A review of process characteristics of plasma electrolytic oxidation of aluminium alloy.

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Abstract -

The Plasma Electrolytic Method of Oxidation (PEMO) is proved powerful for producing the wear and corrosion-resistant coating of the oxide layer for exclusive applications. This technique is having a better capability for industrial use for growing proactive coatings on material like Aluminium, Magnesium, Titanium for diverse application of lightweight and high temperature requirements applications. The paper consists of review of papers primarily based on the surface modification amendment method of plasma arc oxidation of aluminium and its alloy. Because the process characterized by the discharge generated by electrode, electrolyte composition and oxide coating morphology which might be crucial component for effectiveness of the technique. The purpose of the paper is to write the systematic review for electrodes, electrolyte compositions, and its effect in improving the characteristics of aluminium alloys. There is a large scope for enhancing the method and monitoring the electric parameters of PEMO for massive applications in regions of automotive wherein the modification in surfaces can replace the components by the lightweight aluminium alloy material. The attempt is made to pick the key elements inside the technique. The scope of designing numerous optimization algorithms and analytical modelling is a key area for further research which can help to screen the technique extra precisely to get an optimized coating microstructure.

1. Introduction

For applications like automotive and marine where in excessive power, light-weight, and high resistance to corrosion are critical parameters for long lasting and light weight components. Aluminium preferred because of the remarkable strength to weight ratio which is requirement now a days in automobile, aerospace, and marine applications. But, the use is restrained due to the deterioration of the corrosion performance[1]. For improving the composition of the alloy material it's necessary to apprehend the processing, microstructure, and composition of surface modification methods. Here the Plasma Electrolytic Method of Oxidation (PEMO) is reviewed for recent traits concerning parameters and result of surface characterization. Micro-arc oxidation technique preferred for Aluminium, Titanium, and Magnesium alloys. It can be considered more for Lightweight and high-temperature applications as more work needs to achieved in this region.

It has wide applications in the car industry due to low weight and super mechanical properties [2]–[4]. Al alloy has applications like brake disc in which the thermal functionality of a surface is important at fluctuating load conditions. Al and its alloy are normally favoured because of higher corrosion resistance and wear protection in auto components[5]. The oxidation technique constitutes of substrate, electrolyte, and electric subject and has a robust affect on the discharge characteristics. This electric powered field generates a plasma state among cathode and anode for the polarization method which develops a material which includes substrate material itself with alloying elements and oxide layer. The plasma oxidation method starts with the oxidation of substrate metals.
Exceptional reactions take vicinity due to the polarization effect. Generally, the insoluble metal substrate releases oxygen in the electrolyte. If the metal is soluble in the electrolyte it forms the salt with the electrolyte components consuming the electrode. One more type of reaction forms the passive layer of oxides or hydroxides of the electrode material. The technique develops the thicker layer at the surface of the substrate which is more hard and uniform in composition. The porosity of the coatings is better as well as greater wear resistant than other different methods. The PEMO approach is extra complicated than anodizing and other techniques of producing coatings. As it’s constantly reconstructing layer throughout the procedure and more depending on the uniformity of discharge formation inside the electrolyte.

This review is particularly focused on the aluminium alloy coatings produced by oxidation techniques in various electrolytes.

2. Literature review -

The electrolyte study also identified a few important conclusions about the compositions of the particles used in the electrolyte. Yong Lian et al. applied a micro-arc oxidation method for alloy with electrolytes containing SiC particles. The effect of Si article found dominant in the layer of the coating during the microstructure study. The pores on coating diminished and the homogeneous distribution of Si is observed in the coated layer. The SiC is dielectric at room temperature but a good conductor of electricity at a higher temperature. At higher temperatures, it absorbs more electrons and reduces the formation of spark resulting in the dense and thinner coating[1]. AA 6061 aluminium alloy applied to the oxidation method of micro-arc oxidation using and . The microstructure of coating formed studied for phase changes and uniformity. The phases appeared at the initial phase of the coating which converted into as the time of the oxidation method increases. The density of the micro discharges improved significantly. Also the pore density appeared more at initial coating phases and crater emerges at later time stages. From the experiment data is has been found that the coating growth was parabolic due to presence of higher amount of Si in electrolyte's higher amount of gives higher hardness to coating [2]. Zheng-yang et al. developed corrosion resistance of the Zr alloy by adding graphene which formed coating on the substrate. The graphene observed to be a good barrier for diffusion. The coefficient of the friction becomes lower along with the wear volume due to strong coating. The microstructure study identified that the formation of the pores becomes less with Si-based electrolytes. These pores get filled by GO particle strengthening the material for wear and corrosion. Go particles did not change the composition of the coating.

