CHAPTER III
PREPARATION AND CHARACTERIZATION OF CdO THIN FILMS

This chapter gives a brief description of the experimental procedures, characterization methods employed for CdO thin films and discussion of the results.

The physical behaviour of thin films are strongly influenced by the stoichiometry, microstructure and the nature of the impurities and defects that are present. These parameters in turn depend on the type of deposition technique employed and the conditions maintained during the deposition process.

Thin films of CdO have been prepared by various techniques such as spray pyrolysis [1], magnetron sputtering [2], reactive sputtering [3-4], dc magnetron sputtering [5], and activated reactive evaporation [6]. In the present investigation, we have chosen spray pyrolysis and the activated reactive evaporation techniques for the preparation of CdO thin films.

PART I: PREPARATION AND CHARACTERIZATION OF SPRAYED CdO THIN FILMS

3.1.1. PREPARATION:

In spray pyrolysis, the film is deposited by spraying the solution onto a heated substrate, where the solution constituents react to form the desired compound. The
chemicals used for spray pyrolysis have to satisfy the following conditions:

i) the desired thin film material must be obtained as a result of thermally activated reaction between the various species/complexes in the spray solution, and

ii) the remainder of the constituents of the chemicals including the carrier liquid should be volatile at the spray temperature.

Growth of the films by spray pyrolysis is determined by the substrate temperature, flow rate of carrier gas, spray nozzle to substrate distance, droplet radius, solution concentration and flow rate.

Fig 3.1.1 shows a typical spraying system employed in the present study. It is provided with flow meters for both the carrier gas and the solution. The substrates kept on a metal block are heated by a kanthal heater and a chromel-alumel thermocouple senses the substrate temperature which in turn is controlled by a temperature controller. Purified air is used as the carrier gas. A random motion of the spray head or substrate or both are used for uniform deposition. The substrate temperature is a critical parameter in spray pyrolysis because the deposition efficiency decreases as the temperature increases above a
Fig. 3.1.1. Schematic diagram of the spray pyrolysis apparatus.
critical value. Spraying in pulses or bursts was employed to assure a reasonably constant substrate temperature. The sprayed droplet on reaching the substrate surface undergoes a pyrolytic reaction thereby forming the film, with the subsequent evaporation of the volatile by-products. The conditions that result in the maximum number of droplets having the size and momentum to land and flatten are critical. The impinging droplets, depending on their velocity and flow rate either flatten or skip or hover along the substrate surface. Concentration of the spraying solution is also an important parameter which determines the properties of the films.

In general, substrate surfaces get affected due to the chemical reaction in spray pyrolysis, and hence the choice is limited to glass, quartz, ceramics or oxide, nitride or carbide coated substrates. Metallic substrates are also found to be suitable for this purpose. The main advantages of spray pyrolysis are; it is inexpensive technique, large areas can be coated, and the cost of the material is low.

In the present study, cadmium oxide films were prepared by the spray pyrolysis technique onto ultrasonically cleaned Corning 7059 glass substrates using cadmium acetate [Cd (CH₃COO)₂·2H₂O] dissolved in methanol and water with the
volume ratio of 1:1. Vasu and Subrahmanyam [7] used water as oxidising agent, which also helps to dilute the spray solution containing ethanol, methanol, propanol and butyl alcohol.

The concentration of the spray solution used was 0.1 M, and it was found to be the optimum for producing CdO films with uniform thickness in a reasonable preparation time. The reaction that takes place at the substrate surface was,

\[
\text{Cd(CH}_3\text{COO)}_2 + \text{H}_2\text{O} \rightarrow \text{CdO} + 2 \text{CH}_3\text{COOH}.
\]

The spray nozzle to substrate distance was maintained at 30 cm. The substrates were maintained at temperatures, 100°C to 250°C. Compressed purified air was used as the carrier gas and the flow rate was fixed at 6 lt/min. The solution flow rate was maintained at 3 ml/min.

3.1.2. CHARACTERIZATION:

Cadmium oxide thin films prepared by spray pyrolysis were characterized by studying their structure, electrical and optical properties.

The spray deposited CdO films were pin-hole free, mechanically hard and strongly adherent to the substrate surface. The thickness of the films was determined using
microbalance (gravimetric method) and spectrophotometric method were in the range 0.8-1.0 µm.

