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DESIGN AND CONSTRUCTION OF THE ULTIMATE ELLIPSMETER, AND
EVALUATION OF SOL-GE†-derived ELECTRO-OPTIC AND MAGNETO-OPTIC
THIN FILMS

by

John Thomas Simpson

A Dissertation Submitted to the Faculty of the

COMMITTEE ON OPTICAL SCIENCES (GRADUATE)

In Partial Fulfillment of the Requirements
For the Degree of

DOCTOR OF PHILOSOPHY

In the Graduate College

THE UNIVERSITY OF ARIZONA

1995
As members of the Final Examination Committee, we certify that we have read the dissertation prepared by John Thomas Simpson entitled Design and construction of the ultimate ellipsometer, and evaluation of sol-gel derived electro-optic and magneto-optic thin films and recommend that it be accepted as fulfilling the dissertation requirement for the Degree of Doctor of Philosophy.

Masud Mansuripur 3/7/95
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Final approval and acceptance of this dissertation is contingent upon the candidate's submission of the final copy of the dissertation to the Graduate College.

I hereby certify that I have read this dissertation prepared under my direction and recommend that it be accepted as fulfilling the dissertation requirement.

Masud Mansuripur 3/7/95
Dissertation Director
STATEMENT OF AUTHOR

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SIGNED: John Thomas Simpson
ACKNOWLEDGMENTS

As with most worthwhile endeavors, this dissertation was far from a solitary effort. I feel very fortunate to have had the opportunity of attending the Optical Sciences Center. The faculty, staff, and fellow students have enriched my life and made my journey through the world of optics both enriching and enjoyable. I would like to give special thanks to the following people:

My major professor Masud Mansuripur. Working with Masud has been intellectually stimulating and fulfilling.

Professor H. Angus Macleod and Tianji Zhao who were of great assistance in various aspects of thin film design and fabrication.

G.T. (G. Teowee) of Donnelly Corporation for the countless numbers of MO and EO samples. (Thanks G.T. for all of your help and technical collaboration).

Kevin Erwin for his technical assistance with the Ultimate Ellipsometer design and implementation.

Warren Bletcher for assisting with the design and implementation of various Ultimate Ellipsometer electronics.

Finally, I give a big thanks to Jim Haidinyak of IBM for allowing me to pursue this degree.
DEDICATION

I dedicate this dissertation to the memory of Mrs. Arnurius, my fifth grade teacher at Norris Elementary School, who ignited an inner spark many years ago that gave me the desire and confidence to set and achieve my goals in life.
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ABSTRACT

The design and implementation details of an advanced type of optical measurement device are presented (the Ultimate Ellipsometer). Design and functional details of its major components and subcomponents are described. A list of its optical measurement capabilities (polarization rotation and ellipticity, grating diffraction order efficiencies and polarization state measurements, magneto-optic, and electro-optic measurements) is presented, along with various measurement results. In addition, optical, magneto-optical, and electro-optical characterization results of sol-gel derived bismuth doped dysprosium iron garnet (B.I.G.), lead zirconate titanate (PZT), and lead lanthanum zirconate titanate (PLZT) thin films are presented.
CHAPTER 1

INTRODUCTION

1.1 RESEARCH GOALS

This thesis describes research directed toward the design and implementation of an advanced type of optical measurement device (the Ultimate Ellipsometer), and its use in evaluating sol-gel derived bismuth doped iron garnets (B.I.G.) magneto-optical thin films as well as lead zirconate titanate (PZT) and lead lanthanum zirconate titanate (PLZT) electro-optical thin films. The Ultimate Ellipsometer research has the immediate effect of introducing a new and powerful measurement tool to the optics and physics research communities. The sol-gel derived B.I.G., PZT, and PLZT thin film research involves the optical, magneto-optical and electro-optical characterization of these materials. Since sol-gel processing of these materials is a relatively recent occurrence (Teowee, 1992), characterization results of these materials help to establish differences and similarities as compared to the non sol-gel processed materials (i.e., bulk, sputtered, vacuum deposited, etc.).

Ellipsometers are opto-mechanical devices used to measure the complex refractive index \((n, k)\) and thickness \((t)\) of thin films. The name, “Ellipsometer” refers to a device that can measure the rotation and ellipticity associated with the ellipse of
polarization. When polarized light interacts with a material, either at an interface or through a thin film, the state of polarization will, to some extent, change. The amount of change in polarization depends on the optical constants (n, k, t) of the material. By measuring the resultant ellipse of polarization, the optical constants can be deduced. For one ellipsometric measurement at one wavelength there are two measured parameters that can be related to two surface parameters, usually the complex refractive index (n) and the absorption coefficient (k). A surface that needs more parameters in its representation (e.g., n, k, and t) requires more measurements. At a constant wavelength this will normally be accomplished by varying the angle of incidence. Obviously, the more data points collected, the more accurate the estimate.

This thesis describes an enhanced ellipsometer type of device that can vary many of the traditional measurement parameters associated with ellipsometry. This new device can also measure certain electro-optical and magneto-optical parameters. This gives us the ability to measure a set of related optical properties for a given sample and then combine the individual results into a composite result. The name given to this new device is the "Ultimate Ellipsometer." The measurement capability of this device goes well beyond the capability of conventional ellipsometers. Our design goals were to improve upon previous ellipsometer designs, to give this device more flexibility, more degrees of freedom, and more overall measurement capability.