The layer of the coating contains mainly and phases[3]. A composite coating formed doping of Manganese and cobalt in the alkaline electrode of KOH and along with and . The
coating has an aluminum oxide layer with major content of doping elements. These materials showed significant improvement in the adhesion of coating with base metal in addition to an increase in corrosion resistance and catalytic activity [4]. To develop the compact porosity of the Al alloy Zhu et al. investigated the effect of binary additives of an electrolyte like \((NaPO_3)_6\) and \(H_2BO_3\) on the 6061 Al. The irregular voids, pores developed during oxidation affects corrosion resistance of coating. Hence thicker coating is generated which contains phases of \(Al_2O_3\). The EIS testing shows decreased current density[5].

Current applied during oxidation and time of process were found to be significant parameters for the quality of the coating. the researcher investigated the formation of a ceramic coating by micro-arc oxidation method on Aluminium foil with silicate-based electrolyte. For current value less than 4A the arc failed while at a higher value of current, more than 9A the ablation started. On the other hand, the insufficient time of oxidation results in the low thickness of the coating, and longer duration increased the coating thickness on account of reduction in the quality of the coating. The power frequency and duty cycle of power inputs affect the ceramic coating considerably[6].

The effect of plasma spray studied by Cihan et al. about the input variables current and spray distance on magnesium alloy in the oxidation method. The hardness and wear resistance of Mg alloy increased due to the coating of \(Al_2O_3\). Amongst input variables of oxidation spray, as the distance goes on increasing and increase in values for current, the hardness and abrasion resistance decreases. However, the friction coefficient also gets decreased[7].

The effect of the addition of material elements like Fe, Ni, Cr on corrosion behavior for hyper eutectic Al-Si alloy studied by Kaiser et.al. The microstructure examination was done using SEM analysis, the gravimetric analysis, resistivity analysis also conducted to find corrosion behavior after alloying the elements. The results showed that the temperature acts as catalysts for accelerating the chemical reactions. The thin passive film on the outer layer of the coating tends to decrease the resistivity [8]. The oxidation method used to form the composite layer on the nano crystallized 2618 Aluminium alloy in two steps with cold forging as pretreatment. The effect of the microstructure improved studied at a higher temperature. It observed from the microstructure study that the forging treatment favors the formation of alpha phases of the aluminum oxide which reduced the formation of pores and cracks and improves the coating hardness. But the friction and wear resistance decreased with an increase in temperature. The study of the effect on wear rates showed a sparse increase[9].

Addition of binary element \((NaPO_3)_6\) in the alkaline electrolyte of \(Na_2SiO_3\), KOH, and \(CaH_2O_3\) used in the oxidation method of Al 6061 material. It is seen that the \((PO_3)_4^-\) electrons in the electrolyte have a strong capacity for the adsorption. The coating thickness and porosity increased due to phosphate ions. Up to concentration of 2g/lit the enhancement in the morphology occurs but then starts decreasing resulting in cracks. This is due to the increased conductivity of electrolyte which turns high spark and shows larger pores on the surface of the coating[10].

The addition of suspended particle \(Al_2O_3\) in the electrolyte was investigated by Ping Wang et.al. for 6061 aluminum alloys. These suspended particles showed significant improvement of properties for Mg-based alloy [11] hence studied for effect on Al alloy. Up to concentration of 4 g/lit shows good coating surface while an increase in the concentration loosens surface and starts peeling. The micro hardness first increases and then reduced after certain values of concentration. Consequently, the addition of \(Al_2O_3\) suspended particles in electrolyte did not affect much on the surface integrity of Al alloy produced by PMEO[12].

