

## ANODISING OF ALUMINIUM IN SULPHAMIC ACID ELECTROLYTE

D KANAGARAJ, V L NARASIMHAN AND S VENKATAKRISHNA IYER

Central Electrochemical Research Institute, Karaikudi 630 006. INDIA

Anodising of aluminium and its alloys in conventional anodising electrolytes such as sulphuric, oxalic, chromic and phosphoric acid to get the required physical properties for desired applications are well known. Anodising in organic acids was also investigated in the past. Hard anodising at ambient temperature to get thick, hard and wear resistance coating on aluminium alloys using sulphamic acid which is one such promising electrolyte. This electrolyte system has been selected due to lesser aggressive power and ability to produce more compact oxide film on aluminium alloys. So a systematic investigation has been carried out and the physical properties of the oxide film produced under various operations conditions are measured. The effect of concentration, temperature, current density and addition agents on the quality of the oxide film are reported and the results are discussed.

**Keywords:** Anodising, sulphamic acid bath and aluminium alloys

### INTRODUCTION

Aluminium is the most widely used non-ferrous metal next to steel for various purposes. It is such a versatile metal that it is suitable for multitude of applications right from kitchen ware upto space applications. Depending upon the functional application, the surface has to be treated to suit the working condition.

Though aluminium can form a thin protective film because of its greater affinity for oxygen, for more aggressive applications, the oxide film are produced artificially by electrochemical oxidation in a suitable electrolyte. Generally for decorative purpose, anodizing is carried out in sulphuric acid at  $293 \pm 2$  K using direct current impressing a current density of 1.2 to 1.6 A/sq.dm. Other electrolytes such as oxalic acid, phosphoric acid, chromic acid are also used for anodizing depending upon the end use. Apart from traditional anodizing electrolyte, use of a moderately aggressive and un toxic electrolytes are being studied to obtain enhanced resistance to corrosion and wear.

R Piontelli patented a process for anodic oxidation of aluminium in a sulphamic bath for the production of porous oxide film. Later on sulphamic bath was studied by Reynolds company in USA [1] and Tajima *et al.* in Japan [2] to determine the mechanical and chemical characteristics of the oxide layers. A detailed study on the sulphamic acid has been made in Russia [3] to establish the optimum conditions of operations to achieve anodic layers which is comparable to that of sulphamic and oxalic acid processes.

A systematic investigation has been carried out on the anodic oxidation of different alloys of aluminium using various addition agents to sulphamic acid bath. Parameters such as

thickness, hardness, coating ratio, were determined to arrive at suitable bath composition for anodising aluminum alloy suitable for engineering applications.

### EXPERIMENTAL

The anodising electrolyte was prepared in a 5 litre glass beaker provided with glass cooling coil. The temperature of the electrolyte was maintained at  $308 \pm 1$  K by circulating chilled ethylene glycol water mixture. Agitation was provided by means of compressed air obtained from a rotary air pump to dissipate and to maintain uniform temperature through out the bath. Graphite cathodes are placed along the walls of the beaker on either side of the sample to be anodised.

The anodising electrolyte contains sulphamic acid and various addition agents such as sodium sulphate, Aluminium sulphate and magnesium sulphate upto 3 gpl. A current density of 1 to 2 A/sq.dm was applied using a CC/CV stabilised power supply to impress constant current.

Aluminium alloy sheets or AA 1100, 6061 and 2024 of size 40 mm x 65 mm x 125 mm were subjected to the usual pretreatment and then anodized.

The hardness of the oxide film was measured with a LECO Micro hardness tester Model M-400 using a diamond pyramid indenter applying a load of 100 g. The thickness of the coating was above 30 microns and the depth of the indent into the film thickness thereby limiting the influence of the base metal. Measurements were made at three points and value was determined. The thickness of the hard oxide film was measured with Dermitron, Model 9-D which works on the eddy current principle.



## RESULTS AND DISCUSSION

### Influence of bath ingredients

Sulphamic acid is used as one of the ingredients of anodising electrolyte because it is less aggressive towards the dissolution of the oxide film. Sulphates of sodium or aluminium or magnesium has been added to sulphamic acid to quicken the aging of the bath and to reduce the anodising voltage and to avoid piercing of the oxide film at the start of anodizing. Without the addition agent, the bath voltage will be 40 to 60 volts. But by adding one of these agents the anodising voltages can be brought down to 30 to 35 volts which favours the formation of the oxide film due to increase in the conductivity of the electrolyte. Since the solvent action of sulphamic acid on the oxide coating is markedly weaker than that of sulphuric acid, the bath can be operated at higher temperature of 303 to 308 K which saves the refrigeration cost while anodising.