Currently amorphous thin films of TbFeCo are the dominant material used in the magneto-optical (MO) data storage industry. These materials exhibit reasonably large MO
effects at red and near infrared wavelengths. As optical data storage moves to higher
densities, there is a need for materials with large MO effects at short wavelengths
(e.g., blue, green). Bismuth doped iron garnets are among the materials being considered
as possible replacements for TbFeCo (Hansen et al., 1983, Gomi et al., 1985).
A considerable amount of time and effort has been spent making and characterizing
B.I.G. thin films. Usually these films are made by RF sputtering, followed by rapid
very little, if any, consideration has been given to sol-gel processed B.I.G. thin films. One
goal of this research was to measure various optical and magneto-optical characteristics
of sol-gel derived Bi-doped dysprosium iron garnet films. This include's index of
refraction (\(n\)), absorption coefficient (\(k\)), film thickness (t), Faraday rotation (\(\theta_F\)), and
coercivity (\(H_c\)) measurements. Further described in this thesis is how various processing
parameters, such as spin speed and annealing temperatures, affect the optical and MO
properties.

Lead zirconate titanate (PZT) and lead lanthanum zirconate titanate (PLZT) are
ceramic perovskite ferroelectric materials that have received much attention due to their
exhibition of a variety of electro-optic (EO), piezoelectric, and nonlinear optical effects
(Wang and Haertling, 1993, Dumont et al, 1991, Potter et al., 1993, Preston and
is usually represented as PLZT(x/y/z) (e.g., PLZT (28/0/100)) which refers to the
following quaternary material composition: \(\text{Pb}_{1-x}\text{La}_x(\text{Zr}_y\text{Ti}_{1-y})_{1-(x/4)}\text{O}_3\). where \(z = 100 - y\).
PZT is usually represented as PZT(x/y) (e.g., PZT(53/47)) which refers to a composition of PbZr_xTi_yO_3. While both materials show large EO effects, the lanthanum doping in PLZT tends to lower the optical absorption and yields a higher optical quality material (Buchanan, 1986, Land, 1967, Land and Thacher, 1968, Land and Thacher, 1969). Bulk PZT and PLZT have been around for over twenty years now. But, like B.I.G., very little consideration has been given to sol-gel derived PZT and PLZT thin films. A third goal of this research was to characterize (using the Ultimate Ellipsometer) some of the optical and electro-optical properties of PZT and PLZT thin films prepared by a spin coating sol-gel process.

This research used two computer software packages for the analysis and design of multilayer thin films structures. The first package is called MACLEOD, and is commercially available from the Thin Film Center Inc. (see references). The second package (MULTILAYER) was written by Dr. Masud Mansuripur (University of Arizona) and is not generally available without the permission of professor Mansuripur.

1.2 STRUCTURE OF THESIS

This thesis contains four chapters. Chapter 1 is an introduction explaining the goals of this research. Chapter 2 describes the design and construction of an advanced optical measurement device called the Ultimate Ellipsometer. This chapter gives examples of
different types of optical measurements performed with this device (i.e., polarization rotation and ellipticity, grating measurements, magneto-optic measurements, and electro-optic measurements). Chapter 3 gives various characterization results of sol-gel derived, Bi-doped dysprosium iron garnet thin films. Chapter 4 gives various characterization results of sol-gel derived, electro-optic thin films of PZT and PLZT. Appendix A gives a Jones calculus polarization analysis of the Ultimate Ellipsometer. Appendix B gives the optical constant analysis results for indium tin oxide (ITO) and doped tin oxide (SnO) films used as transparent electrodes in conjunction with the PZT and PLZT films.
CHAPTER 2

THE ULTIMATE ELLIPSMETER

2.1 OVERVIEW

As the name “Ellipsometer” implies, this device measures the rotation and ellipticity of polarization when a linearly polarized laser beam interacts (either in reflection or transmission) with a sample material. “Ultimate” implies that this device allows every conceivable degree of freedom to be fully utilized.

2.1.1 MAJOR FEATURES

2.1.1.1 LARGE ANGULAR RANGE

A major feature of this ellipsometer is its large angular range. Both the Laser Arm and the Detector Arm subassemblies are free to independently revolve about the sample holder a full 360°. This gives the ability to measure a sample in reflection and transmission. We can also measure various orders of diffracted beams (in the case of gratings and samples with grooved surfaces) over a wide range of angles. For instance, in reflection the
incident angular range is from $9^\circ$ to $90^\circ$, while in transmission the incident angle can range from $0^\circ$ to $\pm 70^\circ$.

### 2.1.1.2 INSENSITIVITY TO WAVELENGTH CHANGES

We have endeavored to make this ellipsometer’s optical components as achromatic as possible. For instance, the quarter wave retarders are Fresnel rhombs that can be considered achromatic throughout the visible and into the near infrared regime ($300 \text{ nm} < \lambda < 1 \mu \text{m}$). We also use a Glan air spaced polarizer that has a design spectrum of $325 \text{ nm}$ to $2.2 \mu \text{m}$. The Detector Arm’s silicon detectors have on-board preamplifiers designed to give a broad band optical response. These achromatic features give us the ability to use different lasers (with different wavelengths), without having to replace the other optical components.

