

DESIGNING AND MICRO CHARACTERIZATION OF METAL BASED TRILAYER THIN FILM DEVICE

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Aluminum based hafnium oxide (HfO₂/Al/HfO₂) multilayer thin film device prepared on a well cleaned commercial glass substrates through physical vapor deposition technology (electron beam evaporation). The film fabricated at a substrate temperature of 30 °C shows nano-polycrystals of HfO₂ surrounded in a amorphous lattice according to X-ray diffraction analysis. Surface characteristics of HfO₂/Al/HfO₂ nano layer device has been studied through atomic force microscopy (AFM) and the observations indicates a smooth surface obtained which is suitable for optical applications. A dual beam spectrophotometer was used to investigate the optical properties and from that data further determination of some optical constants like refractive index etc. Overall transmittance value varied from 80 to 85% and the % R found to be high up to 28% in the UV region to visible and near infrared regions at room temperature substrate.

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1. Introduction

Thin film coatings specially optical coating has been used widely in different industrial applications. When a metal mirror layer with high reflection is embedded between two dielectric layers, this dielectric/metal/dielectric (D/M/D) multilayer system can suppress the reflection from the metal layer in the visible region, and achieve a selective transparent effect. As the band gap value of hafnium oxide is higher, so due to this characteristics it provides a wide spectral range from ultraviolet to infrared ranges [1]. Due to its large band gap and high refractive index, HfO₂ is an interesting candidate for optical applications. HfO₂ is used as an optical coating for astronomically charged coupled devices[2], antireflective multilayer coating for night vision devices[3], high reflectivity mirrors and for IR optical devices[4,5]. The HfO₂ coatings have been mainly prepared by electron beam evaporation, atomic layer deposition, pulsed laser deposition, chemical vapor deposition and sputtering methods[6]. Aluminum is highly resistant to most forms of corrosion. Some valuable properties of this metals including high reflectivity, heat barrier properties and heat conduction. The metal is malleable and easily worked by the common manufacturing and shaping processes.

Present research work reports experimentally observed data on the structural and optical properties of nanostructured HfO₂/Al/HfO₂ thin films deposited by electron beam evaporation. These films were characterized using X-ray diffraction (XRD), atomic force microscopy (AFM) and Spectrophotometry.

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2. Experimental

The thin films were cleaned ultrasonically through an organized in proper way before deposition in the working chamber. A commercial glass substrate was used for the fabrication. The hafnium oxide and the reflecting material(Al) was kept in the graphite crucible. Then the electron beam has been focused upon these materials to deposit a tri layer structure as dielectric/metal/dielectric (D/M/D). A metal layer of thickness 5 nm grown as inter layer between the 10 nm hafnium oxide layer under vacuum ($>10^{-5}$ mbar) in electron-beam evaporator (BOS Edward AUTO500). The metal layer serves as the reflecting layer and the hafnium oxide as dielectric layer. The deposition has been carried out at Room temperature (30 °C). Crystal structure of these films was investigated through X-ray diffraction (XRD) patterns recorded at room temperature using PANalytical's X' Pert PRO diffractometer equipped with Cu K_{α} radiations in the 2θ range from 20–80°. The surface topographical modifications of thin multilayer films were investigated by Agilent 5100 atomic force microscopy (AFM) in contact mode. A sample area of (1x1 μm^2) was scanned by a silicon tip having radius in the range from 5–10 nm with a frequency of 0.5 Hz. Statistical analysis of AFM images and distribution of grain size (by the watershed technique) was carried out using Gwyddion software. The transmission and reflectance of these dielectric/metal/dielectric structured film observed using Perkin-Elmer Lambda 9 UV/Vis/NIR spectrophotometer. For reflectance measurements a separate assembly was inserted and % R was measured from both of the film (from coated and un-coated side).