Structural properties:

The crystallographic and microstructural features such as structure, crystallite size and orientation, and lattice parameters were determined using X-ray diffraction (XRD) and scanning electron microscope (SEM). The powder X-ray diffraction pattern of bulk CdO reported by Tanaka et al [8] is shown in fig.3.1.2. The XRD profile showed peaks which correspond to (111), (200), (220), (311), and (222) orientations respectively and the structure reported was cubic with (111) preferred orientation. The relative intensities, 'd' and (hkl) values reported are given in the table 3.1.1. The lattice parameter reported for bulk CdO was a = 4.66 Å.

The X-ray diffraction (XRD) spectra and scanning electron micrographs (SEM) of CdO films spray deposited at various substrate temperatures (T_s) are shown in figs.3.1.3 and 3.1.4 respectively. The films formed at T_s = 100°C were polycrystalline in nature exhibiting cubic structure with some amorphous background. As the deposition temperature increases, the amorphous background diminishes and the films are strongly oriented along (200)
Fig. 3.1.2. XRD spectrum of CdO powder
Table 3.1.1. The relative intensities, d’ and (hkl) values for CdO powder [8]

<table>
<thead>
<tr>
<th>(hkl)</th>
<th>Intensity</th>
<th>d values</th>
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<tr>
<td>(111)</td>
<td>100</td>
<td>2.71</td>
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<tr>
<td>(200)</td>
<td>88</td>
<td>2.35</td>
</tr>
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<td>43</td>
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<tr>
<td>(311)</td>
<td>28</td>
<td>1.41</td>
</tr>
<tr>
<td>(222)</td>
<td>13</td>
<td>1.315</td>
</tr>
</tbody>
</table>
Fig. 3.1.3. XRD spectra of sprayed CdO films formed at different temperatures
Fig. 3.14. Scanning electron micrographs of Sprayed films formed at different substrate temperatures.

$T_s = 250^\circ \text{C}$

$T_s = 200^\circ \text{C}$
direction normal to the substrate surface. However, when $T_s > 200^\circ C$, the preferred orientation of CdO films marginally reduced. Similar type of behaviour was observed by Chitra Agasha et al [9] in the case of sprayed SnO$_2$:F films. It is seen from the fig.3.1.3 that there is a slight change in the 'd' values when compared to CdO powder which may be probably due to the slight change in the concentration of oxygen vacancies. Chu and Chu [1] also observed a similar shift in spray deposited CdO films. The grain size was found to increase with increase in $T_s$, which is observed in all semiconducting films. The grain size of the CdO films evaluated from half intensity widths of the XRD peaks were in the range 150-200 nm. The films formed at temperatures around 100$^\circ C$ have smooth surface with smaller grain size while those deposited at 200$^\circ C$ have textured surface with large grain size. The structure of the CdO films formed at $T_s = 200^\circ C$ was cubic and the evaluated lattice parameter 'a' was 4.71 Å. The grain size evaluated from SEM is in accordance with that obtained from XRD.

**Electrical properties:**

The electrical resistivity ($\rho$), Hall mobility ($\mu$) and carrier density ($n$) of the spray deposited CdO films were
measured by the standard techniques. The resistivity of the films was mainly influenced by the deposition parameters. All the films exhibited n-type conductivity.

The film formed at substrate temperature, $T_s = 100^\circ C$, exhibited high resistivity with low mobility and carrier concentration as seen from fig.3.1.5. With increase in the substrate temperature, the growing films became homogeneous, exhibited low resistivity, and high mobility with better degree of crystallinity. A minimum resistivity of $2 \times 10^{-3}$ ohm-cm was observed in the films formed at $T_s = 200^\circ C$. For higher substrate temperatures ($T_s > 200^\circ C$), the resistivity was found to increase due to deficiency of cadmium. When $T_s > 250^\circ C$, the adhesion was poor. Fig.3.1.6 shows the effect of air flow rate on $\rho$, $\mu$ and $n$ of spray deposited CdO films formed at $T_s = 200^\circ C$. It is seen that the films prepared at an optimum air flow rate of 6 lt/min exhibited better electrical properties.

