the quality of the coating decreases with increasing coating time that is why finding the optimized coating time has a vital importance in MAO coatings, with the help of SEM analysis we can see the amount of pores on the coating and this will help us in understanding the quality of the coating. From the results shown above, it has been understood that the 120 $\mu$ m(micronmeters) coated specimens have more pores than the 60 $\mu$ m(micronmeters) coated specimens, but 120  $\mu$ m(micronmeters) coated specimens showed better results on fatigue tests because of the high quality bonding between the base

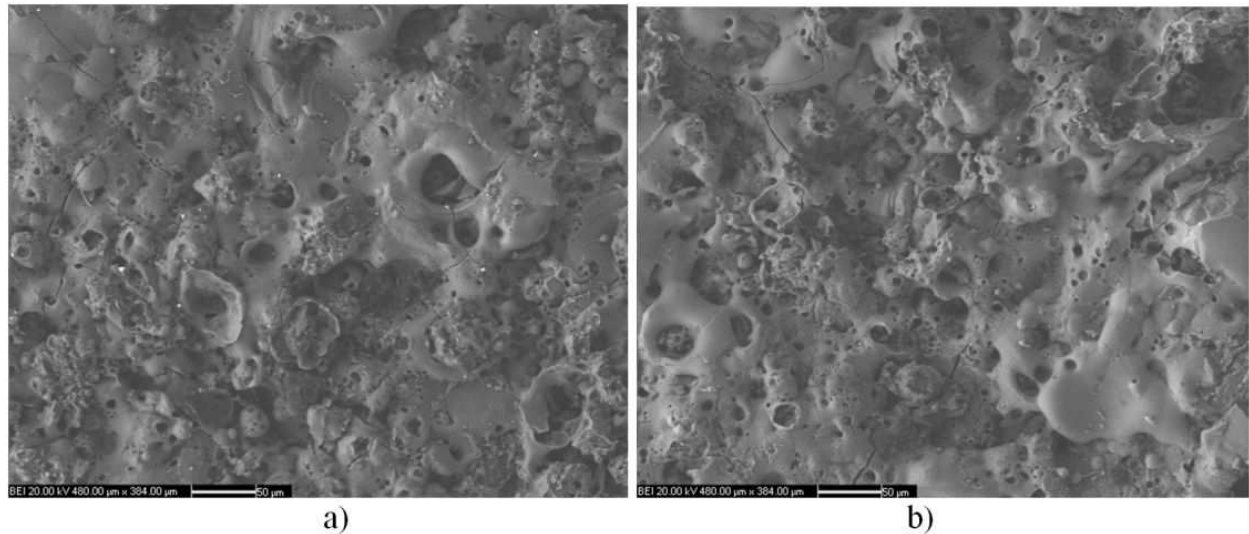
material and coating material. As expected before, 20  $\mu\text{m}$ (micronmeters) coated specimens have the best results.



