

PULSE ANODIZING OF AA1100 ALUMINIUM ALLOY IN OXALIC ACID ELECTROLYTE

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Pulse anodizing of AA1100 aluminium alloy has been studied in oxalic acid electrolyte at 303 K using indigenously developed pulse generator, which can deliver different 'On' and 'Off' time, base and peak current and anodizing time. The influence of pulse parameters such as duty cycle, Pulse 'On' and 'Off' time and current density on the properties of the anodic oxide coating such as thickness, hardness, breakdown voltage and coating ratio has been investigated in order to assess the quality of oxide coating.

Keywords: Aluminium alloy, anodizing, pulse, duty cycle, oxalic acid, current density, thickness, hardness, coating ratio and breakdown voltage.

INTRODUCTION

Anodizing is carried out generally to produce thick and hard anodic oxide coating on metals especially on aluminium and its alloys. Several anodizing electrolytes are introduced to produce oxide film on aluminium and its alloys for use in different fields [1-7]. A large proportion of wrought aluminium alloys is anodized in sulphuric acid [1], chromic acid [2] and phosphoric acid [3] to yield a film consisting of a thin barrier layer and an outer porous layer. For decorative and architectural purposes, anodizing has been carried out in sulphuric acid or oxalic acid electrolyte [8-10]. Other solutions such as sodium tartrate [4], boric acid [5] and dihydrogen ammonium ortho phosphate [6] yield barrier layer only useful for electronic applications. Sulphamic acid [7] is used for hard anodizing of aluminium and its alloys even at room temperature.

DC or AC superimposed DC or AC modified DC power sources are not suitable for hard anodizing due to burning of oxide film which occurs during the formation of oxide film. Pulse anodizing [11-12] is of recent origin which helps to anodize aluminium alloys having higher percentage of silicon and copper which are normally very difficult to anodize with greater easiness than by DC or AC or AC superimposed DC processes.

A detailed study on anodizing of aluminium and its alloys in oxalic acid electrolyte has already

been made using DC [8-10]. But reports on anodizing of aluminium alloys in oxalic acid using pulse current technique is very little. In our earlier paper [7], we have studied the anodizing of aluminium in sulphamic acid electrolyte using pulse current technique with and without addition agents. In this paper pulse anodizing of AA1100 aluminium alloy in oxalic acid electrolyte has been reported.

EXPERIMENTAL

Aluminium alloy sheets of AA1100 of size 50 mm x 20 mm x 1 mm were subjected to usual surface pretreatment. After surface pretreatment, the clean dry surfaces of aluminium samples were anodized in oxalic acid electrolyte. The aluminium sample to be anodized was kept as anode and was connected to the positive terminal and the two lead sheets on either side of the anode act as cathodes and were connected to the negative terminal of the pulse current source. The anodizing electrolyte was prepared in a 5 litre glass beaker provided with a glass cooling coil. The electrolyte used was 5% (w/v) oxalic acid. The temperature of the bath is maintained at 303 K by circulating chilled ethylene glycol water mixture. Air agitation was given to dissipate the heat uniformly. Average current density was varied between 1 A.dm⁻² and 2 A.dm⁻². The duty cycle was varied between 40% and 90%. The pulse unit employed for carrying out the pulse anodizing

experiments was a PC based pulse console developed at EEI division, CECRI, Karaikudi [7]. The thickness of the oxide film was measured by Dermatron, model 9D which works on the eddy current principle. Measurements were made at three points and mean value was determined. The hardness of the oxide film was measured with a LECO micro hardness tester model M-400 using a diamond pyramid indenter by applying a load of 100 g. The breakdown voltage of the oxide film was evaluated with breakdown and insulation tester model RM 215-G supplied by British Physical Laboratories, India. The coating ratio of the oxide film was evaluated after stripping the oxide coating using the stripping solution containing 30 ml.lit⁻¹ phosphoric acid and 25 g.lit⁻¹ chromium trioxide using the following formula

$$\begin{aligned} \text{Coating ratio} &= \frac{w_2 - w_3}{w_1 - w_3} \\ &= \frac{\text{Wt of oxide coating}}{\text{Wt of total aluminium dissolved}} \quad (1) \end{aligned}$$

where w_1 is weight of the sample before anodizing, w_2 is weight of the sample after anodizing and w_3 is weight of the sample after stripping.