3. Electrolyte compositions and operating conditions -
The basic two phase model of reaction occurs in conventional electrolysis which is changed by four phases containing steel, dielectric, gas, and electrolyte. The system is working at above important voltages at point $U_1$ and $U_5$. Those phases results the voltage drop across the method\cite{6}. In duration of the oxidation process, the alternate current generates negative polarization. The impact of this cathodic current studied through the statistical approach. Similarly validation can be carried out by kinematic parameters of the oxidation process\cite{7}. Researchers tried to broaden mathematical modelling of the cathode delimitation process and its simulations. The equations have been complex primarily based on the recordings of the experimentations and solved using algorithms. The model developed based on the data which gave a foundation for the analysis of coating failure \cite{8}

As proven in Table 1, most of the electrolyte is alkaline-based totally containing $Na_2SiO_3$ and $KOH$. The pH value generally reaches up to 13 because of this composition. The PEMO process is more dynamic and transient because the composition and electric powered field performs an important role. The electrical conductivity of the electrolyte impacts the current and voltage fluctuation across the coating process. Generally, elements of the electrolyte are included within the coating structure in three distinct ways as forming substrate oxide layer or forming exclusive oxide together with elements and suspensions. $SiC$ particles develop all throughout the PEMO process of metal substrate like aluminium. Table 1 discusses such electrolyte mixtures with different parameters and discussed in detail in the following segment.

### 4. Additives / Particle addition

The oxidation of Al 5052 in electrolyte of $Na_2SiO_3$ with additives $Na_2PO_4$ in different concentrations showed growth of the oxide layer over surface but the surface finish remains same. The higher proportion of the $Na_2PO_4$ shows the formation of mullite which created denser coating\cite{10}. Additives like ($Na_2PO_4$) showed significant reduction in voids of coating surface when added in aqueous solution of $Na_2SiO_3$, $KOH$, $C_2H_2O_2$ in deionised water. The microstructure showed presence of $γ-\text{Al}_2O_3$ which increases hardness of the surface \cite{10}. The addition of the additive materials like Fe, Ni, Cr on hyper eutectic alloy showed that temperature helps to make the chemical reactions. Also, the higher temperature has a considerable effect on the formation of pores and cracks in the coating microstructure. On another side, the friction and wear resistance decreased at the higher temperature.

### 5. Coating microstructure
The micro-arc coatings are a bit complicated in nature because the thickness gets constructed in the oxidation method by utilizing the electrolyte. The oxide layer thickness also depends upon the electrical parameters subsequently, controlling the parameters is one of the crucial area whilst the small thickness of the oxide layer needs to be produced. The oxide layer can verify with high wettability with sturdy surface bonds. The coatings of metallic oxide can repair the cracks at the surface. The oxidation technique can produce hard coatings compared to the anodizing method.

The X-ray diffraction gives the microscopic elemental distribution on the coating surface. Coating microstructure of Al alloys produced by the PEO method based on a mixture of amorphous ($\gamma-\text{Al}_2\text{O}_3$) and crystalline ($\alpha-\text{Al}_2\text{O}_3$) alumina phases induced by electrolyte composition. The possibility of having phases like $\eta$- and $\delta-\text{Al}_2\text{O}_3$, and mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) depending upon the operating conditions of the process. The presence of mullite increases the thermal properties of the substrate along with hardness[14]. The phases like $\alpha,\beta,\gamma, \delta$ phases of $\text{Al}_2\text{O}_3$ have significant effect on mechanical properties of substrate[15].

The oxidation of Al6061 conducted using alkaline electrolyte of $\text{KOH} \cdot \text{C}_2\text{H}_5\text{O}_2$. Due to presence of $\text{(RO}_2)^{-2}$ ions in electrolyte which is having strong adsorption capacity the porosity and thickness increased. As the concentration goes on increasing the cracks starts developing on the surface of the coating. Due to higher percentage of the additives the conductivity of the electrolyte increased creating spark and larger pores on the surface.
Table 1 Oxidation parameters for Aluminium alloy