**Figure 8.** SEM photographs of 20  $\mu\text{m}$ (micronmeters) coated specimens. a) non-homogenized, b) homogenized

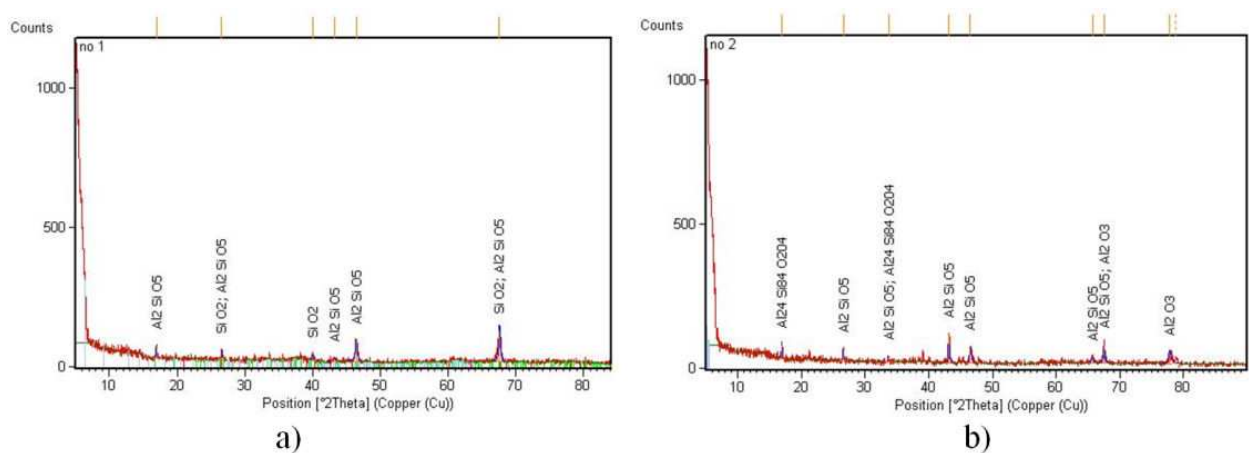


**Figure 9.** SEM photographs of 60  $\mu\text{m}$ (micronmeters) coated specimens. a) non-homogenized, b) homogenized



**Figure 10.** SEM photographs of 120  $\mu\text{m}$ (micronmeters) coated specimens. a) non-homogenized, b) homogenized

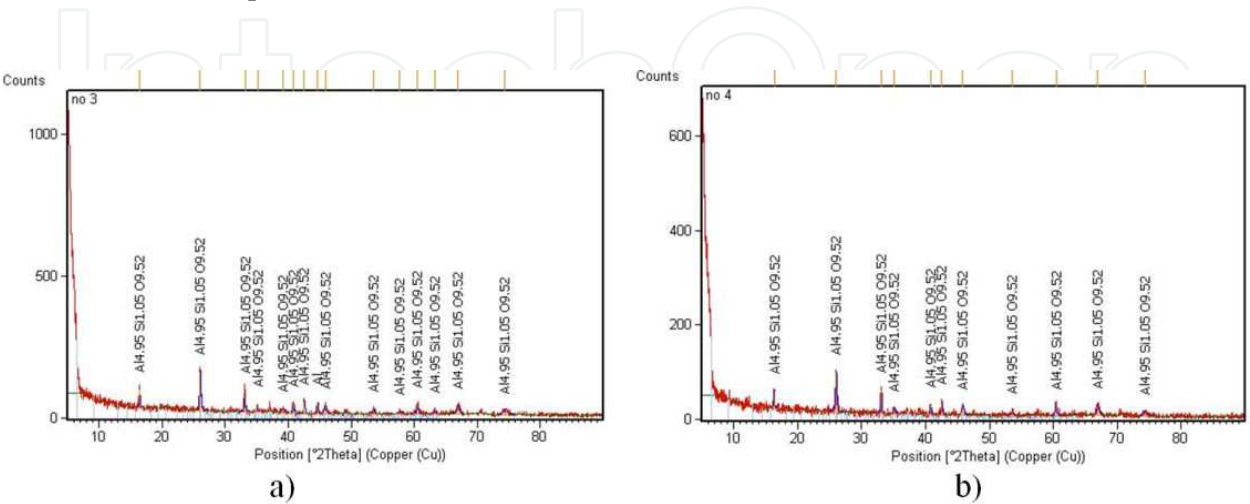
X-ray diffraction test will help us understand the phase composition of the coating. Basically, this test will show the penetration depth of the coating in different angles and will give us some ideas about the composition of the coatings. Nie and Matthew used this test in angles  $0.5^\circ$  and  $2.0^\circ$  respectively and found out that in different depths of both porous and dense layer the coating composition remains same in mixture of both  $\alpha \text{ Al}_2\text{O}_3$  and  $\gamma \text{ Al}_2\text{O}_3$ . [3,12,14].