### 2.1.1.3 COMPACTNESS

The Laser Arm and Detector Arm are both designed to be as compact as possible. For instance, the Laser Arm uses micro posts and micro holders mounted on a mini rail to rigidly hold its optical and mechanical components.
2.1.1.4 POWER MEASUREMENT CAPABILITIES

By diverting half the laser beam intensity to a reference detector and using it for normalization purposes, we are able to measure a sample material's reflectance and transmittance. The equal intensity beam splitting is accomplished regardless of the laser's state of polarization or the orientation of the linear polarizer.

2.1.1.5 DIFFRACTION GRATING MEASUREMENTS

The ability to measure a sample's reflectance and/or transmittance along with the freedom to independently position the Laser Arm and the Detector Arm allow for measurements of the angular separations of the diffraction orders, diffraction efficiencies, and the polarization states of the various diffracted beams.

2.1.1.6 MAGNETO-OPTICAL MEASUREMENTS

A computer controlled electromagnet, producing a bipolar field of up to 10kOe, allows the measurement of a sample's magneto-optical properties (e.g., Kerr and Faraday rotation angles).
2.1.1.7 ELECTRO-OPTICAL MEASUREMENTS

A computer controlled, high precision, bipolar 0 to 100 volt power supply allows the application of variable voltages across thin films of electro-optic material. These applied voltages generate electric fields across the EO material. The differential detector scheme utilized in the detection arm of the ellipsometer is capable of measuring the small changes in the state of polarization that are usually the signature of the electro-optic effects induced by these fields.

2.1.1.8 COMPUTER CONTROLS

All photodetector signals (S0, S1, and S2) are computer monitored. The magnetic and electric field amplitudes and polarities are computer controlled. Our physical design allows for future enhancements that will enable greater control via computer hardware and software. Planned automatic control features include stepper motors to control various angular displacements. Currently all angular displacements are set and recorded manually.
2.1.2 MAJOR SUB-ASSEMBLIES

Figure 2-1 shows the physical relationship among the major subassemblies. Figure 2-2 shows the sequence of components encountered by the beam as it traverses the ellipsometer. Figure 2-2 also defines the relative angular relationships of this system.

2.1.2.1 LASER-ARM

This subassembly is free to revolve a full 360° about the center of the sample. Its major functions are:

1. To provide a coherent laser beam of any specified angular orientation of linear polarization, to the sample’s front surface.

2. To provide a wide range of incidence angles to the sample holder.

3. To provide the flexibility of using any one of a number of different lasers with differing sizes and wavelengths.

4. To provide variable attenuation of the laser light.

5. To provide a reference light power signal for use in reflectivity, transmissivity, and grating diffraction efficiency measurements.
2.1.2.2 DETECTOR-ARM

This subassembly is also free to revolve a full 360° about the front face of the sample holder. Its major functions are:

1. To monitor the total amount of laser power reaching the Detector Arm.

2. To provide the typical ellipsometer functions of 90° relative phase retardation along with an analyzer function (used together to extinguish elliptically polarized light).

3. To be insensitive to changes of laser wavelength.

4. To provide a wide range of incident angles relative to the sample holder.

2.1.2.3 SAMPLE HOLDER

The Sample Holder subassembly is located in the center of a 85cm long by 85cm wide by 1cm thick aluminum base plate. Its function is to rigidly hold sample materials of varying sizes and shapes in such a manner that the sample’s front surface is at the common center of revolution of the Laser Arm and the Detector Arm.
2.1.2.4 ELECTROMAGNET

This removable subassembly is mounted on the base plate and surrounds the sample holder. Its major functions are:

1. To provide a bipolar, uniform magnetic field (from 0 to 10k Oe) to the center of the sample holder.

2. To provide as wide a range of incident angles for the laser beam as possible.

Figure 2-1. “Ultimate Ellipsometer” showing the major subassemblies (i.e., Laser Arm, Detector Arm, Sample Holder, and removable Electromagnet).
Figure 2-2. Schematic of optical components encountered by the laser beam. Also shown is the angular relationships among the various components.

ASP = Air spaced polarizer
PBS = Polarizing beam splitter
FR1, FR2 = Fresnel rhombs
WP = Wollaston prism
PD1, PD2 = Silicon photodetectors
2.2 ULTIMATE ELLIPSOMETER SUBASSEMBLY COMPONENTS

2.2.1 LASER-ARM

The Laser Arm consists of an aluminum plate attached to a steel rod that can rotate in the horizontal plane about the front face of the sample holder. Metallic rollers, attached to the bottom of the aluminum plate, make any required rotation smooth and continuous. Attached to the top of the aluminum plate is a series of optical, mechanical, and electrical components mounted on a mini-rail structure (see Figure 2-3).

2.2.1.1 OPTICAL COMPONENTS

1. A removable laser (e.g., red HeNe, green HeNe, 780nm diode laser, etc.). Any laser (polarized or non-polarized) whose length is 30cm or less can be accommodated.

2. Two mirrors (M1, M2) mounted on adjustable mirror mounts used to redirect and align the beam.

3. A neutral density variable attenuator used to vary the laser beam's intensity.

4. A pair of diaphragms (Irices I1 and I2) which are used in conjunction with M1 and M2 to align the laser beam when changing from one laser to another.
5. A high quality Glan air spaced polarizer (ASP) used to ensure a highly linear polarization with its transmission axis at 45° from horizontal. Regardless of the laser’s initial polarization state, the beam exiting the ASP will be linearly polarized at 45°.