Tables.1. Deposition conditions

Crucible	Graphite
Substrate	Commercial Glass
Total Sample Thickness	25 nm
Al-layer thickness	5 nm
HfO ₂ -layer thickness	10 nm
Chamber Pressure	4.90×10^{-5} mbar
Substrate Temperature	30° C

3. Results and Discussion

3.1 X-ray diffraction Analysis

The HfO₂ multilayer thin films deposited at 30 °C substrate temperatures with a total film thickness of ~ 25 nm on clean Commercial glass substrates was physically even and had very good adhesion to the substrates. Fig.1 shows XRD pattern for HfO₂/Al/HfO₂ tri-layer which depicts some weak diffraction peaks for HfO₂ system. A wider diffraction peak between 25 and 35° (involving diffraction peaks (011), (-111) and (020) for HfO₂) gives the informations about the amorphous/disordered nature of the deposited film [7]. Broadening of diffraction peaks signifies nanosize of the crystallites. However, the observed diffraction peaks have been indexed and identified using JCPDS ref. 06–0318 corresponding to HfO₂ monoclinic structure [8]. Presence of metallic intermediate layer provoked the crystallization of oxide (HfO₂) layer [9].

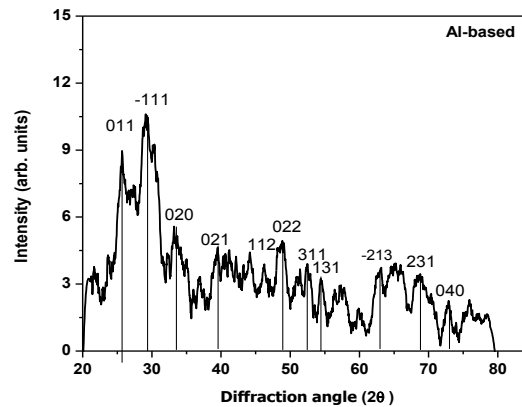


Fig.1: X-ray diffraction pattern for $\text{HfO}_2/\text{Al}/\text{HfO}_2$ multilayered films.

3.2 Surface Analysis

Fig. 2(a) show the two- and three-dimensional atomic force microscopic images of $\text{HfO}_2/\text{Al}/\text{HfO}_2$ with scanned area ($1\mu\text{m} \times 1\mu\text{m}$). Micrographs of this thin film composed of hafnium and aluminum having total thickness at 30°C substrate temperature indicates that this nano structure does not show regular and continuous long holes as seen in Fig.2(a). Such surfaces shows that these are somehow crack free surfaces. Such an irregular or random distribution of the grains suggested a random mechanisms of nucleation [10,11]. In order to study the quality of the surface of the thin films, root mean square roughness (RMS) is calculated about the surface under investigations. The AFM data measured using a soft ware (Gwyddion software) indicates a plain and smooth surface which may be suitable in the optical coating applications. The average roughness value which is measured is (R_a) ~ 3.71 and RMS value is (R_q) ~ 5.16 for this fabricated device film (a roughness plot also shown in Fig.2(b)). The height distribution plot shown in Fig.2(c) seems to be symmetric. Minimum value of skewness ($R_{sk} \sim 0.352$ nm) indicating that the peaks are slightly shows that peaks are slightly dominant on the surface, and low value of kurtosis ($R_{ku} \sim 0.657$) gives us the information about the distribution of grains over the targeted projected or scanned area as shown in table 2. The kurtosis map also drawn from the images shown in Fig.2(d).

