Optical Dielectric Thin Film Design in Hollow Glass Waveguides (HGWs) for Infrared Laser Delivery & Spectroscopy Applications

Carlos M. Bledt and James A. Harrington

Department of Materials Science & Engineering

Rutgers, the State University of New Jersey, Piscataway, NJ 08854, USA

Research Objectives

* To explore the theory of and deposit optically functional dielectric thin films in Hollow Glass Waveguides (HGWs) for low-loss delivery of infrared radiation at ranges between λ = 8 - 14 μm
* To create a model correlating the dependency of the optical response of dielectric coated HGWs on the chemical kinetics behind the relevant thin film deposition processes
* To optimize processing of dielectric thin film designs in HGWs for maximum transmission at desired wavelength(s) by altering dielectric thin film thickness as determined necessary

Optical Thin Films & High Reflection Coatings

- Functional optical dielectric (non-conducting) thin films below 1 μm in thickness have been widely used in optical systems and devices for enhancing or preventing reflectivity as desired for a given application
- High reflection coatings (HR) consisting of single or multiple dielectric thin films on an appropriate substrate allow for enhancement of reflective properties at desired incident by wavelengths by essentially creating dielectric mirrors
- The functionality of dielectric mirrors is based on thin film interference effects where the incident light upon the dielectric mirror reflects from the interfaces and exists the system in phase, resulting in constructive interference
- Thin film materials for HR coatings must be highly transparent at target wavelengths and have a mismatch in refractive index (n) between adjacent layers

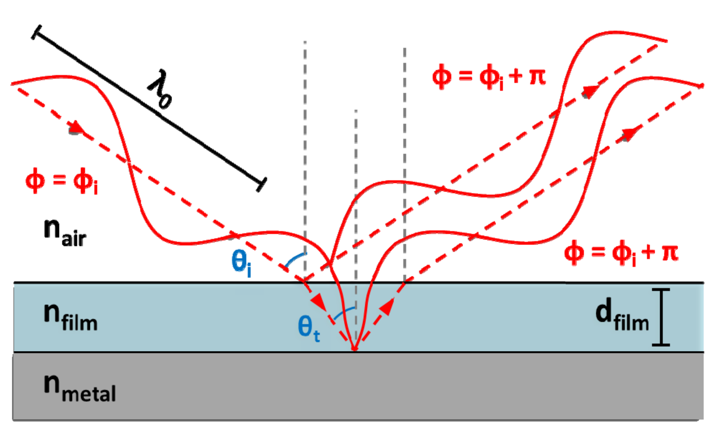
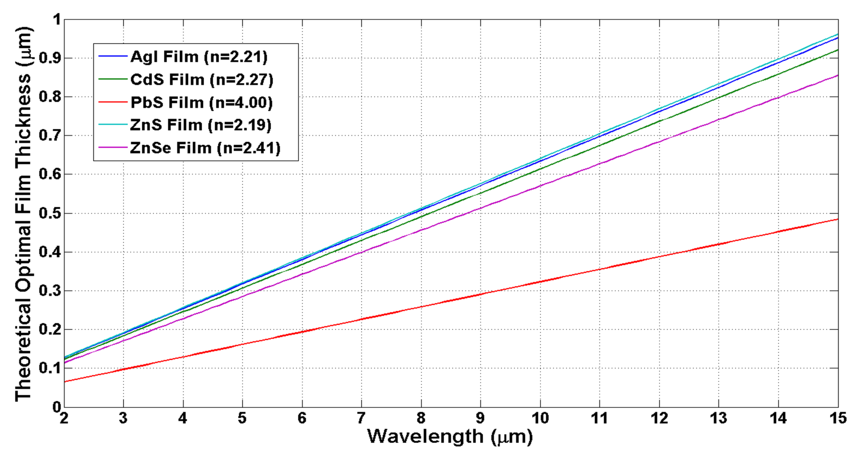


Diagram of Thin Film Interference Effects for HR Coatings with Quarter Wave Dielectric Film where $n_{air} > n_{film} > n_{metal}$

- Suitable infrared materials for HR thin films include highly infrared transparent materials such as silver iodide (AgI), cadmium sulfide (CdS), lead sulfide (PbS), zinc sulfide (ZnS), and zinc selenide (ZnSe)



Theoretical Reflectivity of Enhanced Silver Mirror Using Dielectric Thin Film Materials as a Function of λ with d_{film} Optimized for λ_0 = 10.6 μm