6. A Fresnel rhomb (FR1) used as an achromatic quarter wave retarder to change the beam from 45° linearly polarized to circularly polarized. A Fresnel rhomb has the effect of translating the beam, and thus the optical axis. Since FR1 has its fast axis fixed along the vertical direction, the resultant optical axis translation is a simple vertical shift (see Figure 2-4).

7. A broad band, high quality polarizing beam splitter (PBS) cube, used to split the circularly polarized beam into two equal amplitude linearly polarized beams.

8. A broad band photodetector (PD0) used to monitor a reference beam that is diverted from the PBS cube.

The combination of air-spaced polarizer (ASP), Fresnel rhomb (FR1), and polarizing beam splitter cube (PBS) produces two linearly polarized beams of equal power. This feature makes the deflected reference beam and non-deflected beam intensities independent of the polarizing beam splitter (PBS) cube’s angular position and thus independent of the polarization orientation.
2.2.1.2 MECHANICAL COMPONENTS

1. Adjustable laser support mounts used to accommodate different size lasers.

2. Translation stages for mirrors M1 and M2 used to redirect and align the laser beam.

3. Rotation stages (RS1, RS2) used to rotate the ASP and the PBS cube respectively. Both rotation stages have graduated scales ranging from 0 to 359°. Their orientations are identified by angles $\theta_{ASP}$ and $\theta_{pol}$ respectively. Note that positive $\theta_{ASP}$ and $\theta_{pol}$ readings correspond to a clockwise rotation with respect to incident polarization (accurate to within ±0.25°). The ASP has a 45° offset. Thus $\theta_{ASP} = 0^\circ$ corresponds to a clockwise rotation of 45° (from horizontal). The PBS cube’s offset is zero. So, $\theta_{pol} = 0^\circ$ corresponds to horizontally polarized light exiting the cube (see Figure 2-2).

4. A stationary customized mount used to rigidly hold FR1.

5. A customized detector mount attached to rotation stage RS2 used to ensure a constant angular relationship between the PBS’s deflected beam and the PD0 detector.

6. A locking mechanism to anchor the Laser Arm at a given incident angular position ($\psi_{inc}$).
2.2.1.3 ELECTRICAL/ELECTRONIC COMPONENTS

1. A high voltage power supply for the HeNe laser heads.

2. A broad band silicon photodetector (PD0) with external amplification and gain adjustment.

Figure 2-3. Laser Arm Subassembly (Top View).

Figure 2-4. Fresnel Rhomb (Achromatic quarter-wave retarder).
2.2.2 DETECTOR-ARM

The Detector Arm also consists of an aluminum plate attached to a steel rod that can rotate in the horizontal plane about the center of a sample holder. Metallic rollers, attached to the bottom aluminum plate, make any required rotation smooth and continuous. Attached to the top of the aluminum plate is a series of optical, mechanical, and electrical components (see Figure 2-5).

2.2.2.1 OPTICAL COMPONENTS

1. A diaphragm (Iris I3) used to align the beam (ensuring that the beam strikes the Fresnel rhomb (FR2) in the center of its front facet) and to block any unwanted reflections.

2. A Fresnel rhomb (FR2) that functions as an achromatic quarter wave retarder.

3. A Wollaston prism (WP) that splits the beam into two orthogonally polarized (s and p) linear components.

2.2.2.2 MECHANICAL COMPONENTS

1. Rotation stage (RS3) rotates FR2 and all other components of the Detector Arm about FR2’s entrance optical axis. As mentioned earlier, a Fresnel rhomb has the effect of
translating the beam, and thus the optical axis. Since FR2 is rotated about its front optical axis, the resulting translated optical axis shifts its position and describes a circular path. (see Figure 2-6). To account for this optical axis translation, the Wollaston prism's rotation stage is coupled to RS3. The result of this coupling is to make the operation of the Wollaston prism independent of the optical axis translation. RS3 has a zero offset graduated scale ranging from 0 to 359° (accurate to within ± 0.5°), and its orientation is designated by the angle θ_F. Note that a positive θ_F readings corresponds to a clockwise rotation with respect to incident polarization (e.g., θ_F = 30° corresponds to a counter clockwise rotation of 30° with respect to horizontal).

2. Inner rotation stage (RS4) rotates the Wollaston prism (WP) about FR2’s exit optical axis in a counterclockwise direction. This rotation stage has a zero offset graduated scale ranging from 0 to 359° (accurate to within ± 0.5°), and its orientation is designated by the angle θ_w. A positive reading for the stage indicates a counterclockwise rotation, (e.g., θ_w = 30° corresponds to a counter clockwise rotation of +30° with respect to RS3).

