



Refractive Index and Extinction Coefficient 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 Tri Ethanolamine used as complexing agent. Optical constant such as refractive index, extinction coefficient, real and imaginary parts of dielectric constant were evaluated from reflectance, transmittance and absorbance curve. The film show high transmittance in the visible/ near infrared region. Reflectance (10% to 20%), refractive index (2 - 2.68), extinction coefficient (0.047-0.083), real (4-6.8) and imaginary parts (0.22-.038) of dielectric constant are obtained in visible/ near infrared region.

I. INTRODUCTION:-

In recent years, one/two –dimensional nanostructures ^[1] such as nano wires , nano rods and nano tubes have attracted much attention under a background of research upsurge of nano science and nanotechnology due to their remarkable properties and potential application. Synthesis of one/two –dimensional nanostructures is highly important for investigating their properties and application. Therefore, developing new and simple method to synthesize nanostructures thin films/particles have become attractive topic ^[1-9].

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 stat reaction method^[24-34], Combustion^[35-45] etc. Many of these methods are expensive and require high vacuum and controlled formation condition. In recent times much interest has been generated around the CBD technique. The technique is simple cost; effective, reproducible and the material are readily available.

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.%. In sample 326 and 330 we used 8ml of 0.1 and 0.01 mole aqueous solution of TEA respectively, we obtain band gap 2.98 & 3.38 eV 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].

III. RESULT AND DISCUSSION:-

3.1 XRD Results:-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 (λ=1.54056 Å). Figure 1 shows XRD pattern in the range of 10°<2θ<60°, 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.According to the XRD results and ref ^[1,2] the structure of lead hydroxide is hexagonal with a=5.28Å,c=12.84Å and c/a=2.43.

The calculated spacing data are extracted using the formula of crystal plane distance for hexagonal crystals,

$$1/d^2 = \frac{4}{3} * \frac{(h^2+hk+k^2)}{a^2} + \frac{l^2}{c^2}$$

Where hkl is miller index and d is crystal plane distance.^[2]Our experimentally observed spacing data match the calculated data very well [Table 1]. This suggest that the crystal structure of $Pb(OH)_2$ is hexagonal.

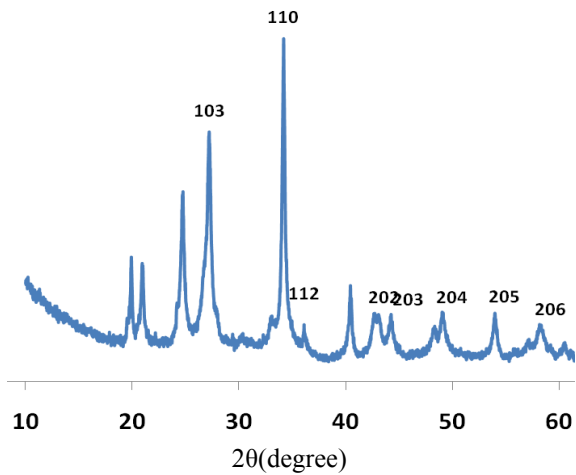


Fig .1. X-ray Diffraction Pattern For $Pb(OH)_2$ film

Table 1: Miller index , crystal plane distance and partical size for lead hydroxide

Li ne	I	Observe d Spacing (Å)	Assigne d Indices (hkl)	Calculated Spacing (Å)	Grain Size (nm) D
1	2094	3.25	103	3.23	10.49
2	2879	2.63	110	2.64	20.94
3	563	2.11	202	2.15	10.65
4	565	2.03	203	2.02	17.19
5	584	1.87	204	1.86	16.56
6	575	1.70	205	1.71	25.76
7	481	1.58	206	1.56	12.56

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

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

The particle size were found in the range of 10-30nm.

3.2 Microscopic Image:-

Figure 2 & 3 show the microscopio image of the prepared sample.

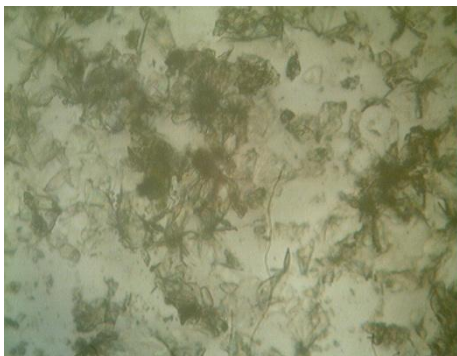


Fig.2. Sample 326



Fig. 3. Sample 330

3.3 Refractive index: -The refractive index has been calculated using the relation reported by Islam and Podder^[21]. The variation of refractive index with wave length for two thicknesses is shown in Figure 5. From fig. it is evident that refractive index between 2-2.68 in the visible/ near infrared region^[12,13,21], means that electromagnetic radiation is 2-2.68 time slower in the oxide films than in the free space and increases with the decrease in thickness of the film, which is in good agreement. In sample 192,284

We obtain value of refractive index 1.5-2.3 Low refractive indexes occurs due to successive internal reflection or due to the trapped photon energy with the grain boundary.