### The effect of concentration of sulphamic acid on anodising

Figs. 1 and 2 show the relationship between the current and thickness and current density and coating ratio in 15% w/v sulphamic acid. It is evident from the graphs that the thickness and the coating ratio increases with increase in current density. But a lower operating temperature gives a higher thickness and coating ratio.

In 15% sulphamic acid sufficient thickness has been obtained to measure the hardness. 250 to 300 VPN hardness value was achieved which indicates that the oxide film is sufficiently hard and more compact. From the above results it can be inferred that 15% w/v sulphamic acid is the optimum concentration at which anodising can be carried out.

### Effect of duration of anodising on the quality of oxide film

Figs. 3, 4 and 5 indicate the influence of anodising duration upon hardness, thickness and coating ratio of the oxide film

produced in 15% w/v sulphamic acid bath. The operating parameters significantly affects the properties of the coating. In Figs. 3 and 4 with increase in time of anodising, the hardness and thickness values also decrease after 60 minutes of anodising. Fig. 5 shows the coating ratio for different period of times (upto 90 minutes) (The coating ratio for different period of times (upto 90 minutes)). The coating ratio increases with increase in anodising time upto 60 minutes. Afterwards there is a decrease in the value of coating ratio. The rapid increase in cell voltage due to maintaining a constant current density leads sometime to burning of the oxide film or powdery deposit of the coating. This will result in inferior quality of oxide film. So the optimum current density of 2 A/sq.dm and a duration of not exceeding 60 minutes are the ideal conditions for hard anodising.

### Effect of addition agents on the properties of the oxide film

Figs. 6, 7 and 8 show the effect of addition of sodium sulphate, aluminium sulphate and magnesium sulphate to the anodising electrolyte on the properties of the oxide film such as hardness, thickness and coating ratio. The anodising was carried out with 2 A/sq.dm. for a period of 60 minutes at 308 K. The concentration of the addition agents was increased from 0.5 to 3 gpl.

In each case the concentration of the addition agents from 1 to 2 gpl, higher values of harness, thickness and coating ratio were achieved. Whereas in the concentration range of 2.5 to 3 gpl only marginal increase in physical properties are noted. Of these addition agents tried magnesium sulphate reduces the anodising voltage, quicken the aging of the electrolyte and gives better quality oxide film.

## CONCLUSION

1. The optimum concentration of sulphamic acid for anodising is found to be 15 % w/v when anodised with 2 A/sq.dm. at 308 K for 60 minutes.

