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PROPERTIES OF CHEMICAL BATH DEPOSITED BERYLLIUM CHLORIDE THIN FILMS USING OPTICAL FIBRE

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ABSTRACT

Over the last two decades, the fiber optic technology has passed through many analytical stages. Some commercially available fiber optic sensors though in small quantities are being used for automation in mechanical and industrial environments. They are also used for instrumentation to measure and control various optical properties.

In the present work, an intensity modulated intrinsic fibre optic sensor is used to study various characteristic properties of beryllium chloride thin film coated micro-slides using optical fibre probes. Chemical bath deposition technique is used to grow thin films with the solutions of beryllium sulphate, Sodium hydroxide and potassium chloride. Sodium hydroxide serves as complexing agent. Beryllium sulphate acts as source for Be^{2+} ions and sodium chloride acts as source for Cl^- ions. Beryllium chloride films were grown at different deposition times and at different concentrations. Various optical characteristics are studied using fibre optic sensors operated at various wavelengths.

KEYWORDS :Chemical bath deposition, Beryllium Chloride thin films; Fiber optic sensor; Optical properties;



1.INTRODUCTION :

Compared with metal cables, optical fibres have very attractive features such as broad bandwidth, long distance transmission, no electromagnetic interference and sensing technology in many fields [1]. A short length of single mode optical fibre with its end faces coated to form an etalon or Fizeau interferometer [2] was used as temperature sensor. A fiber optic sensor with some portion of the clad removed in the middle was used by Mr. V. Sreehari babu, et al to study the properties of binary

solutions of benzene with methanol and ethanol [5].

The chemical bath deposition (CBD) also known as solution growth or chemical deposition (CD) technique has become very popular in recent decades, especially in the thin film formation due to its low cost since no sophisticated and expensive vacuum equipment are involved, ease of handling and also ease of applications to many compounds such as oxides, selenides, sulphides, etc. The advantages of CBD technique over other deposition techniques include: film deposition on all kinds of

hydrolytic substrates, simple and also inexpensive for large area deposition and easy control on growth of the films by controlling growth parameters like complexing agents, concentration of solutions, deposition time, etc. [3].

In the present work, beryllium chloride thin films are grown on micro glass slides using chemical bath deposition technique [4] and optical properties like transmittance, reflectance, absorbance; refractive index, absorption coefficient and extinction coefficient of these thin films are studied using optical fibres.

2. EXPERIMENT

a. Reagents used: Beryllium sulphate (> than 99% purity) is purchased from Aldrich. Sodium hydroxide, sodium chloride and nitric acid (> 98.5% purity) are purchased from Finar chemicals. All the chemicals are used without any further purification.

b. Coating thin films on micro glass slides: Prior to deposition of thin films on micro slides, the glass slides called substrates (25 x 76 x 1 mm) were degreased by sinking them in nitric acid for 48 hours, then removed from nitric acid, cleaned with detergent, distilled water and air dried. This creates nucleation centers on the substrates. Solutions for growth of thin films are prepared in three different baths with different concentrations of beryllium sulphate, sodium hydroxide, sodium chloride and distilled water. The bath parameters used in the present experiment are given below in the table 1.

Table 1: Bath parameters of various chemical solutions to grow BeCl₂ thin films

Reaction Bath	(BeSO ₄) _{2.4} H ₂ O		NaOH		NaCl		H ₂ O	pH	Deposition time (Hours)
	Moles M	Volume ml	Moles M	Volume ml	Moles M	Volume ml	Volume Ml		
B ₁	0.1	2	1	4	1	2	32	11.7	24
B ₂	0.5	5	1	10	1	5	20	11.8	24
B ₃	1.0	5	1	10	1	5	20	12.0	18

The glass substrates are placed vertically in the solution baths with the help of synthetic supporters. These baths are kept aside carefully in a closed container, so that the baths are not disturbed until deposition times given in the last column of the table 1. After completion of deposition times, the substrates on which thin film is deposited are taken out from the baths B1, B2 and B3 cleaned with distilled water, dried in air and then preserved in desiccators.

c. Experimental Procedure:

Three plastic (PMMA) fibres A, B and C of length 50cm each are considered for this experiment. The core diameter, core refractive index and numerical aperture of the fibres are 0.980mm, 1.497 and 0.50 respectively. One end of the fibre A is connected to 5mW, 632.8nm He-Ne laser (HNL050R). Remaining two fibres B and C are connected to power meters P1 and P2 [BD502A Handheld Optical Power Meter (-70~+10dBm) from Fiberstore industries, China] respectively. The fibres A, B and C are arranged as shown in figure 1, like displacement sensor with small gap between the fibres.