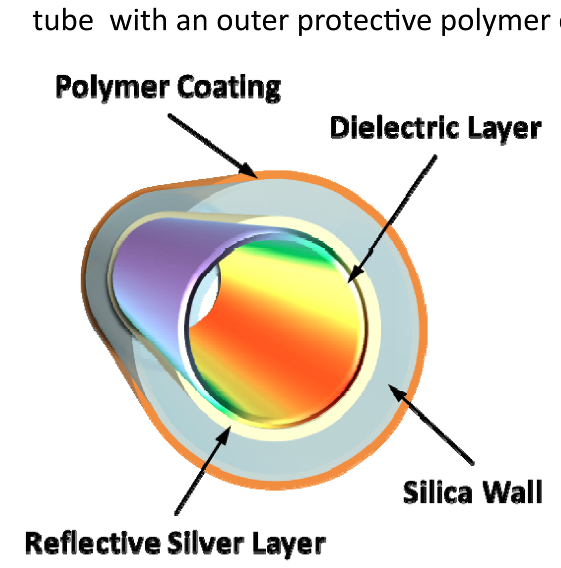
- The optimal thin film thickness (d_{film}) will depend on the refractive index of the material (n_{film}), incident angle (θ_i), and target wavelength (λ_0)
- The reflectance of the system over a range of wavelengths will then depend on d_{film}

$$d_{film} = \frac{1}{2n_{film}\cos\theta_t}\left(\frac{\lambda_0}{4}\right)$$

Equation for Determining Theoretical Optimal Film Thickness of Several IR Materials at Normal Incidence and λ_0 = 10.6 μm

Background on Hollow Glass Waveguides

- Hollow Glass Waveguides (HGWs) are an attractive alternative to solid core infrared (IR) fibers such as chalcogenide, fluoride glass, and oxide crystal fibers for low-loss transmission at essential infrared wavelengths [1]
- Advantages of HGWs over other IR waveguides include their broadband transmission and easily customizable properties including physical dimensionality and optical response through alteration of production parameters
- The basic non-functional physical structure of HGWs consists of a continuously drawn, high purity silica capillary tube with an outer protective polymer coating to prevent atmospheric degradation

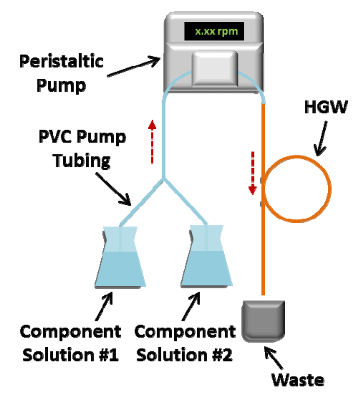


Cross-Sectional Diagram of a Single Layer Coated Silver Based Hollow Glass Waveguide

- The optically functional structure of HGWs consists of a highly reflective silver thin film deposited on the inner surface of the glass capillary upon which subsequent dielectric thin films of IR transparent materials such as AgI, CdS, PbS, ZnS, ZnSe, and PS are deposited to enhance reflectivity based on thin film effects
- Due to the ability to deposit dielectric thin films of varying thicknesses via aqueous solution deposition, HGWs can be tailored for maximum reflectivity, and thus maximum transmission, at a certain wavelength range as desired for a given application
- Furthermore, the low-loss wide broadband λ range of HGWs due to thin film interference effects, along with high incident power threshold and low production costs make HGWs highly attractive

Production of Hollow Glass Waveguides

- Functionally optical thin films in HGWs are deposited via Dynamic Liquid Phase Deposition (DLPD) techniques involving deposition of desired materials from precursor species containing aqueous solution(s)



