

Nano Pore Formation in Anodic Alumina

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Development of Nano pores in anodic alumina has been investigated, because of their dominant role in corrosion resistant coating¹, macro/ nano filtration² and ultra high density magnetic recording³. The main issues of these investigations are:-

- (i) effect of mixed electrolytes (maleic acid/oxalic acid, malonic acid/oxalic acid and tartaraic acid/oxalic acid) on anodic oxidation and pore formation in commercial aluminium.
- (ii) effect of mechanical deformation on pore pattern and pore density in anodic alumina
- (iii) possible explanation for self assembly of pores and their deviation from hexagonal pattern⁴

These electrolytes have been used in aqueous solution, near 0°C, with anode (commercial Al sheet of 6cmx2cmx0.35cm size) to cathode (stainless- steel) separation of 1cm (at current densities 5 A/dm² and 10A/dm²). Experiments indicated that (a) an impervious layer of alumina is first formed quickly around 60 ± 5volts hindering further anodization. To continue the process at the same rate, (b) the cell voltage has to be increased by about 15 to 20volts (depending upon the type of electrolyte mixtures) and possibly (c) this causes pore formation by puncturing the oxide film. When pores are formed (d) the anodizing voltage drops (Fig.1). Further growth continues (e) on the average of 1volt per second for every 1.96nm (±0.33nm) of increase in anodic alumina. The pores grow with alumina layer as cylindrical tunnel providing continuous source of oxygen ions.

Fig.2 and Table.1 show film thickness with time, pore diameter and abrasive resistance of alumina coating respectively. It is evident from above figure and table that combination malonic acid/oxalic acid is certainly more corrosive then maleic acid/oxalic acid and tartaric acid/oxalic acid combinations. It is interesting to find that loss in anodic alumina due to pore formation is only a small fraction i.e. around 6% to 8% and any additional loss may be purely due to alumina dissolution from planar area. Fig.4 Compares the actual weight of coating with one calculated on the basis of Faraday’s law. The coating efficiency is only about 26 to 28%. The pores in commercial anodic alumina were noticed to be denser along rolling bands and deviated significantly from the hexagonal pattern⁴. Also they grow in a near tortuous manner in the direction normal to the anodizing surface. This nature has been explained on the basis of repulsive forces between semi plastic cells (containing pores) due to internal stress on account of outwardly growing volume and electrostatic repulsion between pores.

References

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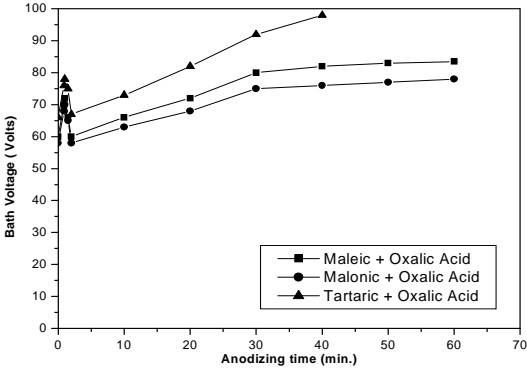


Fig.1.Effect of Anodizing Time on Bath Voltage in Various Electrolytes (at 5A/dm²)

Sl #	Anodizing electrolyte	Pore Separation (nm)	Pore Dia. (nm)	Micro-hardness (VPN)	
				Stirring	Without stirring
1.	Malonic acid (1 mol/l) +Oxalic acid (0.7 mol/l)	65	21.2	430	390
2.	Tartaric acid (1 mol/l) + Oxalic acid (0.7 mol/l)	61.2	16.2	465	440
3.	Maleic acid (1 mol/l) + Oxalic acid (0.7 mol/l)	65	17.5	480	460

Table 1: Effect of Corrosive Nature of Electrolytes

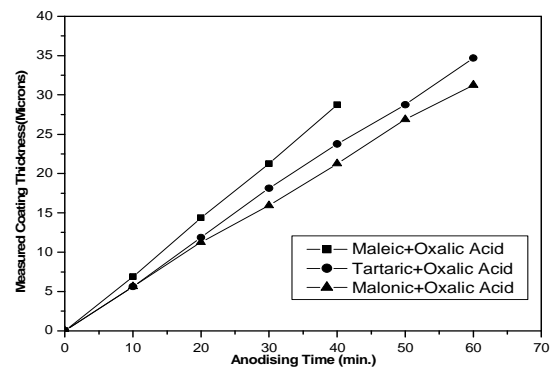


Fig.2.Variation between coating thickness and anodizing time in different anodizing time in different anodizing solutions at 5 A/dm²

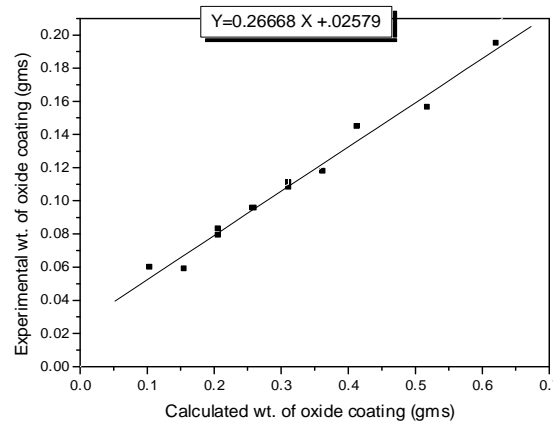


Fig. 3. Experimental & theoretical estimation of anodic alumina coating