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The Effect of Electrolyte Temperature on Formation of Porous Aluminium Oxide Films in Anodising Process

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ABSTRACT

A porous aluminium oxide film formed on aluminium was prepared in a mixed electrolyte of phosphoric acid and acetic acid solution. The morphology, growth and thickness of the film were investigated. The anodising process was done by varying the anodising temperature from 5 °C to 25 °C. The constant anodising voltage was applied at 130 V and the anodising was done in the mixture of H_3PO4 1M and CH_3COOH 0.25M. The results indicated that the growth of aluminium oxide film was greater in temperature of 15 °C. The morphology, pore size and thickness of the oxide film were examined by scanning electron microscope (SEM). Results indicated that at 15 °C to 20 °C, larger pore diameter was obtained. This study was explored the effect of anodising temperature to the formation of porous aluminium oxide film in the anodising process.

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INTRODUCTION

Porous aluminium oxide film can be grown in the mixed solution of phosphoric acid and acetic acid solution. It can be generated from a highly pure aluminium substrate by anodising process. The aluminium oxide film that is formed on an aluminium substrate has straight and uniform nanopores that form a hexagonal structure [1]. The aluminium oxide film has a wide range of applications with a highly-ordered porous structure. It can be used in the manufacturing of hard disc, adhesives, templates for nanoparticles, magnetic materials, photocatalysts and also prepare ordered or patterned nanostructure arrays [1 - 8].

Highly ordered of porous aluminium oxide film structured can be obtained by controlling the parameters of anodising process. This process is strictly influenced by important factors such as temperature, voltage, electrolyte used and also anodising duration. Furthermore, the dimensions of pores in the aluminium oxide film, such as diameter, length and density can be controlled by varying the anodising conditions [1,9]. The controlled temperature for aluminium anodising is one of a key factor which is the nature of the aluminium oxide film grown on substrate surface with controlled morphology can be determined.

In this study, mixed acid solution was used to create the aluminium oxide film with controlled morphology. However, the porous aluminium oxide film properties is strictly influenced by anodising process parameter such as anodising voltage, electrolyte temperature, acid concentration and duration of anodising process. Thus, in this study new hypothesis will be generated with exploring the relationship between electrolyte temperature and formation of aluminium oxide film on aluminium substrate in order to enlarge its applications.

Experimental:

The fabrication of aluminium substrate is done by melting and casting process. In this experiment, pure aluminium pellets (99%) were melted in a graphite crucible in induction furnace under vacuum atmosphere at temperature 850 °C. The melt was cast into stainless steel 304 mould (Ǿ 20 mm) and cooled in open air at room

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temperature. The samples then were annealed at temperature 500°C for two hours. The 3 mm thickness samples were ground and then polished using diamond paste. The mixture of phosphoric acid (1M) and acetic acid (0.25M) was used as anodising electrolytes. This anodising process was done at 130 V with temperature 10 °C to 15 °C for 60 minutes. The morphology and structure of porous aluminium oxide film obtained were characterised by Scanning electron microscope (SEM) model JEOL JSM-6460LA SEM.

RESULTS AND DISCUSSION

Morphology and pore size of porous film:

Figure 1 shows the morphology of the film anodised in different temperatures, from 5 °C to 25 °C respectively. At 5 °C, the surface of oxide formed possessed irregular structure with small pores and separated by thick pore walls. The structure of oxide formed at 10 °C look like similar to oxide structure formed at 5 °C. The structure formed was irregular and the pores were smaller and discontinuity, whereas with increasing the temperature to 15 °C, the structure became more open with increasing pore diameters and the pores formed uniformly. For 20 °C anodising temperature, the pores become smaller and separated by thick pore walls. The structure of oxide formed was uniform and continuity. At 25 °C, the structure of oxide film was irregular and non-uniform. The pore diameters were the smallest compared to the others and the pore walls formed were very thick.