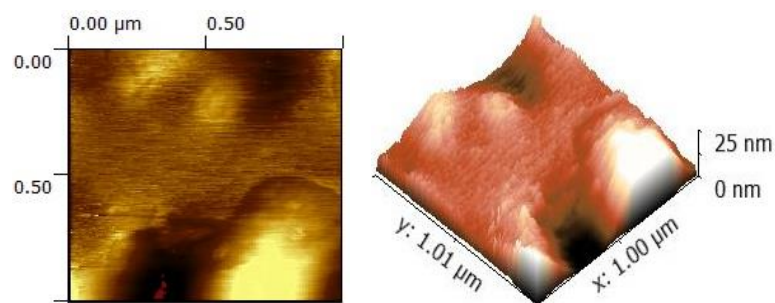


Fig. 2. (a) Two- and three-dimensional AFM images of $\text{HfO}_2/\text{Al}/\text{HfO}_2$ device with a scan area of $(1 \times 1) \mu\text{m}^2$

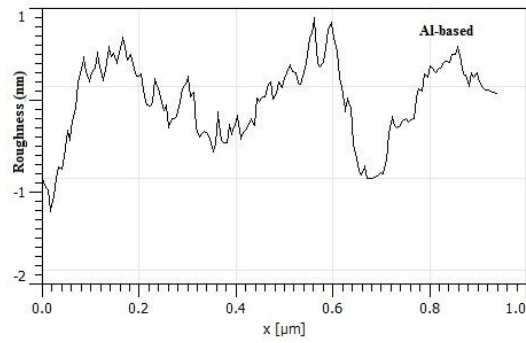


Fig.2: (b) The RMS roughness plots of $\text{HfO}_2/\text{Al}/\text{HfO}_2$ device as a function of position x

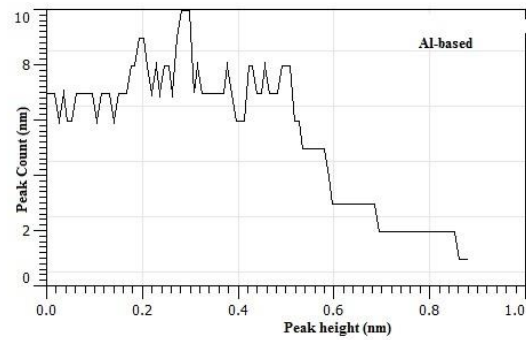


Fig. 2. (c) The height distribution showing peak count as a function of peak height determined from a scan area of $(1 \times 1) \mu\text{m}^2$

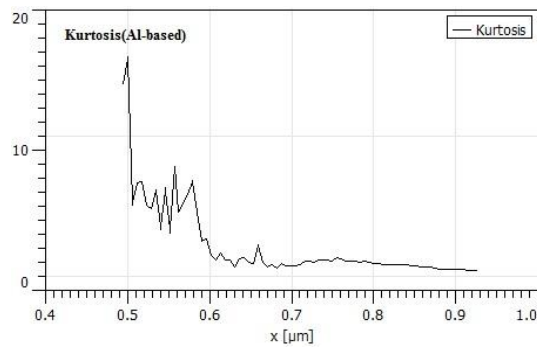


Fig.2. (d) The Kurtosis curves $\text{HfO}_2/\text{Al}/\text{HfO}_2$ device with a scan area of $(1 \times 1) \mu\text{m}^2$.

3.3 Optical Analysis

For optical analysis, a dual beam Perkin Elmer Lambda 9 spectrophotometer has been used to measure the parameters. The transmission and reflection has been measured by inserting separate assemblies in the spectrophotometer. The % reflectance has also been measured from both of coated and uncoated side of the thin film ($\text{HfO}_2/\text{Al}/\text{HfO}_2$) deposited by electron beam evaporation in the wavelength range 250 to 2500 nm. Fig.3 shows the % Transmittance and % Reflectance curves. The deposited thin film is found to highly transparent in the visible to near infrared regions as clearly seen from the plots. Almost the average transparency value is 80 to 85%. These highly transmitted regions are due to having more energy than the energy band gap of hafnium oxide. in some cases from the literatures was confirmed that with the increase in metal layer the transparent effect will reduced [12].

Reflectance is found to be high up to 28% in the UV region to Visible and near infrared regions at room temperature substrate. The %R variations are found to be low from the uncoated side of the film so using metal as an inter layer it might be served as a reflector layer which is further used in the energy efficient applications.