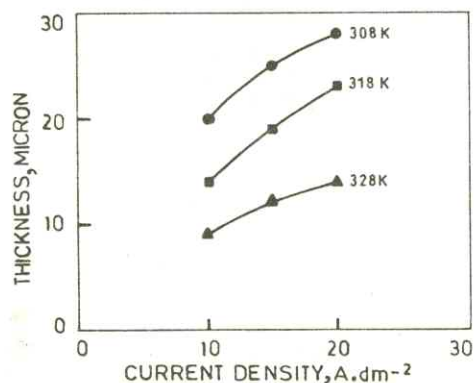


Fig. 1: Effect of current density on thickness in 15% sulphamic acid

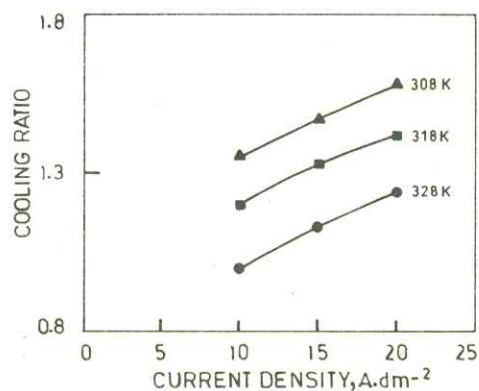


Fig. 2: Effect of current density on coating ratio in 15% sulphamic acid

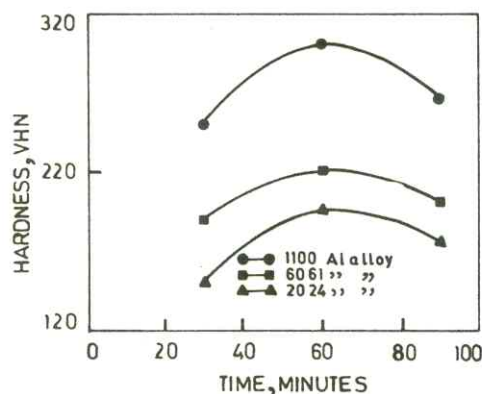


Fig. 3: Effect of anodising time on hardness in 15% sulphamic acid

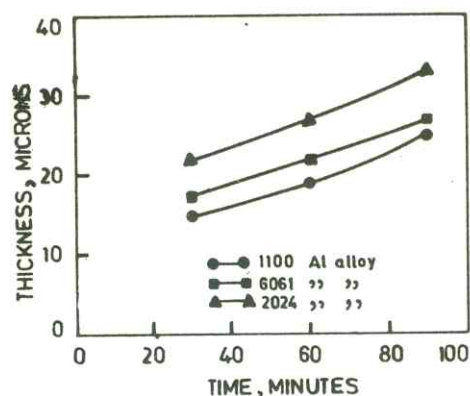


Fig. 4: Effect of time on thickness in 15% sulphamic acid

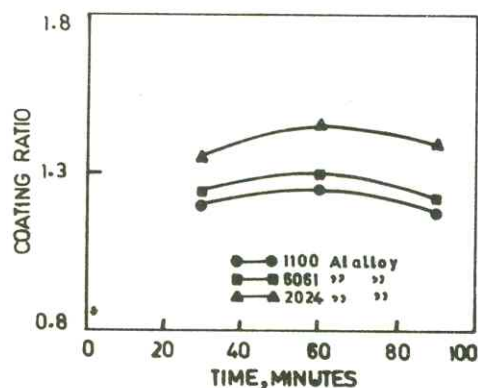


Fig. 5: Effect of time on coating ratio in 15% sulphamic acid

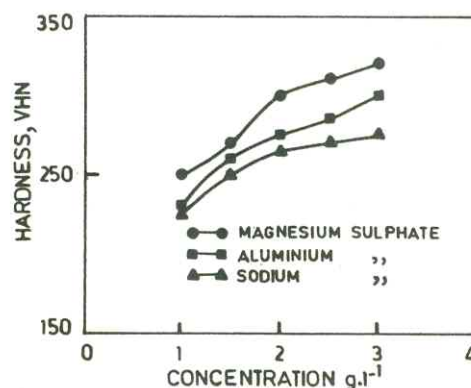


Fig. 6: Effect of additives on the variation of hardness in 15% sulphamic acid at 308 K

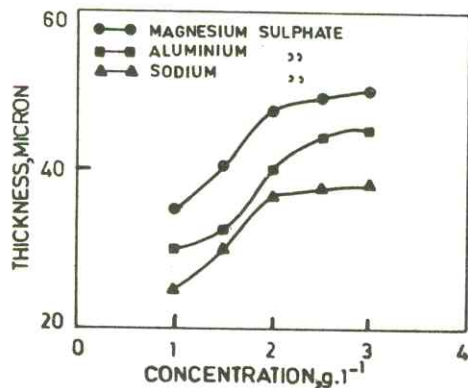


Fig. 7: Effect of additives on the variation of thickness in 15% sulphamic acid at 308 K

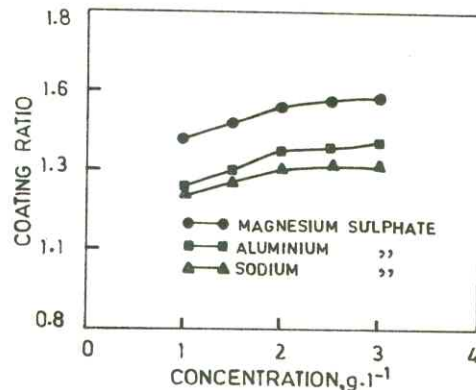


Fig. 8: Effect of additives on the variation of coating ratio in 15% sulphamic acid at 308 K

2. The magnesium sulphate addition improves the physical properties of the oxide film, quicker aging of the anodising electrolyte and lowers the voltage
3. The physical properties such as hardness 250 to 325 HV and thickness of 45 to 50 microns are achieved.

## REFERENCES

1. Reynolds Metal Co Inc, *Finishes for Aluminium* (1946)
2. S Tajima, Y Kimura and T Fukushima, *Metal Finishing*, 50, 10 (1952) 67; 11 (1952) 65
3. A U Bogoyavlensku and N M Vovodina, *ZW Prike Khim*, 40 3 (1967) 565