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Morphology, Structural and Dielectric Properties of Vacuum Evaporated V$_2$O$_5$ Thin Films

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Abstract

Vanadium pentoxide (V$_2$O$_5$) thin films were deposited on well cleaned glass substrate using evaporation technique under the pressure of 10$^{-5}$ Torr. The thickness of the films was measured by the multiple beam interferometry technique and cross checked by using capacitance method. Metal-Insulator-Metal (MIM) structure was fabricated by using suitable masks to study dielectric properties. The dielectric properties were studied by employing LCR meter in the frequency range 12 Hz to 100 kHz for various temperatures. The temperature co-efficient of permittivity (TCP), temperature co-efficient of capacitance (TCC) and dielectric constant ($\varepsilon$) were calculated. The activation energy was calculated and found to be very low. The activation energy was found to be increasing with increase in frequency. The obtained low value of activation energy suggested that the hopping conduction may be due to electrons rather than ions.

Keywords: Thin films; Vacuum; Structural; Optical; band gap.

1. Introduction

Vanadium oxide (V$_2$O$_5$) and V$_2$O$_5$/Al$_2$O$_3$ mixed oxides have received much attention in recent years due to their optical properties, which make them interesting for various applications such as photocatalysis, gas sensors, as a window for solar cells, for electrochromic devices, color filters, reflectance mirrors, smart windows and surfaces with tunable emittance for temperature control of space vehicles and in the synthesis of nanocomposites [1, 2].

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Amorphous V$_2$O$_5$ is a potential candidate for the fabrication of thin-film batteries (TFBs) with high cycle performance and an excellent cycling stability when cycled in the same potential range [3]. Furthermore, V$_2$O$_5$ is an excellent catalyst due to the variety of vanadium oxidation states, ranging from 2$^+$ to 5$^+$, and the variability of oxygen coordination geometries [4]. Many studies have been carried out on V$_2$O$_5$ doped by different elements such as Ag, Cu, or Cr [5] in order to improve the discharge capacity of TFBs. Vanadium Pentoxide (V$_2$O$_5$) has been extensively studied since it tends to form a layered structure which allows the insertion / extraction of different ions between its layers. V$_2$O$_5$ undergoes a semiconductor-to-metal phase transition at 257± 5º C. A large change in electrical behaviour accompanies the phase change and thermally activated electrical switches have been fabricated from this material. Since optical and electrical behaviors are coupled, V$_2$O$_5$ may have potential use in optical switches and write-erase media as well [1]. V$_2$O$_5$ is especially interesting in thin film form because of the possibility of integration into microelectronics circuit [1]. Alternatively V$_2$O$_5$ films can be employed in conjunction with electrochromic tungsten oxide films in charge-balanced devices [2] for display purposes in informatics, for variable- reflectance mirrors, for variable-transmittance (smart) windows in energy- efficient buildings, and for variable emittance surfaces for temperature control of space vehicles. Recently, a considerable amount of work has been reported on structural, electrical and optical properties of nanostructured V$_2$O$_5$ and their devices [6-9]. Present work deals with the morphology and dielectric behavior of vacuum evaporated V$_2$O$_5$ thin films.

2. Experimental

V$_2$O$_5$ thin films were prepared by thermal evaporation method using Hind High Vacuum unit at a pressure of 10$^{-5}$ Torr. First the aluminum was evaporated from a tungsten filament on well-cleaned glass substrate (dimension 3.75X2.5cm) through suitable masks to form the base electrode. V$_2$O$_5$ was then evaporated from molybdenum boat to form the dielectric layer and then suitable masks were used to form the top electrode, so as to complete the aluminum- V$_2$O$_5$-aluminum capacitor structure. The vacuum evaporated V$_2$O$_5$ films on well-cleaned glass substrate were used for structural studies and surface analysis. A constant rate of evaporation (1Å/sec) was maintained to prepare all the V$_2$O$_5$ thin film samples. The thickness of the samples was measured by multiple beam interferometry (MBI) technique. The structural studies were carried out by X-ray diffraction technique in reflection mode with filtered Cuk$\alpha$ radiation ($\lambda$=1.5418Å) (The X-ray generator is Shiefert diffractometer, Germany). The surface morphological studies for V$_2$O$_5$ films have been carried out by scanning electron microscope (Stereo Scan LEO 440, Cambridge, and U.K). The capacitance and dielectric loss (tan $\delta$) for the V$_2$O$_5$ film were measured using a digital LCR meter (LCR-819, GW instek, Good will Instrument company Ltd., Taiwan).

3. Results and Discussion

3.1 Structure and surface morphology

Fig.1 shows the X-ray diffraction of V$_2$O$_5$ film of thickness 195 nm. The absence of any prominent peaks in the diffractogram revealed that V$_2$O$_5$ film possessed amorphous structures [10, 11]. Fig. 2 shows a typical SEM micrograph of V$_2$O$_5$ film of thickness 195 nm. It shows
that the surface appears to be not smooth without pits and pin holes were observed on the surface.

