Low Temperature Formation of Electrodeposited Aluminum Oxide Thin Films


Abstract— Aluminum oxide thin films are prepared at low temperature of 5°C. Electrodeposition method is used for deposition of aluminum oxide thin films. The deposition is carried out at voltage of 3V. Reaction time is varied from 30min to 120min to study the effect of variation in reaction time on structural and optical properties. XRD results indicate the formation of crystalline α-Al2O3. The predominant environmental factors influencing spacecrafts are to be addressed to increase their environment. The unique combination of ultra-lightness, high specific strength and quite good mechanical characteristics [3,4]. Whereas, low thermal conductance of aluminum alloys limit their applications to use directly in severe space structural applications in aerospace industry because of the hydroxides of aluminum [2].

Keywords—Aluminum oxide, Electrodeposition, Low Temperature, Optical.

I. INTRODUCTION

Among various materials of interest, Alumina (Al2O3, sapphire, aluminum oxide), is most widely studied and used materials in nuclear industry and aerospace industry as optic window [1]. Al2O3 offer unique advantages as high melting and boiling points, high dielectric constant, it’s chemically stability and its high strength. In addition to these properties its ability of being radiation resistive makes it an important candidate for in nuclear reactor as optic window [1]. Moreover, alumina exists in seven different polymorphs (α, β, γ, θ, η, κ, χ) [3] and it crystallizes in rhombohedral unit cell (D3d space group). However the most thermodynamically stable phase is α-Al2O3 and all other are transitional phases obtained during calcinations of Al2O3. J. A. Woollam Variable Angle Spectroscopic Elliposmter is used to study the optical properties. With increase in deposition time the thickness of the films increases. The refractive index of low temperature electrodeposited alumina thin films are in the range of 1.65-1.8. The bandgap is also affected by changes in deposition time and is in the range of 4.019-4.375eV.

II. EXPERIMENTAL DETAILS

Analytical grade reagents were used in this research work for electrodeposition. Microcomputer-controlled Versa stat 4 potentiostat/galvanostat was used for the deposition of alumina thin films. Aluminum alloy 7407 was used as the working electrode and is placed in vertical configuration. Electrolyte with neutral pH was used for the purpose of electrodeposition. Microcomputer-controlled Versa stat 4 potentiostat/galvanostat was used for the deposition of alumina thin films. Aluminum alloy 7407 was used as the working electrode and is placed in vertical configuration. Electrolyte with neutral pH was used for the purpose of electrodeposition. Electrodeposition was carried out using three electrode cells and electrolyte was de-aerated every time before the next deposition. The working electrode was vertically arranged in the electrolytic solution and the temperature of electrolyte was maintained at 5°C. Films are deposited at a constant potential of 3V and with variation in electrodeposition time from 30min to 120min with interval of 30min.

Before electrodeposition was carried out the aluminum alloy 7075 was ultrasonicated in acetone and isopropyl alcohol for 15minutes to remove the residual organic impurities.

Alumina thin films were the characterized structurally using Rigaku D/MAX-IIA X-ray Diffractometer (XRD). Copper was used as the target material with nickel filter to achieve monochromatic X-rays of λ=1.4505Å. The XRD is operated at a voltage of 30kV and current of 22.5mA. Optical properties of alumina thin films are studied using Variable Angle
Spectroscopic Ellipsometer (VASE) by J. A. Woollam.

III. RESULTS AND DISCUSSION

Fig. 1 show XRD pattern of alumina thin film deposited at a temperature of 5°C for 30min. The presence of (110), (006) and (117) peaks confirm the formation of FCC α-Al₂O₃. No preferred orientation is observed in case of the electrodeposited alumina thin films and the films are randomly oriented. XRD patterns of films deposited for 60min, 90min and 120min show similar results. However the peak intensities corresponding to (110) planes increases with increase in deposition time. In order to observe the effect of deposition temperature on alumina thin films the films were also deposited at room temperature. Detailed results of films deposited at room temperature are reported earlier [9] but it is worth mentioning here that the films deposited at room temperature under similar conditions show preferred orientation along (222).