3. A customized mount that rigidly holds the rhomb FR2.

4. A locking mechanism to anchor the Detector Arm at a given angular position.
2.2.2.3 ELECTRICAL AND ELECTRONIC COMPONENTS

1. Two silicon photodetectors with on-board preamplifiers providing broadband responsivities.

2. Outboard amplification of the photodetector signals (S1, and S2) with computer controllable gains.

3. A differential amplifier providing the (S1-S2) difference signal.

Figure 2-5. Detector Arm Subassembly (top view).
Figure 2-6. Translation of optical axis by the Fresnel rhomb.
2.2.3 SAMPLE HOLDER

Figure 2-7 shows the Sample Holder used to secure the samples under test. The Sample Holder consists of the following mechanical components:

1. An aluminum yoke mounted to a vertical steel rod that is located at the center of the ellipsometer’s base plate (see Figure 2-7a,b).

2. Two anodized aluminum clamping plates. We have anodized (treated by oxidation) both clamping plates to make them electrically non-conducting. These plates have threaded holes that allow a set of brass screws to fix the plate’s vertical position (maximum vertical adjustment -6.5cm to +5.5cm). Both plates have tapered grooves that force the sample’s front surface (with gentle downward pressure on the top plate) to the common center of revolution of the Laser Arm and the Detector Arm (see Figure 2-7c).
Figure 2-7. Sample Holder (a) Front view, (b) side view, (c) expanded side view of clamping plates.
2.2.4 ELECTROMAGNET ASSEMBLY

The electro-magnet assembly consists of the following components:

1. A bipolar 10kOe field electromagnet with soft iron core and removable pole pieces.

2. Ten pounds (10 lbs) of 16 gauge copper wire wrapped around the core and cooled by chilled water that runs through copper tubing.

3. The magnet is driven by a computer controlled 40 Volt, 10 Amp power supply.

4. The pole pieces have been tapered down to concentrate the field in the gap. Each pole piece contains a pair of slits. For small angles of incidence (from $10^\circ$ to $30^\circ$) the beam will travel within the pole piece slit. This allow us to maintain a relatively large angular range while the electro-magnet is in use (see Figure 2-8).
Figure 2-8. Electro-magnet pole piece (a) Side view, (b) Front view, (c) Top view.
2.2.5 CONTROLLER

The controller is defined as a group of components used to control certain aspect of the ellipsometer. These controlling components are:

1. A Dell Personal Computer (486DX2 - 66MHz).

2. Signal amplifiers used to amplify the detector signals from the Laser Arm (S0) and the Detector Arm (S1, S2, and S1-S2).

3. Das16 PC I/O interface card and an I/O extender cable box from MetraByte Corp.
   1. Two D/A channels used to control the magnetic field strength and Electro-optic voltage levels.
   2. Four A/D channels used to monitor detector signals S0, S1, S2, and S1-S2.
   3. One digital output used to switch the magnet polarity.
   4. One digital output used to disable the EO 100 volt power supply.

4. A bipolar, 15 volt power supply.

5. BASIC software package subroutines from MetraByte Corporation.

6. Custom designed BASIC program written in Microsoft Quick Basic.

7. A Hewlett Packard 6267B (0-40Volt, 0-10 amp) computer controlled power supply.

8. A custom built switch (high current and computer controlled) used to change the magnetic polarity during magneto-optic measurements.
9. A computer controlled bipolar 100 volt high precision power supply used to vary the applied voltage across a set of electrodes during electro-optic (EO) measurements.
2.3 TYPES OF MEASUREMENTS

2.3.1 ELLIPSOMETRY (ROTATION AND ELLIPTICITY) MEASUREMENTS

In ellipsometry one measures the rotation and ellipticity of an ellipse of polarization caused by a sample material (Azzam and Bashara, 1987). Once the values of rotation and ellipticity are recorded, these values are used to estimate the sample material’s optical constants \((n, k, t)\). Using the Ultimate Ellipsometer, we can measure the resulting rotation and ellipticity induced on a linearly polarized incident beam after it has interacted with a sample material.

The Laser Arm subassembly, shown in Figure 2-3, produces a linearly polarized beam. The setting of PBS’s rotation stage (RS2) determines the beam’s polarization orientation (with respect to horizontal) with an accuracy of \(\pm 0.25^\circ\). After the beam interacts with the sample material, the resulting reflected/transmitted beam’s polarization will have rotated and/or developed ellipticity. In our notation any rotation caused by the sample is with respect to the incident polarization orientation (i.e., any induced rotation is relative to \(\theta_{\text{pad}}\)).

**Beam extinction** (or null ellipsometry) is a simple method of determining the rotation and ellipticity caused by the sample. When the beam reaches the Detector Arm, the Fresnel rhomb (FR2) and Wollaston prism (WP) are rotated such that one of the beams
emerging from the Wollaston Prism is extinguished (see Figure 2-5). By rotating the Fresnel rhomb (via rotation stage RS3), the relative phase difference between the two mutually orthogonal amplitude components (E1 and E2) can be eliminated. The resultant beam once again becomes linearly polarized. The amount of RS3 rotation required to linearly polarize the reflected beam is equal to the rotation caused by the sample material. Once the beam has become linearly polarized, rotation of the Wollaston prism will extinguish one of the two emergent beams. Since the rotation of RS4 is with respect to RS3, the amount of rotation required of RS4 is equal to the ellipticity caused by the sample material. This is a simple and straightforward method of measuring the rotation and ellipticity of the beam. A problem with this method is that solid state detectors are relatively insensitive at low light levels. When the beam approaches extinction, this insensitivity limits the detector’s ability to distinguish between small intensity variations. This detector insensitivity problem is avoided by using the human eye as a detector (i.e., beam extinction is visually observed).

