

Optical Properties of Chemical Bath Deposited Pb(OH)₂ Thin Film

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Abstract:-Lead hydroxide thin films were deposited on glass slide from aqueous solution of Pb(NO₃)₂ and NaOH by chemical bath deposition method. The films of various thicknesses have been obtained by varying the concentration (1M- 0.01M) of aqueous solution of TEA used as complexing agent (Tri Ethanolamine). Structural, optical properties and surface morphology of the deposited thin film have been studied by XRD, U.V spectrophotometer (Varian) and binocular research microscope vision 2000. The optical band gap of the Pb(OH)₂ thin film was found in the range of 2.59 to 3.38eV.

I. INTRODUCTION:-

Lead and lead compounds, as important industrial materials have wide applications. Such as in building construction, lead acid batteries, bullets weights and shot, part of solder, fusible, alloys glass industry and pigment. Lead hydroxide also has wide applications, such as staining and absorbance. The synthesis of Pb(OH)₂ nanostructures has rarely been reported probably because the stable presence of simple hydroxide Pb(OH)₂ in the solid state is doubtful.^[1-22]

In recent times, several techniques have been adopted for thin film deposition such as Sol-gel,^[4] ionized Cluster Beam Deposition,^[5] dc reactive magnetron, sputtering,^[6] pulse laser deposition,^[7] chemical bath deposition^[8-16] spray pyrolysis,^[17] plasma-enhanced chemical vapor deposition^[18-23], Solid state reaction method^[24-34], Combustion^[35-45] etc. Many of these methods are expensive and require high vacuum and controlled formation condition.

Another advantage of the CBD method over other method is that the film can be deposited at different shapes and size of substrates.

II. SYNTHESIS:-

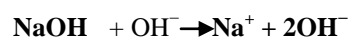
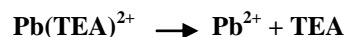
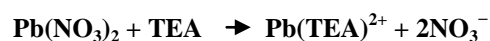
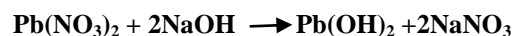
In our experiment the Pb(OH)₂ thin films were prepared by chemical bath technique at room temperature (28°C). The reaction bath is composed by Pb(NO₃)₂, NaOH and TEA (tri ethanolamine) used as complexing agent. For deposition of the film, commercial quality glass microscope slides of

dimension 16mm x 26mm x 1mm are used. Prior to use, these glass slides were soaked in aqua regia, a mixture of concentrated HCl and HNO₃ in the ratio of 3:1. They were removed after 24 hours and washed thoroughly in cold detergent solution, rinsed in triple distilled water and drip dried in air. The properly degreased and cleaned substrate surface has the advantage of producing highly adhesive and uniform film.

The substrate was immersed vertically at the centre of reaction bath in such a way that it should not touch the walls of the beaker. Only one concentration of Pb(NO₃)₂ (0.04M) and of NaOH (0.08M) were used in this method. Tri ethanolamine was used either directly or as aqueous solution with varying concentration of TEA to prepare various samples.

At the end of the dip period, the films are washed and drip-dried in hot air oven at 50°C.^[14] The optical absorbance, transmittance, and reflectance of the film were studied in the spectral range of 285-1000nm using UV spectrophotometer (Varian). Surface morphology observed by binocular research microscope vision 2000. Thickness is calculated by the formula.

$t = m/A * \rho$ Where m = mass, A = area, ρ = density. Reaction mechanics is as follow^[12, 13]



In direct use of TEA (sample 192) the color of the film was white but when we decrease the concentration of TEA in aqueous solution the whiteness of the deposition film slightly decreases, and their band gap also increases. In sample 192 (2.8ml of direct solution of TEA) we obtain band gap 2.4eV^[12] and transmittance between 25-35%. In sample 284 we used 8ml of 1mole aqueous solution of TEA, we obtain band gap 2.57 eV and transmittance between 40- 50%. In sample 326 and

330we used 8ml of 0.1and 0.01 mole aqueous solution of TEA respectively, we obtain band gap 2.98&3.38eV respectively, and transmittance between 50-80%.Higher concentration of TEA will increases the reaction rate, band gap decreases and vice verse. our previous paper also reported this type of results[19,20]. The deposition parameters and thickness of the film are shown in table - 1

