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Research Article

PREPARATION OF ALUMINUM OXIDE FILM BY ANODIC OXIDATION AND EFFECT OF PLASMA ETCHING ON ITS SURFACE

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ABSTRACT
Although properties of Al_2O_3 seems to be well known, it has been extensively studied both in bulk as well as in thin film form because of its possible application ranging from micro electronics and optical applications to wear resistance coatings. Thin films of alumina are prepared by anodic oxidation of aluminium surface. These films were treated in air plasma for various duration of time. Plasma treatment is done in a plasma reactor using capacitively coupled RF glow discharge. Glow discharge plasma is obtained at a pressure of 0.1 torr. Surfaces of both the untreated and plasma etched films are studied using scanning electron microscope (SEM) and atomic force microscope (AFM). SEM shows the surface morphology and the AFM produces the three dimensional image of the surface as well the surface roughness. Thus the extent of plasma etching of films prepared by anodic oxidation is studied.

Key Words: *Aluminium Oxide, Anodic oxidation, Plasma Etching, Surface Morphology, AFM, SEM*

INTRODUCTION
Aluminium oxide thin films are widely used in many mechanical, optical and microelectronics applications because of their excellent properties in terms of chemical inertness, mechanical strength, hardness as well as insulating and optical properties (Kong, 1981). Thin films of Alumina can be prepared by various deposition methods, e.g., evaporation, sputtering and also by direct anodic oxidation of aluminium surfaces (Fahy**,** 1979). 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 (Wright**,** 1979). In this paper, Aluminium oxide films are obtained by carefully performing anodic oxidation of aluminium.The surface roughness and growth structure of these films are studied using AFM and SEM techniques. The films are then treated in plasma of oxygen gas(Air plasma etching).The surface morphology of plasma etched films are again studied by AFM and SEM (Binning,1986).Difference in the surface studies of Aluminium thin film, before and after plasma etching, is noted down.

Experimental details and characterization of the films
Thin film processing can be divided into four parts: 1.Mechanical cleaning by using MgO paste.2.Chemical treatment of the substrate to remove contamination from the substrate surface.
3. Etching of the substrate surface in NaOH to remove any acidic impurities.4.Deposition of the thin film on the subtrate, by the process of anodic oxidation (Maduskar, 1986).

MATERIALS AND METHODS
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,1.5cm X 3.5cm ,1.5cm X 3.0cm & 1cm X 3.5 cm are cut from the pure sheet of aluminium.They 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.4.Anodisation of the clean Al surface was carried at room temperature with platinum wire as the cathode and polished aluminium plate as the anode.5.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.

In the present investigation, morphological studies were carried out using Atomic Force Microscope (AFM) (Binning, 1982). The two most commonly used modes of operation are Contact-mode and Tapping-mode. For the present work AFM was operated in contact mode. AFM gives more detailed information of surface topography in three dimensions. 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.

RESULTS AND DISCUSSION

Surface studies was carried out by AFM and SEM for all the three samples i.e,
Sample I: Untreated Aluminium sample (Pure Al plate)
Sample II: Aluminium oxide film formed on Al substrate by the process of anodic oxidation.
Sample III: Aluminium Oxide film etched in Air Plasma
The surfaces of these samples are again studied using SEM technique. Difference in the surface morphology of the Sample surfaces, before and after plasma etching, is noted down.

Table 1: Shows a summary of the parameters of the anodizing process and the AFM parameters, characterizing the surface roughness for all three samples including the parent Al substrate.

Sample	T(K)	I ($mAcm^{-2}$)	t(min)	RMS roughness (nm)
I	293	5.714	20	78.4071
II	303	5.111	20	88.6536
III	313	5.142	30	138.592

SEM (SCANNING ELECTRON MICROSCOPY)

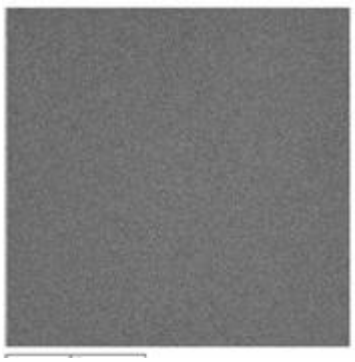


Figure 1: SEM image of untreated pure Aluminium plate at anodizing temperature T=293K

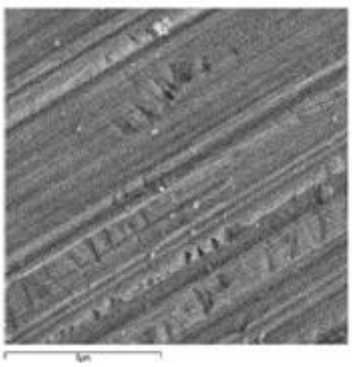


Figure 2: SEM image of Aluminium oxide film formed on Al substrate at anodizing temperature T=303 K by the process of anodic oxidation