| Sr. No. | Metal substrate          | Electrolyte                                                                 | Current, voltage and time configurations | Temperature (K) | Reference |
|---------|--------------------------|------------------------------------------------------------------------------|------------------------------------------|-----------------|-----------|
| 1       | AA1060 pure aluminum     | 0.03 mol/LiT Sodium silicate and 0.05 mol/LiT Sodium hydroxide               | 44 A/cm² for 15 min                      | 298 ± 2         | [16]      |
| 2       | AA 7075-T651 Al          | 1 g/L potassium Hydroxide, 20ml/l hydrogen peroxide, Sodium Aluminate in different concentrations (6,9,14 g/l), 0.5 g/lit Graphite | 15 kW DC rectifier                        | -               | [17]      |
| 3       | 1050 Al                  | 1-2 g L⁻¹ of KOH, 2-2.5 g L⁻¹ of Na₃PO₄ and 2.5-3.5 g L⁻¹ of Na₂SiO₅        | Voltage 50 Hz sine wave, 25 Adm-2         | -               | [18]      |
| 4       | AlSi10Mg aluminum alloy  | 25 g/l of Na₂SiO₅ and 2.5 g/l of NaOH                                         | 0.35 Acm-2 for 5 minutes                 | 298             | [19]      |
| 5       | AA 6061                  | Sodium Silicate (in concentrations of 20,40,60,80 gl-1)                       | Varying current from 20- 100 mAcm-2 at time variation upto30 min | 298             | [20]      |
| 6       | AA 6061                  | (5-10) g/L Na₂SiO₅ and (5-15) g/L KOH in deionized water                    | 0.2 Acm-2                                | 311             | [2]       |
| 7       | 2618 Al Alloy            | Na₂SiO₅ (5 g/L) and KOH (0.5 g/L)                                            | 5 A/m², 500 Hz, 60 min                   | 293             | [9]       |
| 8       | 7075 Aluminium alloy     | 10 g/L (Na₃PO₄)₆, 8 g/L Na₂SiO₅, 2 g/L NaOH                                 | 500 V for 3 min, 500 % duty cycle and 8% duty cycle | -               | [21]      |
| No. | Alloy Type       | Composition/Conditions                                                                 | Parameters                                                                                   |
|-----|-----------------|----------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------|
| 11  | 2A12 Aluminium alloy | 15 g/l sodium silicate ($Na_2SiO_3$) and 30 g/l sodium hydroxide ($NaOH$)                | 240 V for 30 min with current density variation (4,7,10 Adm$^{-2}$)                          |
| 12  | 7075 Al alloy    | 10 g L$^{-1}$ $Na_2SiO_3$ + 2 g L$^{-1}$ KOH with varying concentrations of 0.5, 1, 2, 3 g/L | Bipolar voltage from 0 to 700 V, 8 Adm$^{-2}$, frequency 657 Hz, duty cycle 33%              |
| 13  | Al Mg 6 Alloy    | 2 g/L KOH and 5 g/L $Na_2SiO_3$                                                         | 25 A/dm$^2$ current, 280 V, 50 Hz frequency and duty cycle 50% for 60 min                   |
| 14  | Pure aluminium   | 35 g/L $Na_2SiO_3$, 5 g/L NaOH and 5 g/L KF                                            | 340,480 V; 2000, 700 Hz frequency; 20, 30 duty cycle                                         |
| 15  | 6061 Al alloy    | 10 g L$^{-1}$ $Na_2SiO_3$, 0.1 g L$^{-1}$ NaOH, 3 ml/L C$_2$H$_3$O$_2$; Al$_2$O$_3$ varies from 0 to 8.0 g/L | Current density of 3 A/dm$^2$ for 20 min, with fixed frequency of 100 Hz and duty cycle 40% |
| 16  | A1050, A2024, A3003, AlSi12CuNiMg | 1. KOH based - L$^{-1}$ KOH, 0.01 mol dm$^{-3}$, $Na_2O$ - 0.005-0.05 mol dm$^{-3}$, $K_2P_2O_7$ - 0.1 mol dm$^{-3}$ | 5 to 20 Adm$^{-2}$, 120 to 40 V                                                             |
| 17  | 7475 Aluminium Alloy | $Na_2SiO_3$ , KOH                                                                      | 10 A dm$^{-2}$, frequency 50 Hz, duty cycle 50%, 30 min                                     |
| 18  | 7050 Al          | Sodium hexametaphosphate 40 g/L Sodium silicate - $Na_2SiO_3$, 5 g/L Sodium, Sodium tungstate 3 g/L | 2.5 A dm$^{-2}$, 500 Hz frequency, 4% duty cycle, time - 200-1000 μs                         |
| 19  | Pure aluminium   | 2g/l KOH, 9.5 g/l $Na_2SiO_3$·$SH_2O$                                                   | 500 V, Duty cycle 8%, pulse duration 200-1000 μs, time - 20, 30, 40 min                    |
| No. | Alloy/Alloy Type | Solution Composition/Conditions | Parameters | References |
|-----|----------------|---------------------------------|------------|------------|
| 20  | Al alloy D16 T  | 1 g/l Potassium hydroxide, 3 g/l Na$_2$SiO$_4$, in aqueous solution with addition of glass | 50Hz, current density 20A.dm$^{-2}$, - | [29] |
| 21  | Al alloy A3     | 10% Na2SiO3 aqueous solution   | 200 v, 10A, time 4 S, - | [30] |
| 22  | 2024 T3, 7075 T6 Al alloy | 4 g/L KOH and 2 g/L Na$_2$SiO$_4$ in the de-ionized water | 0.1 A.dm$^{-2}$, 75 kVa AC power supply, 293±5 | [31] |
| 23  | 2017, 5083 Al alloy | Na$_2$SiO$_4$ + KOH | 80-400 V, 3 to 5 min, 295 | [32] |
| 24  | M 224 Al Alloy  | 1-4 g/l KOH, 1-4 g/l Na$_2$SiO$_4$, water glass in varying concentration - | Capacitor capacitance 100-400 μF, ratio of anodic and cathodic current 1,120 min, 323 | [33] |
| 25  | Al 5052        | 15 g Na$_2$SiO$_4$, Na$_2$PO$_4$ in concentrations of 1,1.5, 2 g/L | 20 kW DC power supply, 420 V for 10 min, 313 | [34] |
Table 2 Significant parameters of coating study.