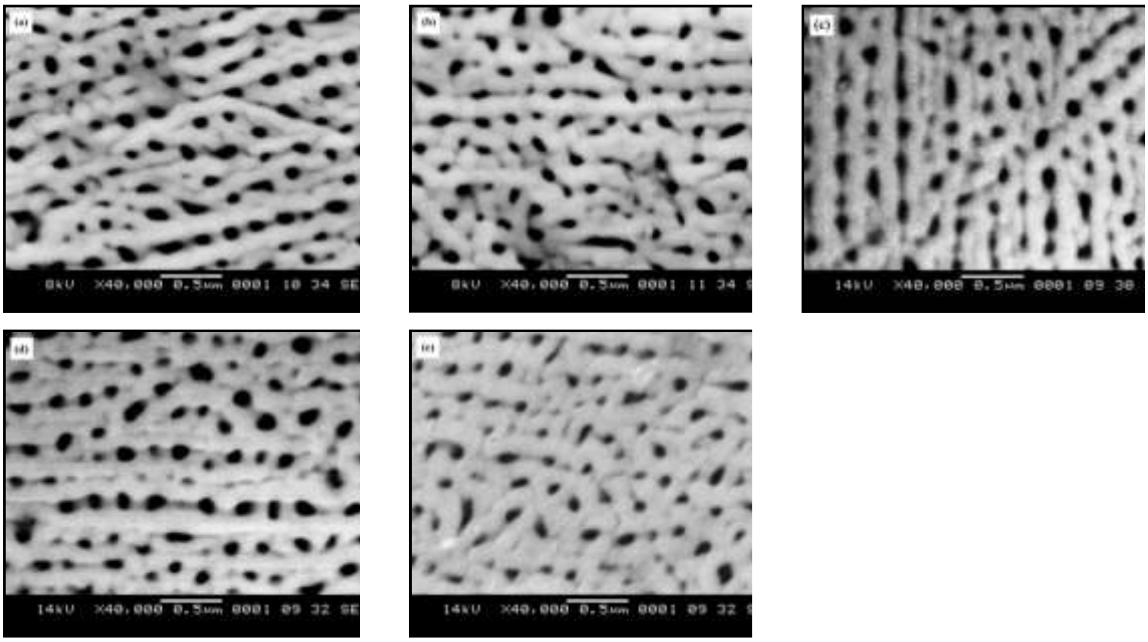

Fig. 1: SEM images of aluminium oxide films formed in H_3PO_4 + CH_3COOH (a) at 5 °C, (b) at 10 °C, (c) at 15 °C, (d) at 20 °C and (e) at 25 °C

The average pore diameter anodised in 5 °C was 70 nm and 69 nm when anodise in 10 °C. The pores were more irregular and smaller. At 15 °C, the average pore diameter became larger which was around 92 nm. At this temperature, the pore diameter was the largest compared to others and the pores were distributed uniformly on the substrate surface. For anodising at 20 °C, the average pore diameter was decreased to 82 nm. The pores were also uniformly distributed on the surface. The increasing of temperature to 25 °C resulted to decreasing of average pore diameter to 66 nm. In this study, the average pore diameter of aluminium oxide film was increased at temperature 5 °C to 15 °C, but it was decreased when the temperature was increased to 20 °C and 25 °C. This trend of result is similar with Shawaqfeh [10] which has reported that the formation of oxide film in phosphate based solution was increased at temperature 20 °C to 25 °C, but it decreased when the temperature reached 30 °C. The effect of temperature on the formation of oxide is well-defined because it affects the rate of ion transport across the barrier layer, the oxide dissolution from the pore wall as well as the oxide surface and heat transport rates within the pore and the bulk electrolyte [9]. Figure 2 shows the average pore diameter for every sample anodised at 5 °C to 25 °C respectively.

