Atomic Force Microscopy of Electrodeposited Aluminum Oxide Thin Films


Abstract—Electrodeposition method is used for preparing alumina Al₂O₃ thin films for their applications as resistive coatings. DC voltage in the 1 – 5volts was used for film preparation. Reaction time is varied from 30mins to 90mins in order to observe the changes in structural and morphology of thin films. Presence of (222), (111) and (100) planes in XRD confirm the formation of FCC α-Al₂O₃ phase. The films are oriented preferentially along (222) plane. AFM results indicate the presence of uniformly distributed grains with smooth surface. Increase in deposition time from 30min to 90min increases the surface roughness, ~ 9 nm, of the films but the films are still quite smooth and have the potential to be utilized as thermal control coatings.

Keywords— Aluminum oxide, Atomic Force Microscope, Electrodeposition, Thin films.

I. INTRODUCTION

Aluminum has been used in aerospace sectors for more than seventy years. Recent advances in aluminum alloying made it best candidate for aircraft structures. Alloying adjustment and microstructure control improve the alloy’s performance. The 7000- series Al alloys are extensively used to increase pay load and fuel efficiency along with cost and weight requirement objectives due to high specific strength, low density and other valuable properties [1,2]. The high strength of aluminum 7075 is due to the fine and uniformly distributed precipitates and main strengthening phase. The films are oriented preferentially along (222) plane. AFM results indicate the presence of uniformly distributed grains with smooth surface. Increase in deposition time from 30min to 90min increases the surface roughness, ~ 9 nm, of the films but the films are still quite smooth and have the potential to be utilized as thermal control coatings.

II. EXPERIMENTAL DETAILS

All the reagents, used in this research work, were of analytical grade. Fresh neutral electrolytes were prepared for depositing aluminum oxide thin films. Electrolyte was de-aerated before each experiment. Electrodeposition was carried out in a three-electrode cell with control of the temperature and at a constant potential of 3V. However the electrodeposition time was varied as 30min, 60min and 90min. Vertical arrangement of the working electrode was used for the growth of anodized aluminum oxide thin films. A microcomputer-controlled Versa stat 4 microcomputer-controlled Versa stat 4
potentiostat/galvanostat was used for all the depositions. Aluminum oxide thin films were characterized structurally by using Rigaku D/MAX-IIA X-ray Diffractometer (CuKα = 1.5405 Å) operating at an accelerating potential of 30 kV and current of 22.5 mA. Surface morphology was studied by using Bruker CP-II atomic force microscope working in non-contact mode.

III. RESULTS AND DISCUSSIONS

Fig. 1 shows XRD plot of aluminum oxide thin film electrodeposited at 3 V for 90 min. XRD plots for rest of the samples electrodeposited for 30 min and 60 min are similar to the results presented in Fig. 1. Presence of (222), (100) and (111) peak confirms the formation of FCC Al2O3. Films are oriented preferentially along (222) plane. However, the peak intensity of (222) is much higher for films deposited for 30 min and 60 min.

Optical properties of electrodeposited alumina thin films were also studied using Variable Angle Spectroscopic Ellipsometer (VASE). A brief overview is discussed in this paper and details of optical analysis are reported elsewhere [11]. The band gap is found to be varied from 3.93 to 4.02 eV with increase in deposition time. The refractive index is 1.78 at a wavelength of 300 nm. Both optical and SEM results indicate the electrodeposited films are dense and have low porosity values. Thickness of around one micrometer is obtained using VASE of all the films [11].

Surface topography measurements were done using Atomic Force Microscope (AFM) operating in the non-contact mode. Fig. 2 show three dimensional (3-D) images of electrodeposited alumina thin films deposited at a constant potential of 3 V for 30 min, 60 min and 120 min. The films show smooth surface with few large grains. The grains are uniformly packed. No coalescence of grains is observed with change in the deposition time. However it can be seen that surface becomes rough with increase in deposition time from 30 min to 90 min at an applied potential of 3 V.

Fig. 3 (a), (b) and (c) show area profile of Al2O3 thin films electrodeposited for 30 min, 60 min and 90 min respectively. The root mean square roughness (Rrms) is 5.066 nm, 6.953 nm and 13.643 nm where as the average roughness (Rav) is 3.8 nm, 5.637 nm and 9.713 nm for films deposited at a constant potential of 3 V for 30 min, 60 min and 120 min respectively.

Rrms is plotted as a function of deposition time in Fig. 4 and it is seen that as the deposition time is increased from 30 min to 90 min the roughness of the films increases. These films are further intended to be utilized as thermal control coatings and low values of average and RMS roughness give the evidence of extremely smooth surface and low roughness of electrodeposited alumina thin films. The mean height for the films is 14.81 nm and 13.28 nm and 39.53 nm and median height is 14.11 nm, 12.13 nm and 37.33 nm with electrodeposition time of 30 min, 60 min and 120 min respectively.
Not only the three dimensional and area profile of electrodeposited alumina thin films taken using AFM is reported in this research work but also the films were analyzed for some particular positions. Fig. 5 show line profile for FCC Al₂O₃ thin films with change in deposition time form 30min to 90min. Line profiles images of AFM also indicate extremely smooth surface with very low $R_{rms}$ of 5.276nm, 5.279nm and 9.103nm for 30min, 60min and 120min electrodeposition deposition time respectively. However as seen in three dimensional AFM images (Fig. 2) that there were few large grains present on the surface of the films and these large grains results in high values of peak to valley ($R_{p-v}$) but otherwise the films are fairly uniform with uniformly distributed grains. $R_{p-v}$ varies from 24.83nm to 22.44nm and the increases to 51.15nm for 30min, 60min and 90min deposition time respectively.

![Fig. 4 Root mean square roughness ($R_{rms}$) and Mean height as a function of deposition time at a constant potential of 3V](image)

Fig. 3 Area profile for electrodeposited Al₂O₃ thin films (a) 30min (b) 60min (c) 90min.
IV. CONCLUSION

Aluminum oxide thin films are prepared using electrodeposition method at room temperature. The films are deposited at a constant DC voltage of 3V. However, the deposition time was varied from 30min to 60min and then to 90min to study the changes in deposition time on structural and morphological properties. Presence of (100), (111) and (222) planes indicate the formation of FCC alumina by electrodeposition. The films are oriented preferentially along (222) plane and peak intensity corresponding to (222) plane increases as the deposition time increases. Detailed AFM analysis of the films indicates that the films are composed of uniformly distributed grains and increasing the deposition time has a great impact on the topography of the films. Electrodeposited alumina thin films are fairly smooth and the average roughness is 3.8nm, 5.637nm and 9.713 nm for 30min, 60min and 90min electrodeposited Al$_2$O$_3$ thin films respectively.
REFERENCES