It is also attributed to the variety of impurities and defects with the increase of the thickness of the film and this implies that there is a strong optical scattering in the sample.^[19]

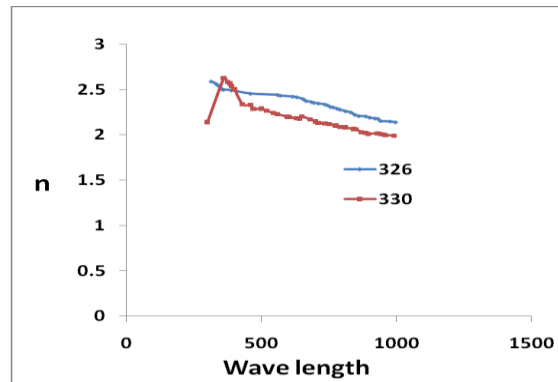


FIG. 4. Refractive index as a function of Wavelength

The real ϵ_r and imaginary ϵ_i parts of dielectric constant were determined using the formula $\epsilon_r = n^2 - k^2$ and $\epsilon_i = 2nk$. The variation of the real and imaginary parts of the dielectric constant for different film thickness is illustrated in Figure 6(a) and 6(b). The fig revealed that the value the real part (4-6.8) is higher than that of the imaginary part (0.22-0.38). From the optical data, it is observed that refractive index (n), extinction coefficient (k), and the real and imaginary parts of the dielectric constant follow the same pattern.

[12, 13, 21]

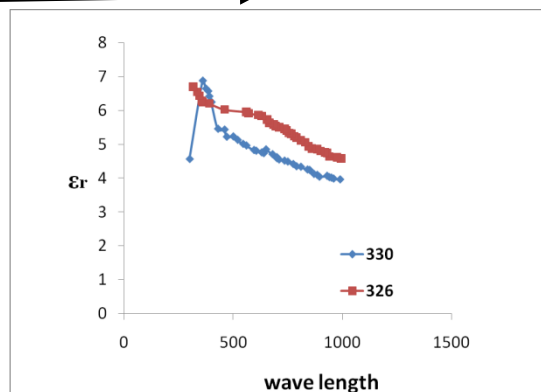
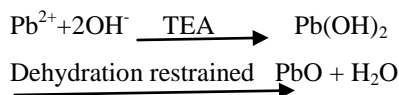


Fig.5(a). Variation of real part of dielectric constant

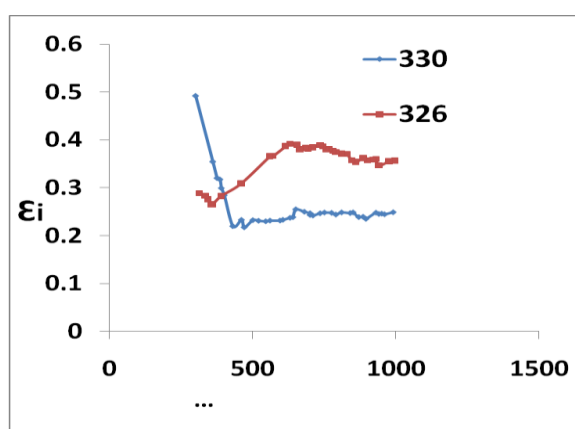


Fig.5(b). Variation of imaginary part of dielectric constant

In direct Use of TEA (sample192), we do not obtain too much rod, sometimes 2-3 rods are seen(Figure-07) , but if we use aqueous solution of,1M, 0.1M and 0.01M concentration of TEA, we obtain thick and thin rod, shown in figure-8 ,9,10. The OH⁻ concentration increased, the average aspect ratio (height /width) of Pb(OH)₂ nano particle,nanorod is decreased [4]. Cheng reported that chloride ions (alkali) are the key factor for the shape controlled synthesis of rod-like lead hydroxide or ZnO. In our Experiment TEA is also complexing element as well as alkali. By adding TEA ions, the dehydration of lead hydroxide could be effectively restrained and the related reaction formula is [1, 2]

IV. 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 band gap of the film is near about 3.38eV. 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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