VASE was also used to measure the thickness and surface roughness of the electrodeposited thin films. Thickness of the films increases from 50nm to 250nm with increase in deposition time form 30min to 120min and the surface roughness is in the range of 5.9nm to 15.395nm. The low values of surface roughness indicate that these films are well suited for its application as barrier coatings.

Fig. 2 show refractive index and extinction coefficient of electrodeposited alumina thin films prepared at a temperature of 5°C at 3V and the deposition time was 30min. Both refractive index and extinction coefficient decreases with increase in wavelength showing normal dispersion behavior. At a wavelength of 300nm the refractive index and extinction coefficient is 1.65 and 0.0836 respectively. With increase in deposition time from 30min to 120min the refractive index increases 1.8. The value of bulk refractive index is reported in literature to be around 1.82 and our values of refractive index close to bulk refractive index indicate that the films are dense with very little pores present in it. Refractive index can be used to calculate the porosity of films also. These porosity calculations require standard data. The standard data required in these studies is taken from material based models present in Variable Angle Spectroscopic Ellipsometry (VASE). The porosity values for the samples are found to be in the range of 0.5-20%. Sample 2 show lowest porosity values.

Another optical constant of interest is absorption coefficient (α). It is measure of the ability of the material to absorb photons. Absorption coefficient is determined using (1)

$$\alpha = \frac{4\pi k}{\lambda}$$

(1)

The absorption coefficient is related to Exciton energy (hν) and band energy as in 2:

$$(a_{hv})^{1/n} = A(h\nu - E_g)$$

(2)

Optical properties of electrodeposited alumina (Al₂O₃) films were studied using Variable Angle Spectroscopic Ellipsometry operated at an angle of incidence of 65° within the wavelength range of 200-1700nm. Not only the effect of increase in the deposition time is studied but also the effect of variation in thickness is also taken into consideration. A three layered model comprising of substrate, Al₂O₃ film and surface roughness is used. The two layered model excluding the surface roughness doesn’t give an appropriate fit with very high MSE (>10). We also introduced an interface layer between the film and substrate but it was found to have least impact on fitting quality and correspondingly on optical properties. Optical properties of films electrodeposited at room temperature have been reported earlier [9,10].
Where, \( h \) is the Planck’s constant, \( \nu \) the frequency of incident radiation and \( A \) is the constant. The factor \( n \) can be 1/2, 2 and 3/2 for direct and indirect allowed and direct forbidden transitions depending on the type of transition taking place in k-space. The \( \alpha^2 \) vs. \( E \) curve in Fig. show direct transition in radiated Al\(_2\)O\(_3\) thin films electrodeposited at 3V for 30min. the band gap of the film is 4.4eV. With increase in the deposition time the band gap decreases from 4.375eV to 4.019eV. Energy band gap values are in consistent to the values reported by Riaz et al. [11] for alumina thin films prepared by sol-gel technique.

![Fig. 2 Refracive index and extinction coefficient of electrodeposited alumina thin film](image)

**Fig. 2 Refracive index and extinction coefficient of electrodeposited alumina thin film**

IV. CONCLUSION

Alumina thin films are electrodeposited at a constant potential of 3V with varying the deposition time to 30min, 60min, 90min and 120min. All the depositions were carried out at low temperature of 5°C. Variable Angle Spectroscopic Ellipsometry results indicate that with increase in deposition time the refractive index increases as with increase in film thickness the films become dense. The refractive index values are in the range of 1.62 to 1.8. The band gap decreases from 4.4eV to 4.0eV with increase in deposition time.

![Fig. 3 Band gap of alumina thin film electrodeposited at 5°C](image)

**Fig. 3 Band gap of alumina thin film electrodeposited at 5°C**

REFERENCES