System Configuration for DLPD Processes in HGWs

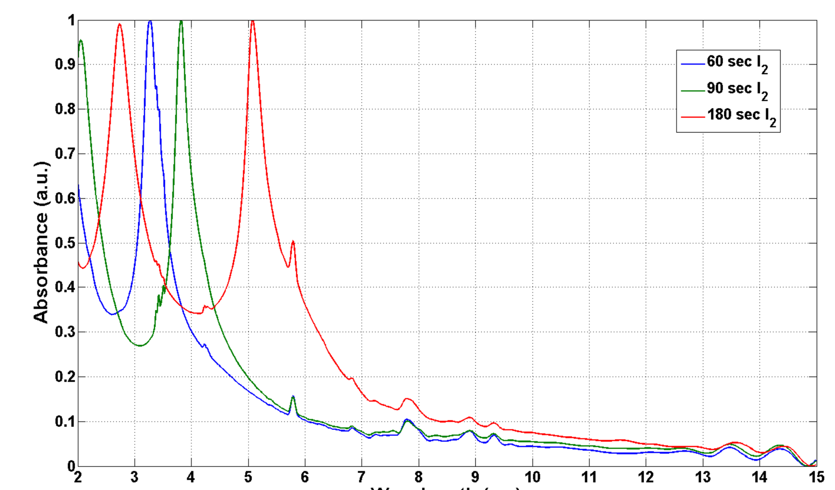
- Low surface roughness, highly reflective silver thin films are deposited via reduction of silver-diammine complexes to metallic silver by organic reducing agents in solution
- Dielectric thin films are then deposited on the reflective silver layer thin film substrate through different reactions/mechanism via DLPD techniques at different flow rates
- The choice of dielectric thin film to be used is determined by the desired optical response as well as factors such as deposition ability and durability of the material
- Understanding of chemical deposition processes is necessary for success
- Correct optimal thin film thickness, good uniformity of deposited films, and low film surface roughness are essential for exceptional HGW functionality at any wavelength
- Variables affecting the manufacturing of HGWs include solution concentrations, deposition time, and fluid flow
- Present study involved 1,000 μm ID HGW samples with flow rates of 11.8 (AgI), 22.6 (CdS), and 16.8 (PbS) mL/min

Spectral Characterization of Deposited Thin Films

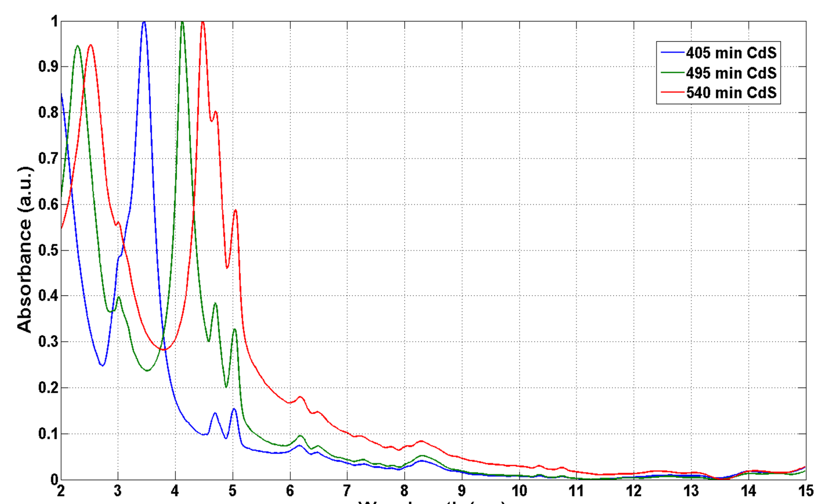
- Successful and reproducible optically functional dielectric thin films of AgI, CdS, and PbS have thus far been deposited in HGWs for both consistent spectroscopic analysis and IR radiation propagation properties
- Spectral analysis of HGW samples involved Fourier-Transform Infrared (FTIR) spectroscopy utilizing a Thermo Nicolet Protégé 460 FTIR spectrometer in conjunction with cryogenic Teledyne InSb and MCT/A IR detectors

Silver Iodide (AgI) Thin Films

- Iodine solution: 39.40 mM I_2 in cyclohexane
- Silver iodide (AgI) dielectric thin films are produced through the subtractive iodization (conversion) of pre-deposited Ag innermost layers in HGWs to AgI
- As a result, formation kinetics of AgI dielectric thin films on Ag substrate are mass transport limited
- AgI HGWs were manufactured at iodization times from 60 to 240 seconds in 30 second intervals