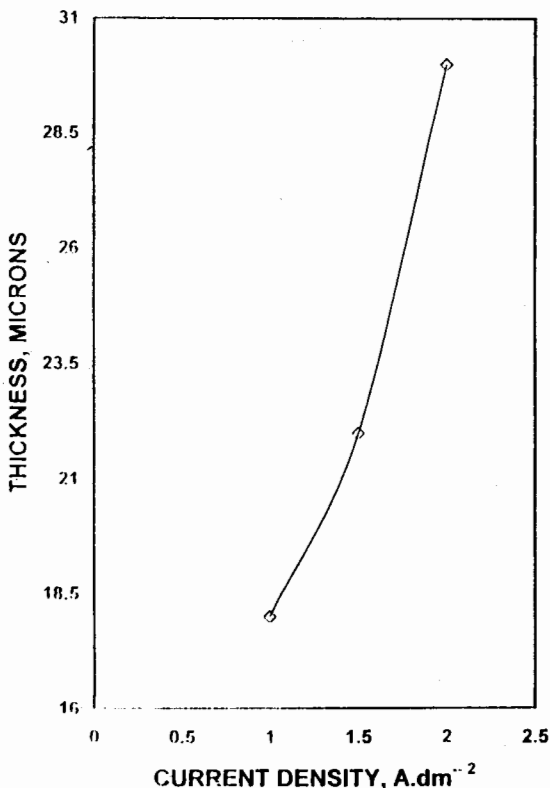


Fig. 1: Effect of current density on thickness of the oxide film

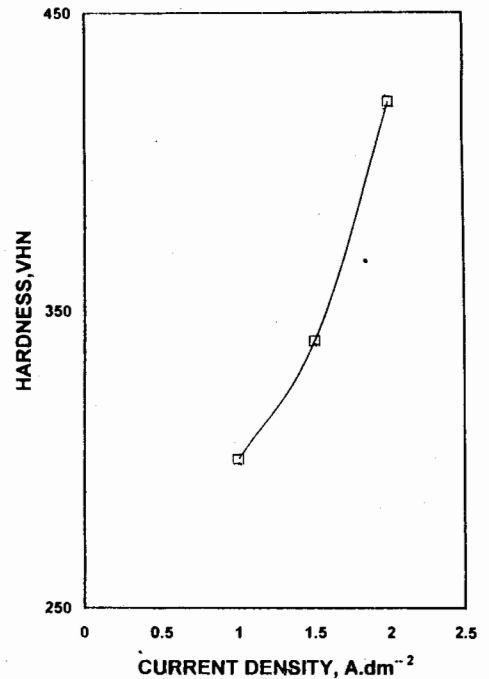


Fig. 2: Effect of current density on hardness of the oxide film

RESULTS AND DISCUSSION

The quality of oxide film on aluminium is assessed by measuring the parameters such as thickness, hardness, coating ratio and breakdown voltage at different current densities and duty cycles especially in the case of pulse anodizing. In the pulse current technique, the influence of 'On' and