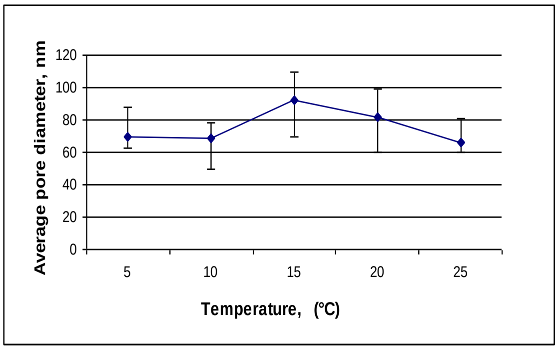

Fig. 2: aluminium oxide film pore diameter anodised with 5 °C to 25 °C

The growth of aluminium oxide film:

Figure 3 shows the effect of anodising temperature on formation of aluminium oxide films on substrate surface. The 5 °C anodising temperature shows the slow reaction of aluminium oxide film growth in 60 minutes. The results indicated that the mass change at 5 °C was only about 0.065%. For anodising with 10° C, the mass change was increased to 0.075%. The growth rate of aluminium oxide film is still slow at this anodising temperature but greater than anodising at 5 °C. The increasing of anodising temperature to 15 °C resulted to the highest reaction rate of formation of nanostructured aluminium oxide film. The result indicated that the mass change at this temperature was about 0.21%. For anodising with 20 °C and 25 °C, the mass change decreased to 0.15% and 0.11%. The growth rate of aluminium oxide film decreased at both temperatures. The mass change increased when the temperature was increased from 5 °C to 15 °C, but it decreased when the temperature was increased to 20 °C to 25 °C.

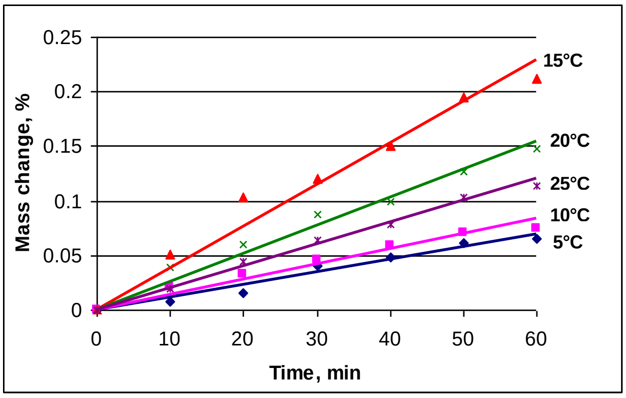

Fig. 3: Effect on different temperatures on growth of aluminium oxide film

Figure 4 shows the thickness of aluminium oxide film formed in different anodising temperatures. From the graph, the thickness of aluminium oxide film was measured by ranging the anodising temperature from 5°C to 25°C and the anodising process was done for 60 minutes. At 5°C, the thickness of aluminium oxide film was 0.63μm. The thickness of aluminium oxide film was continually improved up to 0.75μm at temperature 10°C. For 15°C, the thickness of aluminium oxide film was reach to the highest value which was about 2.82μm. This value was decreased to 1.08μm at temperature 20°C and the thickness of aluminium oxide film was slightly increased to 1.27μm as the anodising temperature increased to 25°C. In this study, the rate of the oxide films expressed as the film thickness formed in anodising process. The formation of oxide film on aluminium surface occurs by the contribution of ions in the electrolyte. The existence and behaviour of the ions in the electrolyte depends on the electrolyte temperature. At temperature 5°C and 10°C, the percentage of mass change and the thickness of aluminium oxide film was decreased. At low temperature, the rate of ions transfer in the electrolyte is low. Therefore, the rate of oxide formation becomes low. The optimum anodising temperature that suitable with this type of electrolyte was at 15°C. At this temperature, the ions are efficiently transport in the electrolyte and caused higher formation rate of oxide. The ions generation was also at high rate at this temperature. This was shown by the highest percentage of mass change and thickness of aluminium oxide film at 15°C temperature. For temperature 20°C to 25°C, the rate of ions transport was decreased compared to temperature 15°C. The rate of ions generation and the ions behaviour become slow at temperature 20°C to 25°C and resulted to low rate of aluminium oxide film formation [11].