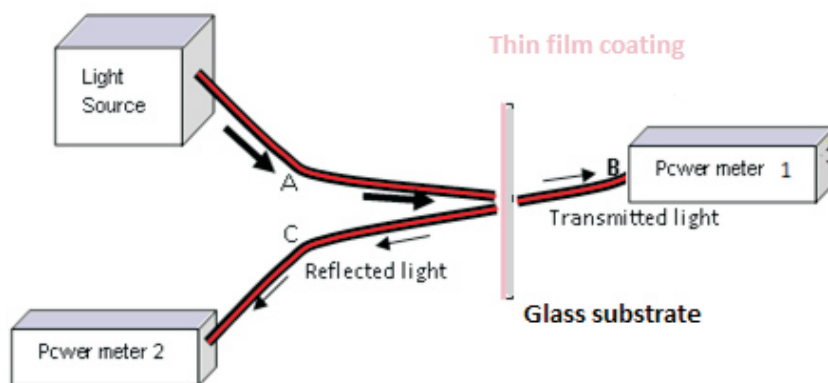


Figure 1: Diagram showing arrangement of fibre optic sensor to study transmittive and reflective properties of thin films

Firstly, one end of the fiber is connected to the 632.8nm light source and the other end is connected to the power meter P1 and the power transmitted through the fiber is measured. Now, a plane micro-glass slide is kept vertically between the fibres A and B. The incident light of wavelength 632.8nm from the source travels through the fibre A and incidents on the plane micro slide. This light partially gets transmitted through the micro slide and partially reflects back from the slide. The transmitted light from the slide is received by fibre B and the output power at the other end of the fibre B can be measured by power meter P1. The reflected light from the micro slide travels through the fibre C and output power in the fibre C can be measured by power meter P2.

The three fibres are aligned in such a way that the transmitted and the reflected powers become maximum. Now, the transmitted and reflected powers measured from the power meters P1 and P2 respectively are noted.

Now the plane micro slide is replaced by Beryllium Chloride thin film coated micro slide B1. The experiment is repeated with B1 slide for the same wavelength 632.8nm. The transmitted and reflected output powers are noted and recorded in table 2. The experiment is repeated with micro-slides B2 and B3 which have varying thin films grown with varying concentrations of solutions and growth time as given in table 1.

The whole experiment is repeated by replacing the 632.8nm wavelength light source by 850nm wavelength source using the same slides B₁, B₂ and B₃. The power transmitted, power reflected are measured from power meters P1 and P2 and recorded in table 3.

Using this data, the transmittance, reflectance, and absorbance are calculated. Refractive index and extinction coefficient are calculated from the following theoretical relations [6].

3.Theoretical considerations and calculations:

1) Transmittance , $T = \frac{I}{I_0}$, where I is the transmitted power and I_0 is the incident power.

Reflectance, $R = \frac{I_R}{I_0}$, where I_R is the reflected power and I_0 is the incident power.

2) Absorbance, $A = 1 - (T + R)$, where R is the reflectance.

3) Absorption coefficient, $\alpha = \frac{\ln\left(\frac{1}{T}\right)}{t}$, where t is the thickness of the film.

4) Refractive index, $n = \frac{1 + \sqrt{R}}{1 - \sqrt{R}}$.

5) Extinction coefficient, $k = \frac{\alpha \lambda}{4 \pi}$, where λ is wavelength of the light source.

Using the data from tables 2 and 3, the absorption coefficient α , refractive index of n material of thin film formed on the micro slides and extinction coefficient k are determined from the relations given above.

4.RESULTS AND DISCUSSION:

a.Thickness of the films: The thickness of thin film coating is measured by Surface profilometer. The thickness of the films grown in baths B₁, B₂ and B₃ are found to be 985nm, 994nm and 969nm respectively.

b.Optical Properties of BeCl₂ Thin films:

Table 2: Optical properties of BeCl₂ thin films at 632.8nm wavelength

Sam ple No	Refer ence Powe r μW	Power trans mitted I _T μW	Trans mittan ce T	Powe r refle cted I _R μW	Reflect ance R	Absor -bance A	Absor ption coeffi cient $\alpha \times 10^6$	Thick ness t nm	Refra ctive index n	Extinctio n coefficie nt $k \times 10^{-3}$
B ₁	4.91	4.57	0.930 (*0.94)	0.15	0.03 (*0.03)	0.038 (0.029)	0.070 4	987	1.41 9	3.71
B ₂	4.91	4.09	0.833 (*0.84)	0.45	0.092 (*0.09)	0.076 (*0.07)	0.184	954	1.87 0	9.665
B ₃	4.91	4.62	0.94 (*0.92)	0.25	0.051 (*0.06)	0.01 (*0.02)	0.061	959	1.57 6	3.196

*These are values from the literature [4], almost tallying with present experimental values

Table 3: Optical properties of BeCl₂ thin films at 850nm wavelength

Sam ple No	Refer ence Power μW	Power transm itted I_T μW	Trans mittan ce T	Powe r reflec ted I_R μW	Refle ctance R	Absor bance A	Abs orpti on coeff icien t $\alpha \times 10^6$	Thick ness t nm	Refra ctive index n	Extinct ion coeffic ient $k \times 10^{-3}$
B ₁	4.06	3.81	0.9384 (*0.93)	0.13	0.032 (*0.028)	0.0296 (*0.02)	0.064	984	1.4357	4.303
B ₂	4.06	3.57	0.8793 (*0.84)	0.31	0.0763 (*0.07)	0.0444 (*0.05)	0.129	955	1.7632	8.705
B ₃	4.06	3.62	0.8916 (*0.90)	0.28	0.0689 (*0.05)	0.0397 (*0.036)	0.114	959	1.706	7.765

