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Research Article

COMPARATIVE AND MORPHOLOGICAL STUDY OF ANODIZED ALUMINIUM OXIDE THIN FILMS FORMED AT DIFFERENT CURRENT DENSITIES

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ABSTRACT
Thin films of Alumina can be prepared by various deposition methods, e.g, evaporation, sputtering and also by direct anodic oxidation of aluminium surfaces. Thin film exhibits pronounced internal growth structure, which evolves during the growth (deposition) process and projects onto the film surface, giving rise to film surface topography and roughness. The structural features affect physical properties of the thin films. Therefore properties of produced films can be controlled by an appropriate selection of the preparation methods and conditions, e.g, chemical composition of the electrolyte and other parameters such as temperature and current density in case of an anodizing preparation process . In this paper, Aluminium oxide films are obtained by carefully performing anodic oxidation of aluminium at different current densities and are comparatively studied.The surface morphology of these Alumina films was studied using Atomic Force Microscopy[AFM] which produces the three dimensional image of the surface as well indicating the surface roughness.

Key Words: *Aluminium, Aluminium Oxide Thin Film, Anodic Oxidation,Current Density,Afm.*

INTRODUCTION
Anodizing is a process which thickens the native oxide film resulting in a heavy Al_2O_3 film of controlled thickness. Anodization has been recognized as a valuable medium for extending the life of aluminium. (Fahy, 1979) It enhances the use of aluminium as a light weight and attractive manufacturing component. Anodizing will convert the surface of aluminium to an oxide. (Hsieh An Kong, 1981) Though it would naturally form Al_2O_3 on its surface, it will be a very thin film. Properties of thin films are dependent on the process parameters. During the anodizing process, several controls are critical to assure the specified film thickness, its abrasion resistance and density. These controls include a precise combination of chemical concentration, temperature and current density.The Surface roughness and growth structure of aluminium oxide thin films formed at different current densities, were studied using AFM.

MATERIALS AND METHODS
Experimental Setup
In the present study, aluminium sheets of 99.99% purity are used for the process of anodic oxidation for deposition of thin film. Three samples of different area i.e, 1 cm X 3 cm,1.5cm X 3 cm & 1 cm X 3.5 cm are cut from the pure sheet of aluminium.They act as anodes and a platinum wire of radius(0.5cm)was used as cathode.The oxidation of aluminium was carried out in a specially designed beaker of 100ml capacity and made of corning glass. Always the beaker was cleaned with chromic acid, and finally thoroughly rinse with distilled water before using it for the oxidation purpose.
Aluminium plates are mechanically clean by using MgO paste and then chemically as follows:1. The plates were immersed in a mixture of 15 parts of 70% HNO_3 with 85 parts of 85% H_3PO_4 for 2 mins at 85 C.2. They were then etched in 1N NaOH for 10 min at room temperature to remove any acidic impurities from the surface.3. These Aluminium plates are finally washed

thoroughly in running deionised water for about 2 mins.Anodisation of the clean Al surface was carried at room temperature with platinum wire as the cathode and polished aluminium plate as the anode (Strohmeir, 1990). The electrolyte (anodizing) bath consisted of 5g of ammonium tartarte added to 100ml of distilled water.pH of the bath was adjusted to 5.5 by adding 0.1N tartaric acid.The temperature of the electrolyte during oxidation was in the range of 293K - 313K, whereas the anodic current density was in the range of 3- 5A/dm^2.Always the volume of the electrolyte used for the anodization in electrolytic cell was kept constant. The main objective of this paper was to study the formation of the anodic aluminium oxide thin film by using anodizing process, Study the influence of the experimental parameters such as the electrolyte bath, current density and anodizing time on the formation of porous alumina thin film and determine the difference features of alumina thin film surface morphology by AFM, affected by the various anodizing parameters.