In order to study the effect of annealing on the electrical behaviour of CdO films prepared by the spray pyrolysis technique, the films were annealed at $400^\circ C$ for 30 min in different ambients, such as vacuum, air and hydrogen and the resistivity was measured at room temperature before and after annealing. No significant change in the
Fig. 3.1.5. Variation of Resistivity (●), Mobility (▲) and Carrier concentration (●) with substrate temperature for sprayed CdO films.
Fig. 3.1.6. Variation of Resistivity (○), Mobility (●) and Carrier density (△) with Air flow rate.
resistivity was observed when the films were annealed in different ambients.

In general the low resistivity of the pure transparent conducting oxide films is due to high carrier concentration and the observed mobility of these films are lower than that of the bulk materials. The stoichiometric deviation may be due to anion vacancies or excess interstitial cations. The observed temperature dependence of the mobility and carrier density can be explained in terms of the scattering mechanisms such as bulk scattering, grain boundary scattering and charged impurity scattering. The contribution of different scattering mechanisms are analysed using Matthiessen's rule [10].

The observed mobility ($\mu_{\text{obs}}$) can be written as

$$\left( \frac{1}{\mu_{\text{obs}}} \right) = \left( \frac{1}{\mu_b} \right) + \left( \frac{1}{\mu_{gb}} \right) + \left( \frac{1}{\mu_i} \right),$$

where $\mu_b$ is the mobility contribution due to bulk scattering such as lattice scattering, $\mu_{gb}$, the contribution due to grain boundary scattering and $\mu_i$, due to charged impurity scattering. The dependence of various mobility components on temperature ($T$) are given by the relations [11]:

$$\mu_b \propto T^{-3/2} ; \quad \mu_{gb} = \mu_0 \exp \left( \frac{e\phi_b}{kT} \right) \quad \text{and} \quad \mu_i \propto T^3/2,$$

where $\phi_b$ is the grain boundary potential.
In order to understand the scattering mechanism in the films, the temperature dependence of the Hall mobility was carried out in the temperature range 150-300 K. The variation of the Hall mobility with temperature is shown in fig.3.1.7. It is seen from the figure that $\mu \propto T^{-3/2}$, indicating the presence of bulk scattering.

Optical properties:

The optical absorption measurements were carried out using Hitachi U3400 UV-VIS-NIR double beam spectrophotometer and the optical band gap, refractive index and figure of merit were calculated.

The optical absorption studies revealed that the spray deposited CdO films showed high transmission in the visible region and high reflectance in the infrared region. By applying the theory of fundamental absorption of direct allowed transitions, the photon energy $h\nu$ can be expressed as a function of the absorption coefficient ($\alpha$) [12] as,

$$(\alpha h\nu) \propto (h\nu - E_g)^{1/2},$$

where $E_g$ is the band gap.

Fig.3.1.8 shows the variation of transmission with wavelength for spray deposited CdO films as a function of deposition temperature ($T_d$). At low deposition temperatures
Fig. 3.1.7. Temperature dependence of Hall mobility
Fig. 3.1.8. Variation of Transmission with Wavelength for sprayed CdO films with substrate temperature

Transmission (%) vs. Wavelength (nm)
(\(T_s = 100^\circ C\)), the films exhibited a low transmission of 40% and at higher deposition temperatures, the reaction between the solution constituents and the oxygen becomes more vigorous resulting in homogeneous films with improved transmission. The absorption edge was sharper for films deposited at \(T_s = 200^\circ C\) and was not well defined for films formed at temperatures above 200 \(^\circ C\). The plots of \((\alpha h\nu)^2\) vs. \(h\nu\) for the CdO films spray deposited at different substrate temperatures are shown in fig.3.1.9. The plots are linear indicating that the CdO films possess direct band gap. Extrapolating the linear portions to the energy axis gave the value of the band gap in the range 2.30 - 2.52 eV for CdO films prepared in the range \(T_s = 100^\circ C - 250^\circ C\). For CdO films formed at \(T_s = 200^\circ C\), the band gap was found to be 2.42 eV which is in good agreement with the reported value [1].