3.2 Dielectric properties

The capacitance and loss factor (tanδ) are important scale factors to analyse the dielectric properties. In the present study the equivalent series capacitance (C) was measured and whenever necessary a series–parallel conversion was made. The dielectric constant of V₂O₅ film was calculated using the formula $\varepsilon' = \frac{Cd}{\varepsilon_0 A}$. Where A is the area of the capacitor, d is the thickness of the dielectric layer, $\varepsilon'$ is the dielectric constant of the material and $\varepsilon_0$ is the permittivity of free space ($8.85 \times 10^{-12}$ F/m).
3.3 Frequency and temperature effect

Fig. 3 Change of capacitance with frequency

The changes in capacitance with frequency at different temperature for $V_2O_5$ film capacitor formed by vacuum evaporation are represented in fig.3. From the figure it is seen that the capacitance decreases with increase in frequency for all the temperatures investigated. It is observed that the rate of decrease is more in the lower frequency region compared to the higher
The decrease of capacitance (C) with increase of frequency is attributed to the trapping of charge carriers due to gap states density in the amorphous films [12].

The large increase in capacitance towards the low frequency region may be attributed to the blocking of charge carriers at the electrodes. Actually, the charge carriers present in the film migrate upon the application of the field and because of the impedance to their motion at electrodes resulted in space charge layer leads to a large increase in the capacitance at low frequencies. The observed decrease of capacitance with increasing frequency is also attributed to the increasing inability of the dipoles to orient themselves in a rapidly varying electric field and slow release of charge carriers from relatively deep traps. Increase of capacitance above room temperature is partly due to the expansion of the lattice and partly due to the excitation of charge carriers present at the imperfection sites [13, 14]. The observed results are in good agreement with the earlier reports on semiconductors. The variations of tanδ with frequency at different temperatures for V₂O₅ film are represented in the Fig.4. It is observed that the value of tanδ increase with frequency for all temperatures. It is found that the presence of loss peaks in the lower frequency region (12-800Hz) shifts to higher frequency region with increasing temperature. Generally the deficiency or the imperfection in the solid state materials pave way to form dipoles which leads to occurrence of Debye type dispersion [15] such a dispersion could be expected in vacuum evaporated V₂O₅ thin films.

![Fig. 5 Variation of dielectric constant with frequency](image)

Fig. 5 shows the variation of dielectric constant with frequency for various temperatures. The observed decrease of dielectric constant with increasing frequency may be due to the tendency of induced dipoles to orient themselves in the direction of the applied field. The following equation can explain the observed dependence of dielectric constant ε(ω) with frequency.

\[
ε (ω) = ε'' (ω) / tan δ = ε'' (ω) / (1/ ωRC + ωRC)
\]
Where the value of \( \varepsilon (\omega) \propto 1/\omega RC \) at low frequency and \( \varepsilon (\omega) \propto \omega RC \) at higher frequency. When \( \omega = 0 \), \( \varepsilon'' (\omega) \approx 0 \) then \( \varepsilon (\omega) = \varepsilon_s \) (static dielectric constant) and when \( \omega (\omega) \), \( \varepsilon'' (\omega) \approx \infty \) then \( \varepsilon (\omega) = \varepsilon_{\infty} \) (dielectric constant at optical frequency). The observed dielectric spectrum shows the weak polar nature of the V$_2$O$_5$ films. The increase of dielectric constant with temperature was due to an increase of total polarization arising from dipoles and trapped charge carriers [16]. It is seen that dielectric constant with frequency curve closely resemble those predicted by the Debye relaxation model for orientation polarization [15].

3.5 Temperature co-efficient of capacitance.

Fig. 6 illustrates the temperature dependence of capacitance for different frequencies. It has been observed that the capacitance increases with temperature for the frequency ranges studied.

![Variation of capacitance with temperatures](image_url)

The temperature co-efficient of capacitance has been evaluated using the equation 
\[
TCC = \gamma_c = 1/C(dC/dT)
\]
the estimated TCC for V$_2$O$_5$ films has been found to be 15620 ppm/K for 1 kHz. Fig.7 represents the temperature dependence of dielectric constant for different frequencies. The temperature co-efficient of permittivity TCP has been evaluated using the equation 
\[
TCP = \gamma_p = 1/\varepsilon(d\varepsilon/dT)
\]
and it is found to be 10600 ppm/K for 1 kHz.
The AC conduction, \( G_p = \omega C_p \tan \delta \) has been calculated at various temperatures from the measured values of capacitance and dissipation factor. From the Fig. 8 the activation energy was calculated for films of various frequencies and was presented in the Table 1. It is seen that the activation energy increases with increase in frequency. The low value of activation energy suggests that the hopping conduction may be in the prepared film was due to electrons rather than ions.
Table 1. Activation energy dependence on frequency

<table>
<thead>
<tr>
<th>Frequency</th>
<th>Activation Energy (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 Hz</td>
<td>0.1651</td>
</tr>
<tr>
<td>1 kHz</td>
<td>0.2102</td>
</tr>
<tr>
<td>100 kHz</td>
<td>0.27</td>
</tr>
</tbody>
</table>

4. Conclusion

In the present study, V$_2$O$_5$ thin films were deposited by thermal evaporation method. The XRD technique indicated that the film possesses the amorphous structure. The SEM analysis showed that no pits and pin holes were found on the surface. The dielectric study reveals that a polarization mechanism prevails in the film. The observed loss-peaks in the lower frequency region (12-800 Hz) shift to higher frequency region with increasing temperature confirm that Debye type polarization predominates in the thermally evaporated films. The activation energy was found to be very low, which increases with increase of frequency. The calculated low value of activation energy suggested that electronic hopping is responsible for conduction in the V$_2$O$_5$ thin films.

References