SAMPLES: A1 - Untreated Aluminium

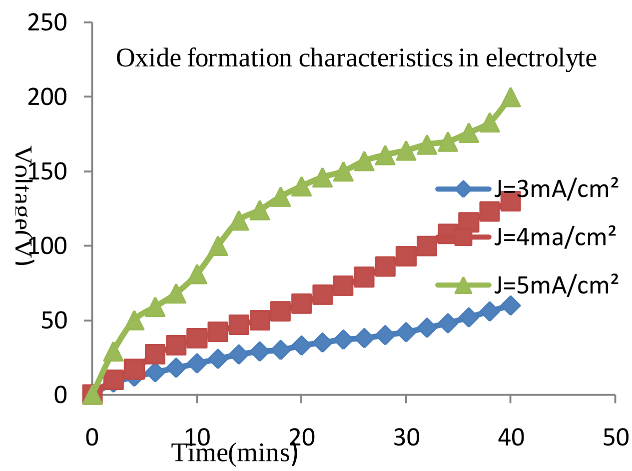
A2 – Aluminium oxide thin film formed at 3 mA/cm^2.
A3 – Aluminium oxide thin film formed at 4 mA/cm^2.
A4 – Aluminium oxide thin film formed at 5 mA/cm^2.

RESULTS AND DISCUSSION

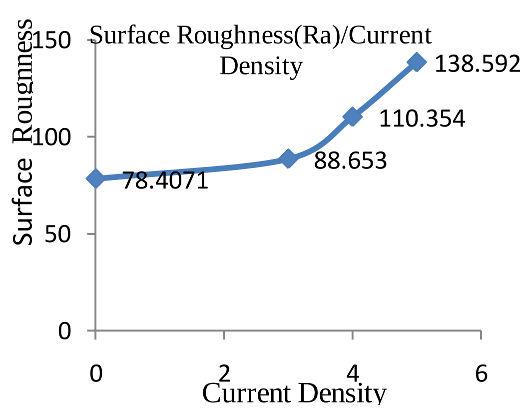
Below Table.1 shows a summary of the parameters of the anodizing process and the AFM parameters, characterizing the surface roughness for several alumina films and also for the parent Al substrate. Surface roughness has been evaluated from topography images using microscope software.

Table 1: Summary of the parameters of anodizin process

SAMPLE	I[Ma/cm^2]	T[min]	R_a[nm]
A1			78.4071
A2	3 ma/cm^2	20	88.6536
A3	4 ma/cm^2	20	110.3540
A4	5 ma/cm^2	20	138.5921



Graph 1

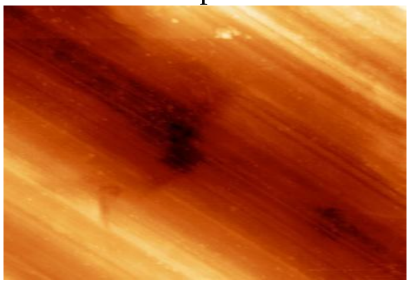


Graph 2

Graph 1: Shows the oxidation characteristics of aluminium in electrolyte at different current densities. The curves represent oxidation characteristics of aluminium at different current densities. It is seen that the oxidation rate is uniform up to 200 volts across the electrodes. Further, it is observed that this oxidation rate increases in current density used for the oxidation. That is, oxidation is faster at higher current densities.

Graph 2: Shows the change in surface roughness of Aluminium Oxide thin film formed at three different current densities

Surface morphology by atomic force microscopy [AFM]: In the present investigation, morphological studies were also carried out using Atomic Force Microscope (AFM). For the present work AFM was operated in contact mode AFM gives more detailed information of surface topography in three dimensions. (Binning *et al.*, 1986) It also gives statistical data of mean surface roughness. So it was thought necessary to study morphology by AFM. AFM studies were carried out using model Nanoscope III from M/s Digital Instruments.