The refractive index of CdO films was calculated from the transmission and specular reflectance measurements using Hitachi U3400 UV-VIS-NIR double beam spectrophotometer. The variation of refractive index with wavelength is shown in fig.3.1.10. The refractive index was found to vary from 1.9 - 2.0 in the wavelength range investigated. The observed refractive index value is in good agreement with the reported value [13].
Fig. 3.1.9. Variation of $(\alpha h\nu)^2$ vs. $h\nu$ with substrate temperature for sprayed CdO films.
Fig. 3.1.10. Variation of Refractive index with Wavelength for sprayed CdO films.
The figure of merit ($\phi$) of a transparent conducting oxide (TC) is given by [14],

$$\phi_{TC} = \frac{T_r^{10}}{R_s},$$

where $T_r$ is the transmission at a particular wavelength and $R_s$ is the sheet resistance.

The wavelength dependence of the figure of merit of the spray deposited CdO films is shown in fig.3.1.11. It is seen that the figure of merit is maximum at 550 nm.

**PART II: PREPARATION AND CHARACTERIZATION OF ACTIVATED REACTIVE EVAPORATED CdO THIN FILMS**

3.2.1. PREPARATION:

The activated reactive evaporation (ARE) is a process used for preparing refractory compound films such as carbides, nitrides, oxides, sulphides and other related materials with high deposition rates. It is now classified as the plasma enhanced deposition process. In reactive evaporation, metal or alloy vapours are produced in the presence of a reactive gas, where the reaction between the metal and the gas molecules takes place by activating/ionizing both the metal and the gas atoms in the vapour phase.
Fig. 3.1.11. Variation of figure of merit of sprayed CdO film with wavelength.
If the evaporated material (either by resistive heating or by electron beam evaporation) is transported through the reactive gas plasma, the deposition technique is called activated reactive evaporation. In this technique, the reaction between the evaporated species and the gas is activated by establishing a thermionically assisted plasma in the reaction zone. The main difference between the reactive evaporation and activated reactive evaporation is the presence of a plasma in the latter case. As a result, in activated reactive evaporation, the vapour no longer consists of neutral atoms or molecules in their low energy states, but contains in addition, positive and negative ions as well as energetic neutrals of both the metal and gas constituents. The chemical characteristics and interactions with the plasma zone significantly influence the film composition and structure. A dense plasma can be generated by employing thoriated tungsten emitter and low voltage diode assembly. The reaction kinetics involved in reactive evaporation can be treated exactly in the same manner as the reactions in heterogeneous systems of condensed phases. The heterogeneous metallurgical reactions involve:

i) transport of the reactants to the reaction interface,

ii) occurrence of the chemical reaction at the reaction interface,
iii) transport of any reaction by-products away from the reaction interface,
iv) the nucleation and growth of the film, and
v) transfer of heat to or away from the reaction interface.

The possible steps associated with the deposition rate control in reactive evaporation are the adequate supply of reactants, adequate collision frequency between reactants, the rate of chemical reaction at the reaction interface, and the rate of removal of any undesired products away from the reaction interface. It may be noted that the physical location of the reactive interface may be the substrate surface, the evaporation source and the gas phase or a combination of these two.

CdO thin films are prepared by activated reactive evaporation method in which cadmium is evaporated through the glow discharge in the presence of pure oxygen. The electrodes that control the discharge in the vacuum chamber are independent of both the source and the substrate. The schematic diagram of the activated reactive evaporation technique employed in the present study is shown in fig.3.2.1. The activated reactive evaporation process parameters namely oxygen pressure, cadmium evaporation rate and the power in the glow discharge should be optimised.
Fig. 3.2.1. Schematic diagram of the activated reactive evaporation technique.

B - Boat
E₁,E₂ - Electrodes for discharge
H - Heater
N.V - Needle valve to admit oxygen
S.H - Substrate holder
With the proper control of these parameters, one can obtain CdO thin films with the desired properties.

In the present investigation thin films of CdO were prepared using 'Hind Hivac 12A4 high vacuum coating system' (fig.3.2.2). The substrates were kept at about 10 cm away from the evaporation source. The vacuum chamber was evacuated to a base pressure of $2 \times 10^{-6}$ Torr before admitting pure oxygen into the vacuum chamber. 5N pure cadmium was evaporated from a tantalum boat. The source temperature controlled the evaporation rate. The substrates were maintained at temperatures in the range 30°C - 300°C and the substrate temperature was measured using a copper-constantan thermocouple. Pure oxygen gas was admitted through the needle valve into the vacuum chamber until the desired pressure is reached. CdO thin films were deposited at various oxygen partial pressures in the range $10^{-4}$ to $10^{-2}$ Torr. The glow power was kept at 1.8 W and the source temperature was controlled depending on the oxygen partial pressure.