The analyzer rotation method is an alternate method of extracting the sample material’s rotation and ellipticity values. In this method, we rotate the Wollaston prism via RS4 a full 360° while keeping all other optical components at fixed angular positions. The resulting normalized detector signal \(\frac{S1-S2}{S1+S2}\) varies sinusoidally versus RS4’s angle of rotation \(\theta_w\). Analysis of this sinusoidal signal yields the desired values of rotation and ellipticity. Even though the rhomb (FR2) is not rotated during the measurement, its initial angular position is very important. Ideally, to maximize the
detector signal’s peak to peak variations, the rhomb’s angular position $\theta_F$, (as set by RS3), should be equal to the PBS’s angle plus the expected amount of rotation and ellipticity (i.e., $\theta_F = \theta_{pol} + \theta_k + \varepsilon_k$). Since this method does not rely on total extinction of the beam, the resulting measurement’s precision is greater than that achieved with the extinction method. This method requires that substantially more data points be measured for each angle of incidence. An advantage of this method is its ease of implementation by use of a computer controlled stepper motor attached to the analyzer rotation stage RS4.

2.3.2 POWER MEASUREMENTS (REFLECTANCE AND TRANSMITTANCE)

Detector PDO provides a reference signal (S0) that can be used, along with signals S1 and S2 from PD1 and PD2, to measure the reflectance and/or transmittance of a sample material. To measure a sample’s reflectance or transmittance, we must first establish reference signals (S0ref, S1ref and S2ref) that correspond to a maximum reflectance or transmittance signal (i.e., $R = 1.00; T = 1.00$). To this end, we turn on the laser, allowing it to warm up and stabilize. By monitoring S0, we can easily determine when the laser has become stable. The time required is laser dependent. Our red HeNe laser needs about 20-30 minutes of warm up time, while our green HeNe laser requires 3 to 4 hours of warm up time to completely stabilize. To set the desired incident beam’s polarization orientation, rotate the PBS cube to the desired angular position. (Orientation should not
be changed while taking measurements). Remove any sample from the sample holder, and ensure that the incident beam goes unobstructed to the Detector Arm. Once the incident polarization is set, rotate the Detector Arm’s rhomb (FR2) via rotation stage RS3, to an angular position approximately 45° from the incident beam’s orientation. This will circularize the polarization exiting the rhomb and will send equal amounts of light to detectors PD1 and PD2. Confirm by monitoring S1 and S2. If the signals are very weak or close to saturation, rotate the Laser Arm’s variable attenuator until both signals are approximately mid-range (=1000). This balancing of power between PD1 and PD2 helps ensure that both detectors are operating in their linear regime. The final step is to measure the combined laser and background light levels reaching the detectors. These signal values are referred to as the reference signals S0ref, S1ref, and S2ref. Try to reduce the background light level as much as possible. (Once the background level is established and the reference signals are recorded, the same background light level must be maintained while reflectance or transmittance measurements are underway.) S1 and S2 are now maximized by positioning the Laser and Detector Arms exactly 180° apart. This should give a maximum sum (S1 + S2) signal. A final adjustment of the variable attenuator is also permissible to maximize detector signals S0, S1, and S2 below their saturation values. Record S0, S1 and S2 as the reference signals. Place the sample material into the sample holder and begin reflection or transmission measurements.

The Ultimate Ellipsometer can measure a sample material’s reflectance and/or transmittance versus angle of incidence with an angular range of 9° to 90° in reflection,
and 0° to 70° in transmission. The Laser Arm’s angular position corresponds to the beam’s angle of incidence ($\psi_{\text{inc}}$). For a given $\psi_{\text{inc}}$, we rotate the detector arm in the opposite direction until the beam crosses the center of the Fresnel rhomb’s front face. Iris I3 has been positioned in front of FR2 such that a beam passing through its center will strike FR2’s front face center. The detector signals S0, S1, and S2 are then recorded. The sum of S1 and S2 divided by the reference signal $S_1\text{ref}+S_2\text{ref}$ determines the raw reflectance/transmittance value. Any laser power fluctuations that occur during the measurement process, are accounted for by normalizing the raw reflectance by $S_0/S_0\text{ref}$.

The recorded reflectance (R) or transmittance (T) is given by equation 2-1.

$$R, T = \frac{(S_1 + S_2)S_0\text{ref}}{(S_1\text{ref} + S_2\text{ref})S_0} \quad (2-1)$$

The validity of this equation is based on the assumption that all detectors and associated amplified signals are operating within their linear regime. It is also important to sample $S_0$, $S_1$, and $S_2$ simultaneously.
2.3.3 GRATING MEASUREMENTS

Gratings are periodic structures that cause an incident laser beam to be split (diffracted) into a series of beams known as diffraction orders (Born and Wolf, 1980, Saleh and Teich, 1991). The angular separation of these orders is a function of the grating periodicity (Ω), wavelength (λ), and incident beam angle of incidence (ψ\text{inc}) (see Figure 2-9). The percentage of incident power diffracted into each order is called the order’s diffraction efficiency and is associated with various physical characteristics of the grating (e.g., amplitude or phase modulation, duty cycle, shape, etc.). With the Ultimate Ellipsometer, we can measure a grating’s angular separation of diffraction orders, associated diffraction efficiencies, and diffraction order state of polarization (rotation and ellipticity).