*These are values from the literature [4], almost tallying with present experimental values.

c. X-Ray diffraction: The X-ray diffraction (XRD) pat terns for the films were recorded by using Rigaku D/MAX 2550 diffractometer with monochromator, Cu K α radiation for the identification of the crystal structures. The prominent intensity peaks at 2θ values of 15.35° , 23.849° , 33.786° , 33.895° , 39.948° and 53.249° that correspond to reflections from (1 1 2), (2 1 3), (2 2 4), (2 1 5), (4 1 1), (4 1 1) and (4 0 8), planes respectively of Body Centered Tetragonal Beryllium Chloride. These are in good agreement with (JCPDS 890300).

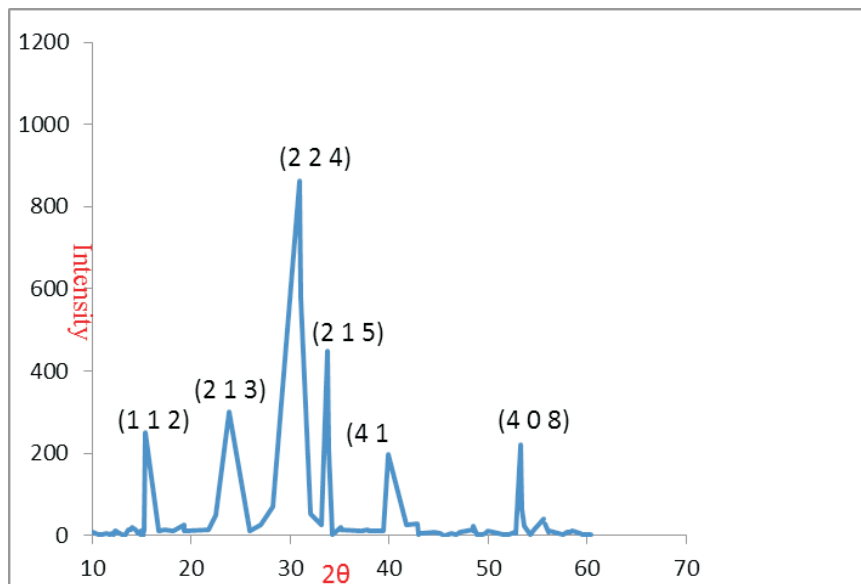


Figure 2: X-ray diffraction pattern of Beryllium chloride thin film grown

5. Comparison of experimental values with optical fibres and literature values:

The transmission and reflection characteristics of chemically deposited beryllium chloride thin films on the micro-slide were studied using optical fibre probes. The power transmitted and reflected by the thin films at various wavelengths are measured and compared with literature values as

measured with spectrometer studies.

Table 4: Transmittance, reflectance and absorbance of BeCl₂ thin films at 632.8nm wavelength: Literature vs Experiment

Sample No	Transmittance		Reflectance		Absorbance	
	Experimental value T	Literature value[4] A*	Experimental value R	Literature value[4] R *	Experimental value A	Literature value[4] A*
B ₁	0.930	0.94*	0.030	0.03*	0.038	0.029*
B ₂	0.833	0.84*	0.092	0.09*	0.076	0.07*
B ₃	0.94	0.92*	0.051	0.06*	0.01	0.02*

Table 5: Transmittance, reflectance and absorbance of BeCl₂ thin films at 850nm wavelength: Literature vs Experiment

Sample No	Transmittance		Reflectance		Absorbance	
	Experimental value T	Literature value[4] A*	Experimental value R	Literature value[4] R *	Experimental value A	Literature value[4] A*
B ₁	0.9384	0.93*	0.0320	0.028*	0.0296	0.02*
B ₂	0.8793	0.084*	0.0763	0.07*	0.0444	0.05*
B ₃	0.8916	0.90*	0.0687	0.05*	0.0397	0.036*

6.Future scope of the study: A device can be designed to measure the transmittance, reflectance, and absorbance, thickness of the thin films, refractive index and extinction coefficient with dedicated computer connected in place of power meters.

7.CONCLUSIONS

Beryllium chloride thin films with thickness 0.0955 to 0.0984µm are deposited on glass slides using chemical bath deposition technique. Their optical properties are studied using Optical fibre. They are found to be highly transparent in visible to NIR region. The transmittance is about 0.83 to 0.94 in visible to NIR region. Hence, they could be useful as thermal control window coatings for cold climates and also antireflection coatings. These thin films have very less reflectance about 0.03 to 0.07 in the same visible to NIR region. The absorbance of light energy is also very low in these thin films 0.02 to 0.05 for this region. It can be expected to have same trend in UV region also.

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