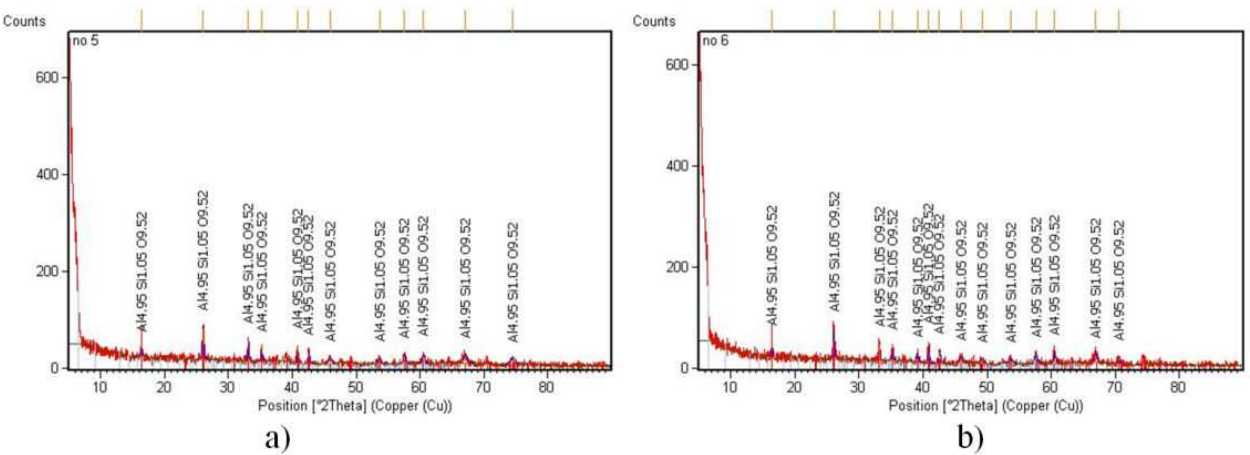
**Figure 11.** XRD results of 20  $\mu\text{m}$ (micronmeters) coated specimens, a)non-homogenized, b)homogenized

XRD results in Figures 11, 12 and 13 show that homogenization have no effect in phase composition, but have a positive coating effect because it increases the combining ratio. According to these results, there is the same combination of coating that includes

3 $\mu\text{m}$ (micronmeters) (in pours layer) and 10 $\mu\text{m}$ (micronmeters) (in dense layer) of  $\alpha$  and  $\gamma\text{Al}_2\text{O}_3$  with another alumina silicate compounds shown in figure. XRD spectra and results collected from the coated specimens indicate a considerable increase in oxygen and silicon content, explaining that the coating has a combination of aluminium oxides and complex Al-Si-O alumina silicate compounds. These results show that all MAO coatings had a similar structure and composition.



**Figure 12.** XRD results of 60  $\mu\text{m}$ (micronmeters) coated specimens, a)non-homogenized, b)homogenized



**Figure 13.** XRD results of 120  $\mu\text{m}$ (micronmeters) coated specimens, a)non-homogenized, b)homogenized

## 4. Conclusion

According to the SEM analysis, XRD results and spectrums, fatigue test results, the effect of the homogenization process on MAO coating process can be summarized as follows;

- The homogenization process has no effect on the phase composition of the specimens.

- According to the SEM analysis homogenization treatment has no effect on the number of pores on the coating material.
- According to the fatigue tests, it is found that homogenization has a positive effect on the quality and the durability of the material.

From these results it is explained that the homogenization treatment does not change the coating material porosity but definitely changes the final coatings durability so this treatment has a positive effect on bonding.

As a conclusion, the MAO process with its advantage is one of the most common coating methods for the light weight materials. Its corrosion resistance, thermal endurance, high hardness value makes this method more important for aerospace and automotive industries. If the investigations on this method come with solution for the expense of the process, it will be the best method for coating light materials.

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