| Sr. No. | Review criteria | References | Observations |
|---------|----------------|------------|--------------|
| 1.      | Additives      | [18],[4],[34],[35],[36],[35],[37],[14] | Graphite, Na$_2$PO$_4$, water glass, K$_2$P$_2$O$_7$, Sodium tungstate are additive compositions frequently studied. |
| 2.      | Wear resistance | [26],[17],[19],[7],[38],[39],[6],[40],[41],[33],[42] | Improving the wear resistance |
| 3.      | Corrosion resistance | [8],[17],[21],[24],[27],[38], | Significant changes in corrosion resistance with other mechanical properties like Fatigue strength and hardness. |
| 4.      | Thermal properties | [43]–[47] | work on the thermal barrier, thermal expansion and thermal conductivity properties of the material for different applications. |

6. Potential area of research -

The figure indicates the important thing area of research within the oxidation technique. The work is done on the coating characterization prominently. The research work can be extended for validation of the mechanical properties like wear, fatigue strength, thermal properties as per application. There's potential work that desires to be executed within the optimization of electrical parameters and impact on the coating structure. Different algorithm techniques can be applied to formulate a relationship among the input parameters and required properties of the surface. The mathematical modeling of the input parameters and output parameters like discharge characteristics, morphology of the surface may be area for studies. This can help to identify the optimized parameter as per coating characteristics required. There is significant scope for setting the electrical parameters and chemical composition of the electrolyte. For this reason, approach for controlling discharge attributes of this dynamic and transient technique may be carried out. The PEMO is proven to be effective for producing the corrosion resistance and wear resistance method for surface change still the validation on applications is missing as evident in available literature.
7. Conclusion -

It is vital to recognize the versatility of the process for various applications. The PEMO coatings are mainly dependent on the electrical input parameters and electrolyte components. For this reason, there may be a large scope of tailoring these inputs to get the coatings as per the functionality. However, there are some demanding situations with the method like establishing a relation among current, voltage inputs and output coating morphology needs to explore more to apply more in the commercial or business use. This can also be supported by using establishing analytical models, optimization techniques between the various process parameters. This will help to make the process more economical and accessible for commercial use.

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