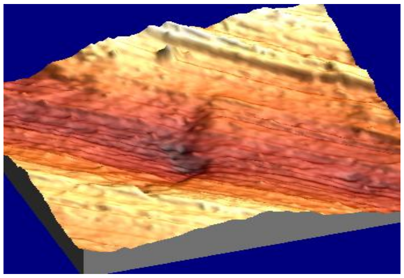
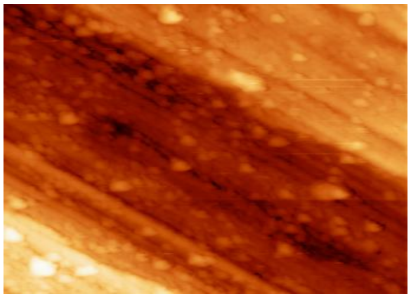


Figure 1: (i) AFM contact mode image and **(ii)** 3-D topography image of Untreated Aluminium



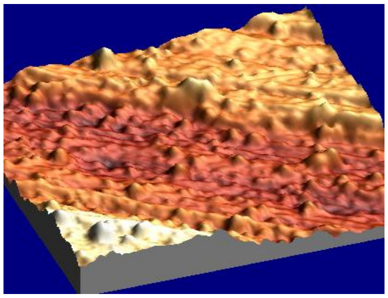
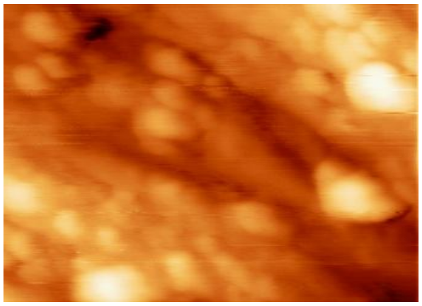


Figure 2: (i) AFM contact mode image and **(ii)** 3-D topography image of Aluminium Oxide film formed at 3mA/cm^2



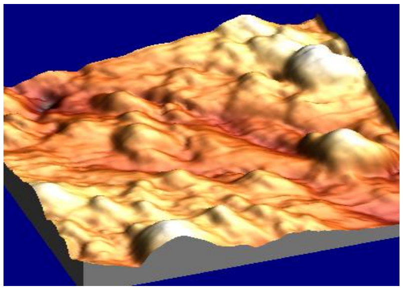
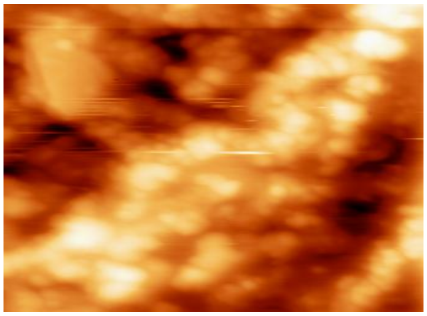


Figure 3: (i) AFM contact mode image and **(ii)** 3-D topography image of Aluminium oxide thin film formed at 4mA/cm^2



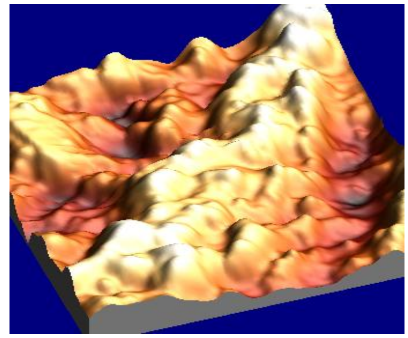


Figure 4: (i) AFM contact mode image and **(ii)** 3-D topography image of Aluminium oxide thin film formed at 5 mA/cm^2

Conclusion

Surface roughness parameters, known and commonly used, are the average surface roughness Ra and surface root mean square rms. Film thickness increases when the anodizing current density and time are raised. The results show that the experimental conditions (anodizing time and current density) are crucial for the final state of the fabricated coatings and especially for the aluminium substrate surface preparation. Anodizing seems to be the growth process which itself can strongly affect the initial roughness of the substrate due to the chemical action of the electrolyte.

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REFERENCES

Binning GB, Quate CF, Gerber C (1986). Atomic Force Microscope *Physical Review Letters* **49**(12) 930.

Fahy FW (1979). Anodized Aluminium-It's Assessment by Accelerated Corrosion, Paper presented at Symposium of Anodized Aluminium.

Hsieh An Kong S (1981). Coloring Anodized Aluminium-A Review *Metal Finishing.*

Strohmeir BR (1990). An esca method for determining the oxide thickness on aluminum alloys, *Surface and Interface Analysis* **15**(21) 51.