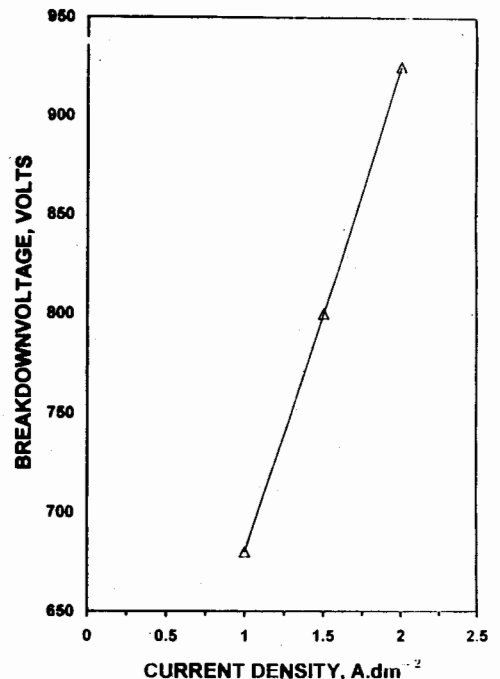


Fig. 3: Effect of current density on breakdown voltage of oxide film

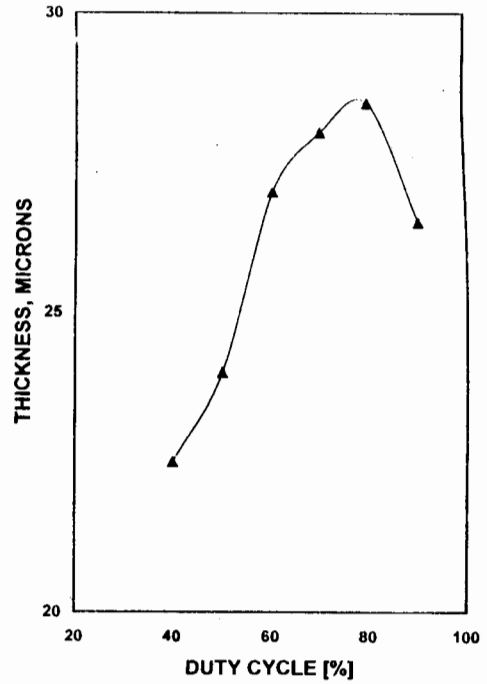
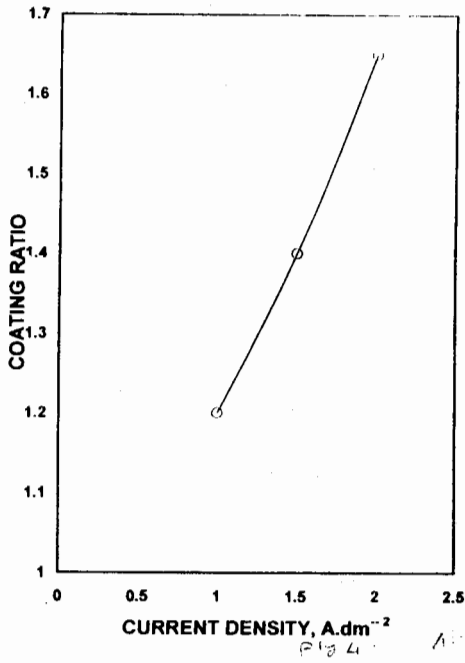


Fig. 4: Effect of current density on coating ratio of oxide film

Fig. 5: Effect of duty cycle on thickness of the oxide film

Off time, duty cycle and current density are the most important factors to be studied.

Effect of current density

Figs. 1-4 show the effect of current density by pulse anodizing on the properties such as thickness, hardness, breakdown voltage and coating ratio of the anodic oxide coating formed.

In all the cases as the current density increases, the value of thickness, hardness, breakdown voltage and coating ratio increases. This may be explained as follows. The rate of formation of the oxide film is determined by current density. During anodizing the rate of formation of oxide film increases gradually as the current density