The activated reactive evaporated CdO films were pin-hole free, uniform and strongly adherent to the substrate surface. The thickness of the films was in the range 0.75 - 1.0 μm.
Fig. 3.2.2. Hind Hivac high vacuum coating system
3.2.2. Characterization:

Activated reactive evaporated CdO films were characterized by studying their structure, electrical and optical properties.

Structural properties:

The CdO films prepared by activated reactive evaporation showed (111), (200), (220), and (311) peaks, exhibited cubic structure irrespective of the changes in the substrate temperature. At low deposition temperatures ($T_s < 100^\circ\text{C}$), the films have amorphous background and are orientated along (111) direction. As the deposition temperature increased, the intensity of the preferred orientation increased up to $T_s = 200^\circ\text{C}$. The XRD profile along with the scanning electron micrograph of CdO film formed at $T_s = 200^\circ\text{C}$ are shown in figs.3.2.3 and 3.2.4 respectively. The evaluated lattice parameter, 4.68 Å, is in good agreement with the value of 4.69 Å reported on activated reactive evaporated CdO films by Girish Pathak and Rakeshlal [6].

In the present study, the observed change in the preferred orientation of CdO films [from (111) to (200)] formed by activated reactive evaporation and spray pyrolysis
Fig. 3.2.3. XRD spectrum of ARE CdO film formed at $T_s = 200^\circ$C
Fig. 3.2.4. Scanning Electron Micrograph of ARE films formed at $T_s = 200^\circ$C
respectively is believed to be due to the differences in the deposition parameters. This type of behaviour was also observed in other transparent conducting oxides like In$_2$O$_3$ [15].

Fig.3.2.5 shows the XRD of CdO films deposited $T_s = 200^\circ$C under different oxygen partial pressures. The films formed at low oxygen partial pressure ($1 \times 10^{-4}$ Torr) exhibited peaks at 31.8°, 34.8° and 47.8° which may be due to unreacted cadmium. With increase in oxygen partial pressure to $1 \times 10^{-3}$ Torr, the peaks corresponding to unreacted cadmium diminishes giving rise to CdO phase with (111) preferred orientation. A reduction in the crystallinity was observed at higher oxygen partial pressures ($> 5 \times 10^{-3}$ Torr).

**Electrical properties:**

The electrical behaviour of these films was influenced by the substrate temperature and oxygen partial pressure. The dependence of $\rho$, $\mu$ and $n$ with substrate temperature and oxygen partial pressure are shown in fig.3.2.6. The resistivity of the films formed at lower substrate temperatures ($T_s < 100^\circ$C) is very high. It decreases rapidly with increase in $T_s$ and gets saturated at a substrate temperature of about 250°C. The mobility and
XRD spectra of CdO films formed at different oxygen partial pressures

Fig. 3.2.5. XRD spectra of CdO films formed at different oxygen partial pressures
Fig. 3.2.6. Variation of Resistivity ($\bullet$), Mobility ($\bigodot$) and Carrier density ($\triangle$) for ARE films with substrate temperature.
carrier density also increase with increase in $T_s$ and saturate at $T_s = 250$ °C. This may probably be due to the perfect alignment of grains with increase in the deposition temperature.

It is seen from the fig.3.2.7 that the films formed at $T_s = 200$ °C with an oxygen partial pressure of $1 \times 10^{-3}$ Torr showed minimum resistivity. However, when oxygen partial pressure was $< 1 \times 10^{-3}$ Torr the resistivity was found to be high with low carrier density. This indicated that at low oxygen partial pressure ($< 1 \times 10^{-3}$ Torr) the film composition is dominated by unreacted cadmium and as the partial pressure increased, it gave rise to CdO phase which is in accordance with the XRD data. But above the optimum partial pressure of oxygen ($1 \times 10^{-3}$ Torr), the increase in resistivity is due to the decrease in the number of oxygen vacancies which reduce the carrier concentration. The increase in the mobility for the films prepared at the optimum oxygen partial pressure ($1 \times 10^{-3}$ Torr) suggest a consistent improvement in the film crystallinity, since oxygen can promote the growth of the crystallites.