FTIR Spectra of Ag/AgI HGW Samples at Various Iodization Times



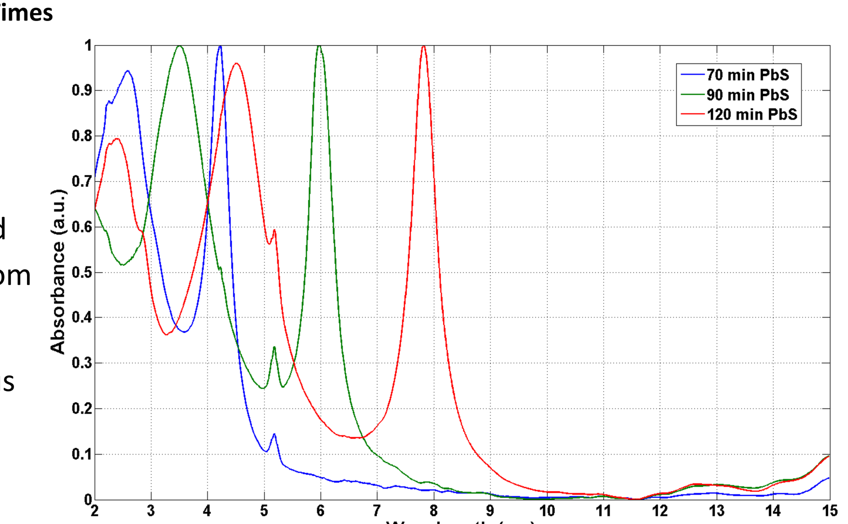
FTIR Spectra of Ag/CdS HGW Samples at Various Deposition Times

Cadmium Sulfide (CdS) Thin Films

- $Cd(NO_3)_2$ Sol: 6.39 mM / $CS(NH_2)_2$ Sol: 41.62 mM
- Cadmium Sulfide (CdS) dielectric thin films are produced through the additive chemical deposition of CdS from solutions in compatible substrate
- Kinetics of CdS film growth limited by homogeneous and heterogeneous nucleation and growth
- Ag/CdS HGWs were manufactured at deposition times from 450 to 720 minutes in 45 minute intervals

Lead Sulfide (PbS) Thin Films

- $Pb(NO_3)_2$ Sol: 3.02 mM / $CS(NH_2)_2$ Sol: 19.70 mM
- Lead Sulfide (PbS) dielectric thin films are produced through the additive chemical deposition of PbS from solutions in compatible substrate
- Kinetics of PbS film growth limited by homogeneous and heterogeneous nucleation and growth
- Ag/PbS HGWs were manufactured at deposition times from 60 to 135 minutes in 15 minute intervals



FTIR Spectra of Ag/PbS HGW Samples at Various Deposition Times

- Analysis of spectral response for deposited thin films as a function of deposition time yielded thin film growth kinetics in HGWs via DLPD processes and allowed for optimization of dielectric HGWs for use at λ = 10.6 μm

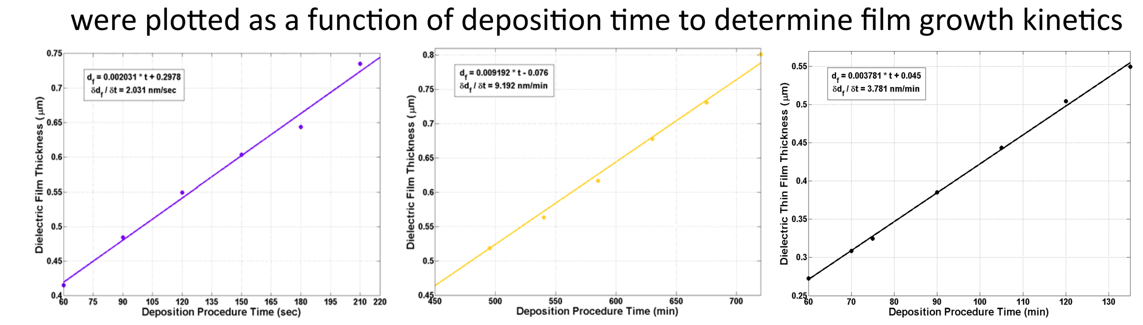
Deposition Kinetics-Optical Response Model

- Analysis of resulting spectra allows for determination of film thickness as function of deposition procedure time
- Thin film thickness can be calculated from wavelength corresponding to first interference peak position (λ_c) and refractive index (n_s) by using Miyagi's formula [1]

$$d_f = \frac{\lambda_c}{4\sqrt{n_s^2 - 1}}$$

Dielectric Thin Film Equation

- Thin film thicknesses for all deposited thin films as derived from spectral analysis were plotted as a function of deposition time to determine film growth kinetics



Film Thickness as a Function of Deposition Time for a) Ag/AgI, b) Ag/CdS, and c) Ag/PbS HGWs