To measure a grating’s diffraction angles, we start by placing the grating in the sample holder such that the “grooves” are aligned vertically. Next, the Laser Arm is revolved about the sample holder to a desired angle of incidence (ψ\text{inc}). By revolving the Detector Arm about the sample holder and observing any diffracted signals, we determine the angular locations of the various diffracted orders. By using the grating equation (2-2), the periodicity of the grating is readily calculated.
Grating Equation:  \[ \Omega \left( \sin \theta_m - \sin \psi_{inc} \right) = m\lambda \]  

where:  
- \( \Omega \) is the grating period  
- \( \theta_m \) is the angle of the \( m \)th order of diffraction  
- \( \psi_{inc} \) is the beam's angle of incidence  
- \( \lambda \) is the laser beam's wavelength  
- \( m \) is the diffraction order

Figure 2-9. Grating diagram of plane wave diffraction into the first few diffracted orders.

The measurement of a grating’s diffraction efficiency is very similar to a reflectance or transmittance measurement. We use the same power setup procedures as defined in section 2.3.2 to establish the required reference signals. We next use the diffraction order angular separation measurement procedure to locate the diffracted beams and record
detector signals S0, S1, and S2. After normalizing by the reference signals, diffraction efficiencies for various orders are readily obtained. To measure a diffraction order’s state of polarization, we use either the extinction method or the analyzer rotation method, as previously described.

2.3.4 MAGNETO-OPTIC MEASUREMENTS

Optical data storage devices make use of the magneto-optic (MO) Kerr effect to read data from optical disks. The essence of Kerr effect is that incident polarized light is reflected differently from a magneto-optic (MO) disk depending on the magnetization state of the disk material. This is a relatively small effect, but large enough to be measured and thus used. When incident s-polarized light is reflected by an MO disk, some of the light is converted to p-polarized. In general this converted light ($r_p$) is not in phase with the other reflected component ($r_s$) thus causing the resultant state of polarization to be both rotated ($\theta_k$) and elliptical ($\varepsilon_k$). $\theta_k$ and $\varepsilon_k$ are referred to as Kerr rotation and Kerr ellipticity respectively. The signs of Kerr rotation and ellipticity are directly related to the material’s magnetization state. By changing the material’s magnetization state while monitoring the normalized differential signal $(S1-S2)/(S1+S2)$, we are able to measure this effect. Two Ultimate Ellipsometer test procedures have been established to measure polarization changes induced by the electro-magnet. The first procedure (differential detector rotation)
switches the material’s magnetization state (between up and down) at a given analyzer (θw) position. (The Fresnel rhomb FR2 is fixed at 45° to incident polarization during this entire measurement). After recording the differential signal, the analyzer is rotated to a new position, where a new set of data is recorded. Methods for analyzing this data have been previously developed (Yeh, 1988, Macleod, 1989, Balasubramaniam et al., 1988) and computer programs exist that can calculate the sample’s optical properties. In this thesis we follow the MO measurement and analysis procedures developed in M. Mansuripur’s Optical Data Storage class notes (1994). Analysis of this data gives Kerr rotation and ellipticity. The second procedure (loop tracing) fixes the analyzer angular position (set at maximum differential variation determined from the first procedure), and incrementally varies the magnetic field strength and polarity. The normalized differential signal (S1-S2)/(S1+S2) then traces the material’s magnetic hysteresis, giving various magnetic characteristics of the material (e.g., coercivity, squareness, magnetic saturation, etc.) (Mansuripur, 1990, Wood, 1988, Wolf 1978). In both procedures the optimum Fresnel rhomb (FR2) angular setting is 45° to the direction of the incident polarization. The following set of equations (Mansuripur, 1994) are used to calculate Kerr (or Faraday) rotation (θk) and ellipticity (εk) given the normalized differential signal (S12) versus the angle of the detection module, θw. (Kerr effect occurs upon reflection, while Faraday effect occurs in transmission). Equation 2-3 shows that S12 versus θw is a sinusoidal curve. S12 is short for (S1-S2)/(S1+S2). From its measured amplitude we obtain $\frac{r_+}{r_-}$,
from its phase we find \( \gamma \). Once we have \( \frac{r_1}{n_1} \) and \( \gamma \), we use equations 2-4 and 2-5 to solve for \( \theta_k \) and \( \varepsilon_k \).

\[
S12 = \frac{2 \left| \frac{r_\perp}{r_\parallel} \right|}{1 + \left| \frac{r_\perp}{r_\parallel} \right|} \sin[2(\theta_w - 45) + \gamma] \tag{2-3}
\]

\[
\tan(2\theta_k) = \frac{2 \left| \frac{r_\perp}{r_\parallel} \right| \cos(\gamma)}{1 - \left| \frac{r_\perp}{r_\parallel} \right|^2} \tag{2-4}
\]

\[
\sin(2\varepsilon_k) = \frac{2 \left| \frac{r_\perp}{r_\parallel} \right| \sin(\gamma)}{1 + \left| \frac{r_\perp}{r_\parallel} \right|^2} \tag{2-5}
\]