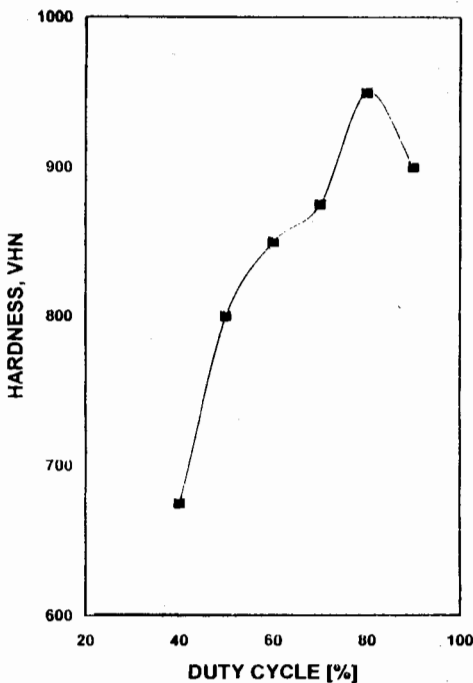


Fig. 6: Effect of duty cycle on hardness of oxide film

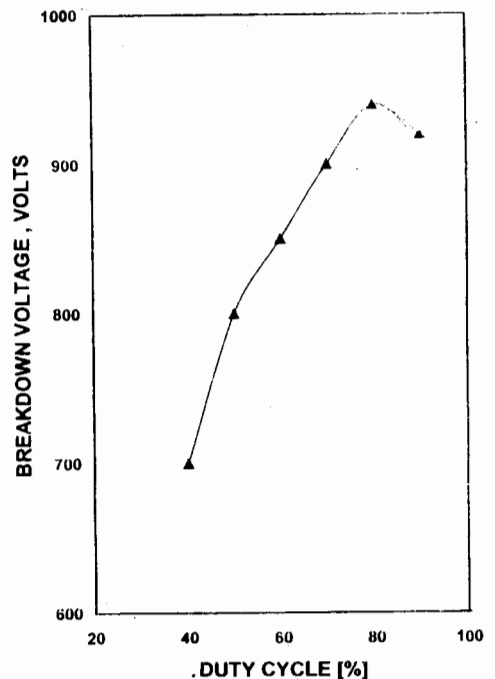


Fig. 7 Effect of duty cycle on breakdown voltage of oxide film

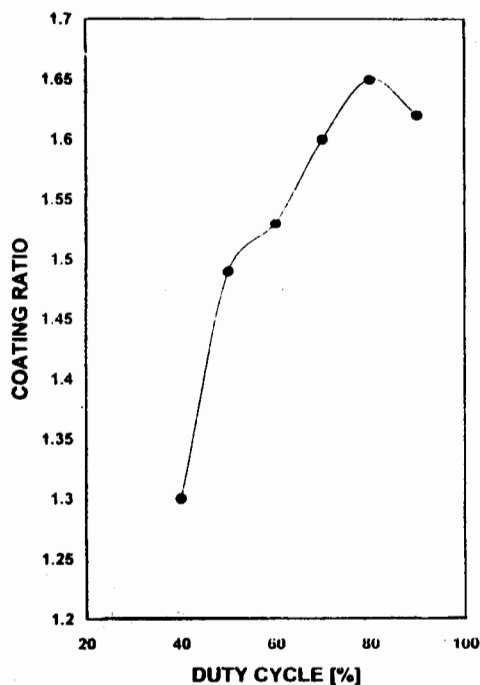


Fig. 8: Effect of duty cycle on coating ratio of the oxide film

increases and it predominates over the rate of dissolution of oxide film which also takes place simultaneously during anodic oxidation. As the rate of formation of oxide film increases, the value of thickness, hardness, breakdown voltage and coating ratio increases.

Effect of duty cycle

Figs. 5-8 represent the effect of duty cycle on thickness, hardness, breakdown voltage and coating ratio of the oxide film formed by pulse anodizing in oxalic acid. From the figures it is seen that as the duty cycle increases upto 80%, all the properties of the oxide film such as thickness, hardness, breakdown voltage and coating ratio increases.

When the duty cycle is increased, the overall current passing through the cell during 'On' time of the pulse cycle also increases. So the current density is impressed for longer duration in higher duty cycle than in lower duty cycle. Hence, higher values of thickness, hardness, breakdown voltage and coating ratio are obtained at higher duty cycle compared to lower duty cycle. The maximum values of thickness, hardness, breakdown voltage and coating ratio are obtained at 80% duty cycle. Above 80% duty cycle the property of the oxide film decreases slowly. This is because above 80% duty cycle, there is no influence of pulse effect

and it behaves like pure direct current and hence the property of the oxide film deteriorates. Because the 'Off' time duration is very short, the relaxation and recovery effect does not exist. As a result of that dissipation of heat produced during the formation of oxide film does not take place.

DC anodizing produces a rough metal oxide interface, which causes concentration of current on the metal peaks protruding in to the oxide film and leads to overheating and subsequent burning of the oxide film. But in pulse anodizing during recovery stage, small current concentrate on peaks and smoothen down the interface thereby avoiding the danger of burning. This results in better quality of oxide film compared to conventional DC anodizing.

CONCLUSION

- Pulse anodizing produces good quality of oxide film compared to DC anodizing.
- Properties of oxide film such as thickness, hardness, breakdown voltage and coating ratio increase with increase of duty cycle and current density and the maximum value is obtained at 80% duty cycle and at 2 Adm.⁻².

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