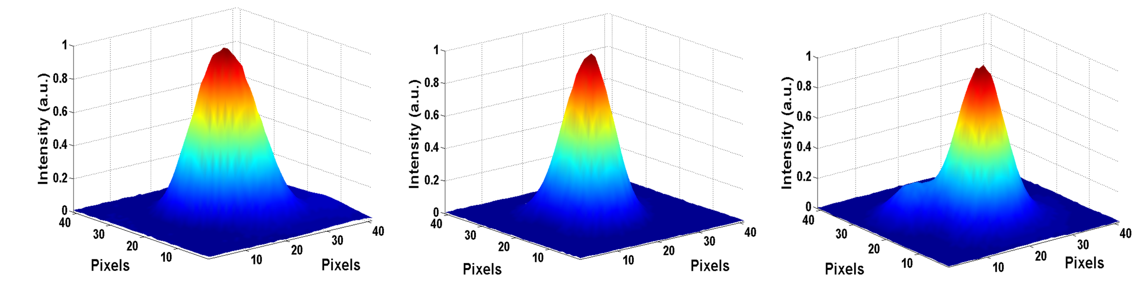
- Analysis of the kinetics of deposition procedures revealed linear film growth
- Good film uniformity seen
- Analysis allowed for HGW optimization at λ = 10.6 μm
- Film growth rates determined to be; 2.03 nm/sec for AgI, 9.19 nm/min CdS, and 3.78 nm/min for PbS

Optical Attenuation in HGWs

- Attenuation measurements and spatial profiles were taken using a Laser Engineering Carbon Dioxide laser 50 Watt maximum output power laser as an IR source emitting at a wavelength of λ = 10.6 μm
- Cutback methodology was utilized for all attenuation measurements to prevent possibility of error due to coupling inefficiencies
- Dielectric thin films seen to greatly reduce attenuation in HGWs

	d_{film} (μm)	α (dB/m)	Δα (dB/m)
Ag Only	N/A	3.823	N/A
Ag/AgI	0.384	0.057	-3.766
Ag/CdS	0.496	0.097	-3.734
Ag/PbS	0.233	0.041	-3.782

Calculated Film Thicknesses, Attenuation Coefficients, and Difference in α from Ag Only



Near Single Mode Spatial TEM Optical Profiles of a) Ag/AgI, b) Ag/CdS, and c) Ag/PbS HGWs

- Lowest α in Ag/PbS HGWs
- All samples showed some single-mode like TEM propagation behavior despite multi-modal nature of large bore HGWs

Novel Dielectric Film Designs for Use in HGWs

- Current research involves the successful thin film deposition of highly lucrative IR materials for use in HGWs including highly IR transmitting ZnSe and ZnS as well as low index polymer thin films such as polystyrene
- Incorporating multiple dielectric layers of appropriate thicknesses consisting of materials of alternating low (n_L) and high (n_H) refractive indices will allow for enhancement of reflection coefficient and thus higher transmission
- ZnSe and ZnS thin films would allow for highly transparent IR thin films with low-medium refractive indices having excellent properties as single films or in along with high index films such as PbS in multi-layer designs
- To date, multi-layer designs including CdS/PbS, CdS/PbS, and CdS/PS alternating layers have been successfully produced and continue to be thoroughly researched for optimal functionality in desired applications

Applications of HGWs & Conclusion

- HGWs are used in a variety of applications involving transmission of light at infrared wavelengths including high power laser delivery for surgical applications and remote chemical sensing involving spectroscopy
- HGWs effectively incorporate dielectric thin film designs for low-loss transmission of infrared radiation
- HGWs can be easily and inexpensively tailored for maximum transmission at desired wavelength(s)

References

[1] Harrington, James A. Infrared Fibers and Their Applications. Bellingham, WA: SPIE Optical Engineering Press , 2004. Print.

[2] Y. Matsuura, M. Saito, M. Miyagi, and A. Hongo, "Loss characteristics of circular hollow waveguides for incoherent infrared light", Journal of the Optical Society of America, Volume 6, Issue 3, pp. 423-427 (1989).

Acknowledgements

The research presented is made possible primarily through funding from the US Department of Energy (DOE) under Award Number DE-SC0001466. Additional funding for the development of ZnSe dielectric thin film coatings is made possible through the Rutgers Aresty Research Center (ARC) for Undergraduate research. The authors would like to thank Dr. Jason Kriesel at Opto-Knowledge Systems Incorporated (OKSI) for continuing support of this research project and to Mr. Daniel V. Kopp and Mr. David Tran for their involvement in the preparation of samples and measurements included in this study.