Sample No.	Avg. thickness μm	Period of deposition (hrs)	Band gap eV
192	0.642	24	2.4
284	0.547	24	2.57
326	0.294	36	2.98
330	0.251	36	3.38

Table-1: deposition parameter and thickness of Pb(OH)₂ thin film

Figure 1and Figure 2, show the optical transmittance, reflectance, and absorption curve of deposited Pb(OH)₂ film of two thicknesses (0.294 μm,.251 μm). Figure 1 show that transmittance increases with decreasing concentration of the TEA and with decreasing thickness. Higher transmittance is observed in IR region. This indicates that film is a good material for warming applications in low temperate regions;it could be used as window glazing to create warmth in the house .It could also be very useful in Agriculture especially creating warmth for chicken in a poultry farm. On the other hand reflectance decreases rapidly with increase in wavelength falling from about 20%near UV region to about 12%near the infrared region.The band gap of the metallic oxide was determined by plotting $(\alpha h\nu)^2$ as a function of $h\nu$, and extrapolating the linear portion of the curve to $(\alpha h\nu)^2=0$ as shown in Figure-(3).The value obtained for the optical band gap is 2.89 and 3.38 eV.^[5]

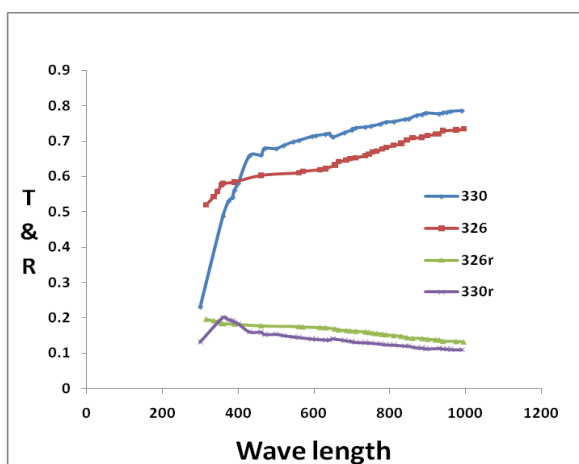


Fig.1. Transmittance & Reflectance as a Function of wave length

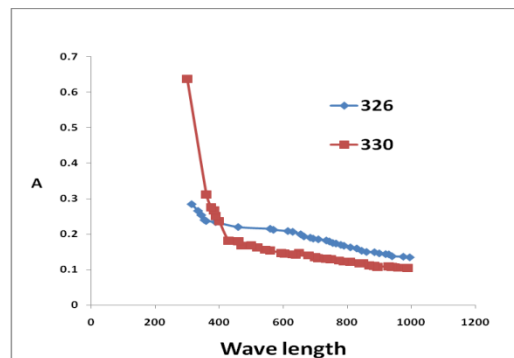


Fig.2. Absorbance as a function of wave length

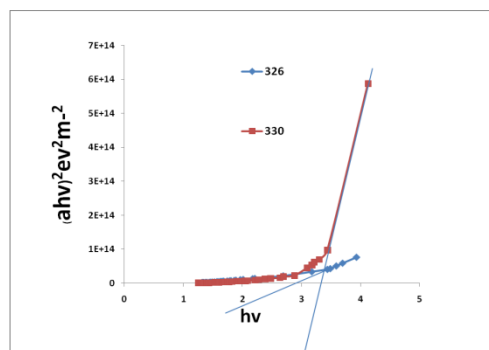


Fig.3. Plots of $(\alpha h\nu)^2$ eV^2m^{-2} against $h\nu$

Islam and Podder reported [21]. The absorption coefficient can be calculated by $\alpha = A/d$,

Where A is the optical absorbance and d is thickness of the film. The extinction coefficient can be obtain from the relation $= \alpha\lambda/4\pi$.