An increase in temperature resulted to an increase in the growth rate of aluminium oxide film formed in phosphoric acid electrolyte. At temperature 20°C to 25°C, the mass of oxide film increased. When the temperature rises up to 30°C, the mass of oxide film decreased. The effect of temperature on the formation of aluminium oxide film is noticeable because the temperature affects the rate of ion transport across the oxide layer, the oxide dissolution from the pore wall and heat transport rates within the pore and the electrolyte [10,11].

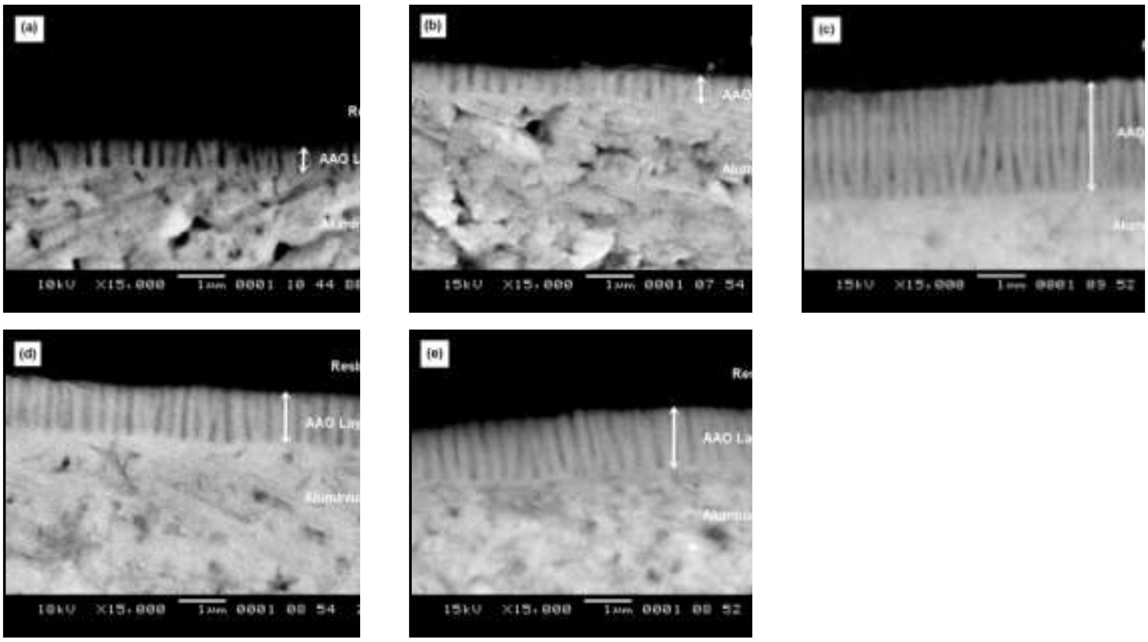

Fig. 4: SEM image of cross sectional film anodised with temperature (a) 5°C, (b) 10°C, (c) 15°C, (d) 20°C and (e) 25°C.

Conclusion:

During anodising of aluminium in H_3PO_4 + CH_3COOH, porous aluminium oxide films were formed. Results for surface morphology and kinetic reactions of the films by varying the anodising temperature were obtained. Anodising at 15 °C led to the larger pores diameter which is around 92 nm and the pores were uniformly distributed on the substrate surface. The percentage of mass change was increased by increasing the anodising temperature until 15 °C and decreased when the temperature increased to 20 °C and 25 °C. In this study, the result shows that aluminium oxide films growth very well on substrate surface at 15 °C and resulted to the large pore size with high thickness. This study showed that the formation of porous aluminium oxide film is strictly influence by anodising temperature and H_3PO_4 + CH_3COOH are suitable electrolyte for growing porous film on aluminium surface with controlled morphology.

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