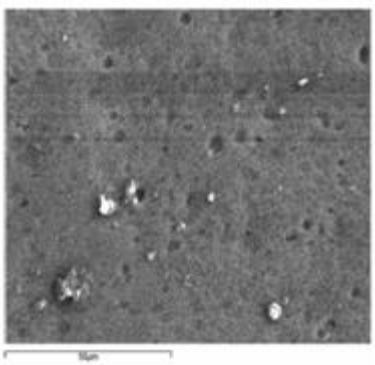


Figure 3: SEM image of Aluminium oxide film etched in air plasma at anodizing temperature T=313 K by the process of anodic oxidation

AFM (ATOMIC FORCE MICROSCOPY)

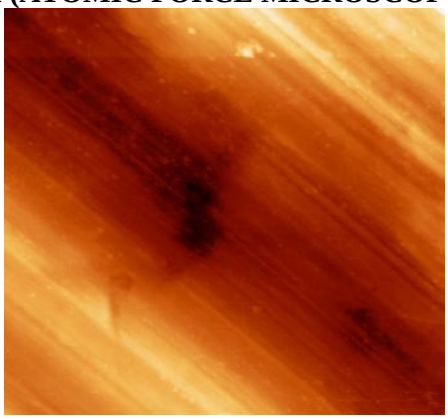
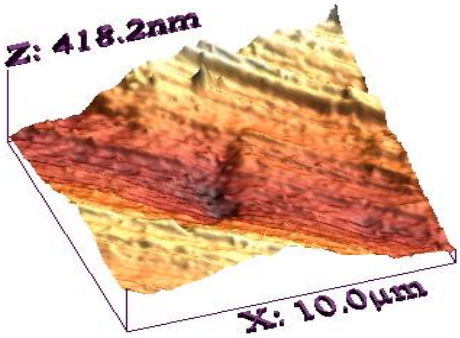


Figure 4:(i) AFM contact mode image



(ii) 3-D topography image of untreated pure Aluminium plate at anodizing temperature T=303 K

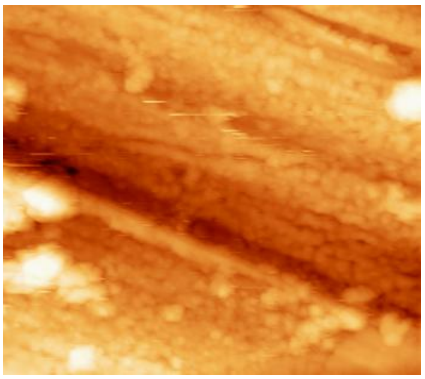
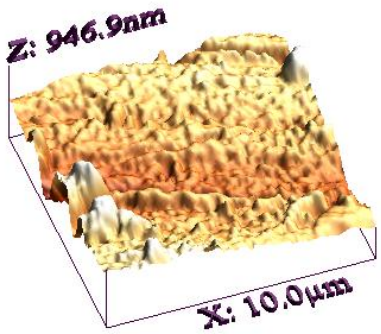


Figure 5: (i) AFM contact mode image



(ii) 3-D topography image of Aluminium oxide film formed on Al substrate at anodizing temperature T=303 K by the process of anodic oxidation

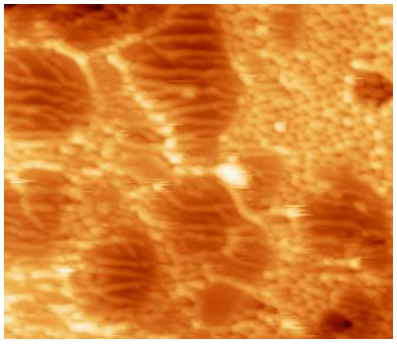
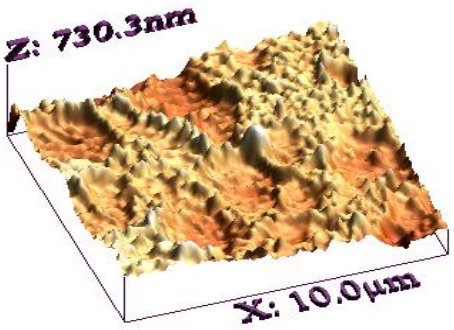


Figure 6: (i) AFM contact mode image



(ii) 3-D topography image of Aluminium Oxide film obtained at anodizing temperature T=313 K etched in Air plasma

The AFM contact technique was used to study the morphology and roughness of the alumina coating films produced by anodic oxidation. Differences in surface roughness and growth structure have been clearly observed in the AFM error signal and 3-dimensional topography images. Those differences can be

related to the parameters of the anodizing process. Oxide coatings appropriate for application were obtained under carefully selected deposition conditions, which ahead preparation of the films with special properties of the deposited oxide coating.

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