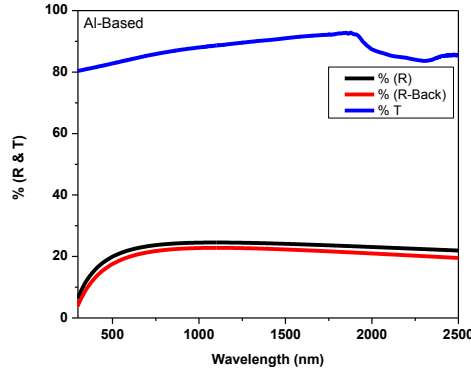


Fig.3: Spectral transmittance and reflectance (%R measured from coated and uncoated sides of the substrate) curves of $HfO_2/Al/HfO_2$ device

The absorbance of this nano device film ($HfO_2/Al/HfO_2$) has been calculated using the reflectance and transmittance data and shown in Fig.4. This is clearly seen from the plots that the absorbance values decreases from the visible to near infrared regions and then slightly increased in the infrared ranges. This increase in the absorption may be an indication about the formation of absorption band. Such curves and the fundamental absorption is well known to provide valuable information regarding optical energy gap, inter-band electronic transitions etc [13].

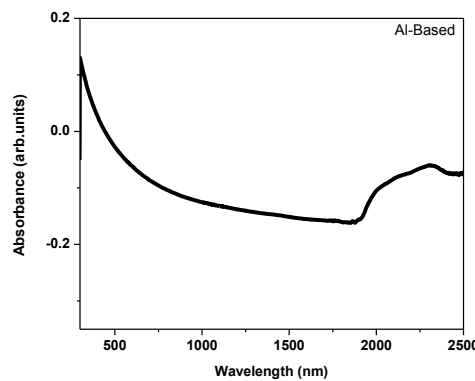


Fig. 4: Optical absorbance curves of $HfO_2/Al/HfO_2$ device

The % reflectance spectrum was used to determine the refractive index n of $HfO_2/Al/HfO_2$ multilayer thin films. Refractive indices depend upon wavelength of light passing through the medium and plot of refractive index as a function of wavelength for hafnium oxide nanostructures is shown in Fig.5. For normal incidence, refractive index n can be calculated by the following relation [8, 14]

$$n = \frac{1 + \sqrt{R}}{1 - \sqrt{R}} \quad (1)$$

The value of refractive index (n) determined from eq. (1) shows as periodic / oscillatory behavior with wavelength as shown in Fig.5. It raises in visible and then behave as constant in near infrared regions. This rise of n might be associated with increase in the density of our films [12].

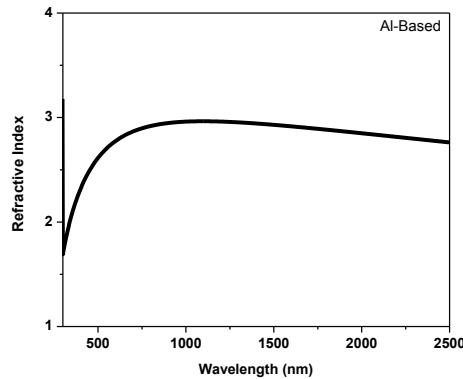


Fig. 5: Refractive Index plots for $\text{HfO}_2/\text{Al}/\text{HfO}_2$ thin nano films.

4. Conclusions

Thin film has been prepared using electron beam evaporation technique. Metal (Al) has been used as interlayer with HfO_2 to fabricate the three layer stack of $\text{HfO}_2/\text{Al}/\text{HfO}_2$. These nano layers has been characterized via X-ray diffraction technique for thier structural knowledge.

AFM has been used to study the surface properties of this device. It is observed that the smooth surface obtained after studding the statistical and amplitude parameters through AFM-software. Optical properties has been determined through optical spectrophotometer. The optical data was used to study the further optical constants,

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