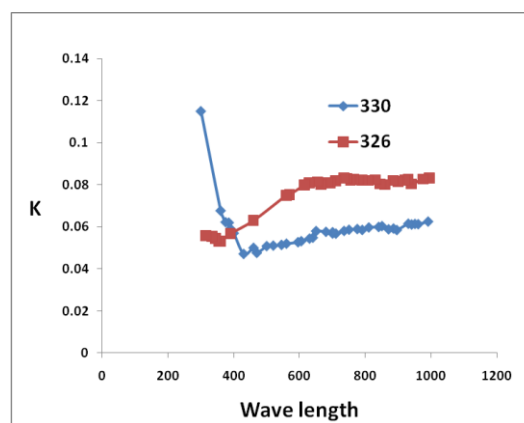


Fig.4. variation of extinction coefficient as a function of wave length

The refractive index (n) and extinction coefficient (K) value provide the optical properties of the film. The variation of extinction coefficient with wave length is shown in Figure (4). It is observed that the extinction coefficient decreases with the increase of the thickness of the film. The rise and fall in the extinction coefficient is directly related to the absorption of light.^[19] From Figure 4 it is clear that K decreases rapidly with increasing wavelength from 300 to 400 and after that value of K gradually increases.

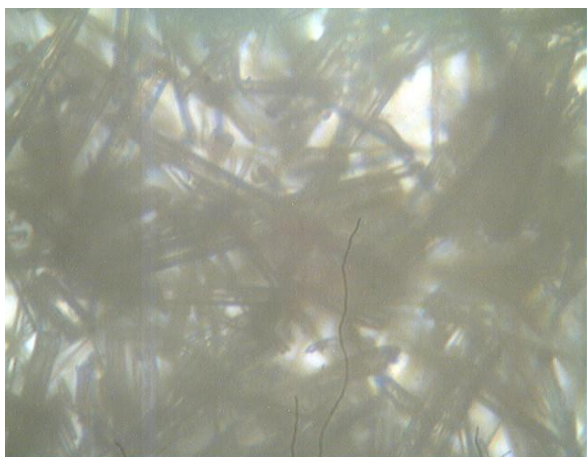
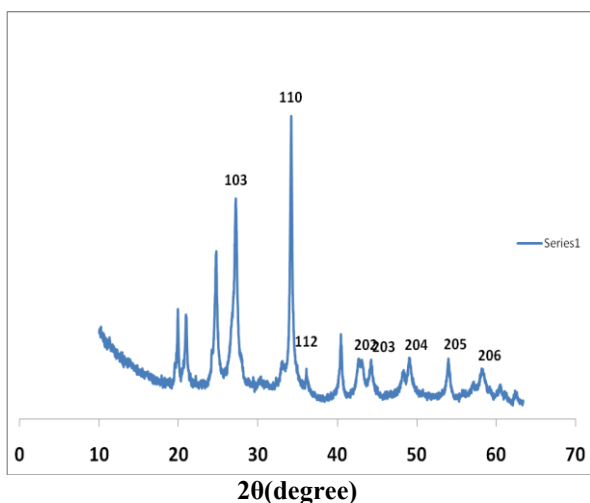


Fig.5.Sample 326



Fig .6. Sample330

To identify the crystal structure of the film, we performed XRD analysis. The XRD patterns were recorded with a D8 advance X-ray diffractometer using a Cu K_α radiation source ($\lambda=1.54056 \text{ \AA}$). Figure shows XRD pattern in the range of $20^\circ < 2\theta < 80^\circ$, most of the diffractin peaks can be assigned to Pb(OH)₂ according to re^[1,2] and file(00-0030607) the assigned indices in the XRD pattern. This suggest that the crystal structure of pb(OH)₂ is hexagonal.

Fig .7.X-ray Diffraction Pattern For Pb(OH)₂ Film

The grain size is caculated by debye scherrar's formula [2]

$$D=0.9\lambda/\beta\cos\theta$$

We found that the size of partical are in the nano range 10.49 to 25.76nm.

CONCLUSION:-

Pb(OH)₂ films have been successfully prepared by CBD method using Pb(NO₃)₂ and NaOH with TEA as complexing agent as well as alkali. Presence of alkali gives rod like morphology.. The film shows high transmittance in the visible/near infrared regions of electromagnetic spectrum. Refractive index is (2-2.68). Value of the real part of dielectric constant is 4-6.8. From XRD data we identify that structure of Pb(OH)₂ is hexagonal and size of particle is in the nano range.

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