Optical properties:

The transmission of activated reactive evaporated CdO films was found to depend critically on the substrate
Fig. 3.2.7. Variation of Resistivity (×), Mobility (□) and Carrier density (●) formed at different oxygen partial pressures.
temperature and oxygen partial pressure used during deposition. The low transmission observed at lower deposition temperatures \( T_s < 100^\circ C \) may be due to the non-stoichiometric film formation. The sharp fall in the absorption edge for the films formed at \( T_s = 200^\circ C \) indicated better crystallinity (fig.3.2.8). The evaluated band gap was about 2.40 eV for the films formed at 200°C (fig.3.2.9). The variation of transmission with oxygen partial pressure is shown in the table 3.2.1. The transmission is maximum in the films formed at an oxygen partial pressure of \( 1 \times 10^{-3} \) Torr. The decrease in transmission at an oxygen partial pressure higher than the optimum value \( (> 1 \times 10^{-3} \) Torr) is due to the increase in light scattering whereas at lower oxygen partial pressures the films are non-stoichiometric which leads to a decrease in transmission. Fig.3.2.10 shows \( (\omega h\nu)^2 \) vs. \( h\nu \) plots for CdO films formed at \( T_s = 200^\circ C \) under different oxygen partial pressures. The evaluated band gap was in the range, 2.32-2.52 eV depending on the oxygen partial pressure. The observed change is due to Moss-Burstein shift [16].

The refractive index of CdO films was calculated from the transmission and specular reflectance measurements using Hitachi U3400 UV-VIS-NIR double beam spectrophotometer. The variation of the refractive index with wavelength is shown
Fig. 3.2.8. Transmission vs Wavelength spectrum of CdO films
($T_b = 200^\circ$C by ARE)
Fig. 3.2.9. Plot of $(\alpha h \nu)^2$ against $h \nu$ for CdO film formed at $T_B = 200^\circ$C by ARE.
Table 3.2.1

Dependence of transmittance on the oxygen partial pressure in CdO films

<table>
<thead>
<tr>
<th>Partial Pressure of oxygen (Torr)</th>
<th>Transmission (%)</th>
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<tr>
<td>$2 \times 10^{-4}$</td>
<td>85</td>
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<tr>
<td>$1 \times 10^{-3}$</td>
<td>90</td>
</tr>
<tr>
<td>$5 \times 10^{-3}$</td>
<td>80</td>
</tr>
<tr>
<td>$1 \times 10^{-2}$</td>
<td>75</td>
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</tbody>
</table>
Fig. 3.2.10. Plots of \((\alpha h \nu)^2\) vs h\(\nu\) for the films formed at different oxygen partial pressures.
in fig.3.2.11. The refractive index was found to vary from 1.76 to 1.98 in the wavelength range investigated. The observed refractive index value is in good agreement with the reported value [14].

Fig.3.2.12 shows the wavelength dependence of the figure of merit ($\phi_{TC}$) for the films formed at $T_s = 200^\circ C$.

$\phi_{TC}$ of CdO films prepared by spray pyrolysis and the activated reactive evaporation techniques are comparable to the $\phi_{TC}$ of other transparent conducting thin films.

It is observed from the present investigation, that the activated reactive evaporated CdO films formed at $T_s = 200^\circ C$ with an oxygen of partial pressure of $1 \times 10^{-3}$ Torr were (111) oriented, showed a low resistivity, a high value of transparency over a wide wavelength range and a band gap of 2.4 eV, and are suitable as window material for solar energy applications.

Stability of CdO Films:

One of the problems that affect the efficiency of a solar cell is the stability of the absorber and window materials. Hence, an attempt was made to study the stability of the CdO films in the present investigation. The samples were kept at room temperature in air during the
Fig. 3.2.11. Variation of Refractive index with Wavelength for ARE CdO films.
Fig. 3.2.12. Variation of figure of merit of ARE CdO films with wavelength.
stability test. Fig.3.2.13 shows the resistivity of the films as a function of time for CdO films prepared by spray pyrolysis and activated reactive evaporation. It is seen that the films were stable even after 150 days of exposure.
Fig. 3.2.13. Stability of the films (SPRAYED and ARE) prepared at $T_s = 200^\circ C$
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