### 2.3.5 ELECTRO-OPTIC MEASUREMENTS

In general, the presence of an externally applied electric field will effect the refractive index of any material. This phenomenon is known as the electro-optic effect. The first order electro-optic effect (i.e., change of refractive index proportional to electric field strength) is known as the Pockels effect. The second order electro-optic effect
(i.e., change in refractive index proportional to the square of the electric field strength), is known as the electro-optic Kerr effect. This electro-optic response is a consequence of the distortion of the localized electron cloud within a material’s fixed atomic structure. In most cases these effects are extremely small and can be ignored. Materials that exhibit relatively large changes of refractive index as a result of an applied field are referred to as electro-optic materials. In addition to a change in the material’s refractive index, the electric field may cause a physical expansion or constriction of the material. This effect is known as electrostriction. Thus, in general, an electric field will cause both a refractive index change ($\Delta n$) and a material thickness change ($\Delta t$). These changes act together to produce a change in the optical path length ($\Delta \text{OPL}$). The optical path length is the product of $n$ and $t$. Therefore $\Delta \text{OPL} = (\Delta n)t + n(\Delta t)$. For simplicity, we’ll let $\Delta t = 0$ and have $\Delta n$ represent the sum of both effects. The reader must recognize that the value of $\Delta n$ given throughout this thesis is an effective $\Delta n$ that combines the refractive index change with the electrostrictive effects. Two test procedures have been established to facilitate the measurement of refractive index changes in the direction of the E-field, induced in an EO material by a dc applied voltage (longitudinal EO effect). The first procedure is used to establish the optimum polarization orientation ($\theta_{\text{pol}}$) and angular positions of the Fresnel rhomb (FR2) and Wollaston prism ($\theta_w$). This first procedure (differential voltage test) alternates between applying a voltage (creating an electric field) and removing the field (no applied voltage) at a given angular position (i.e., fixed $\theta_{\text{pol}}$, $\theta_F$, and $\theta_w$). After recording the differential signal, a particular stage is rotated and a new set of data is
recorded. Analysis of this data allows us to establish the angular positions that maximize the EO induced differential detector signal. The second procedure (incremental field differential detection) fixes the ellipsometer’s angular positions (set at maximum differential variation determined from the first procedure), and incrementally varies the applied voltage. The normalized differential signal $S_{12}$ then traces a signal (related to the material’s $\Delta n$) vs. the applied voltage. Note that these measurements are made with a dc applied voltage in conjunction with a differential detector signal, as opposed to an ac applied voltage in conjunction with a lock-in amplifier signal (typical ac applied voltage and lock-in ac frequencies are from 1 kilohertz to 100 kilohertz). There is no reason why an ac voltage with a lock-in amplifier could not be used by the Ultimate Ellipsometer. It just so happens that the Ultimate Ellipsometer is sensitive enough to measure the electro-optic effect for the materials of interest in this thesis (PZT, and PLZT) with a dc applied voltage.

2.3.5.1 CALCULATION OF $\Delta n$

To calculate the voltage-induced refractive index change ($\Delta n$) given the normalized differential signal ($\Delta S_{12}$), we use the results of Jones Calculus analysis from appendix A. Equations 2-6 and 2-7 show the general expressions for $S_1$ and $S_2$. 
\[ S_{1\text{(normalized)}} = \left| V_x \cos \theta_{pol} [\sin \theta_w \cos \theta_F - i \cos \theta_w \sin \theta_F] + V_y \sin \theta_{pol} [\sin \theta_w \sin \theta_F + i \cos \theta_w \cos \theta_F] \right|^2 \]  
\[ (2-6) \]

\[ S_{2\text{(normalized)}} = \left| V_x \cos \theta_{pol} [\cos \theta_w \cos \theta_F + i \sin \theta_w \sin \theta_F] - V_y \sin \theta_{pol} [-\cos \theta_w \sin \theta_F + i \sin \theta_w \cos \theta_F] \right|^2 \]  
\[ (2-7) \]

\( V_x \) and \( V_y \) represent the sample’s Jones matrix diagonal components (the off-diagonal components are assumed to be zero). In general \( V_x = r_x e^{i \phi_x} \) and \( V_y = r_y e^{i \phi_y} \) where \( r_x \) and \( r_y \) are the Fresnel reflection coefficients and \( \phi_x \) and \( \phi_y \) are the associated component phases.

Equation 2-8 defines \( S_{12} \) as the normalized differential signal, while \( \Delta S_{12} \) is defined as the difference between a differential signal with no applied voltage and the differential signal with an applied voltage.

\[ S_{12} = (S_1 - S_2)/(S_1 + S_2) \]  
\[ (2-8) \]

\[ \Delta S_{12} = S_{12_0} - S_{12_{\Delta n}} \]  
\[ (2-9) \]

The strategy for determining the electro-optic induced \( \Delta n \) from a \( \Delta S_{12} \) measurement is as follows. We use the MULTILAYER (Mansuripur, 1990) program to get an initial set of \( V_x \) and \( V_y \) values and to solve equations 2-6, 2-7, and 2-8. This gives an initial \( S_1, S_2, \) and \( S_{12_0} \) value. The problem then becomes: how much \( \Delta n \) do we have to add or subtract from the EO material’s \( n_z \) component in order to achieve the measured \( \Delta S_{12} \). Again, \( \Delta n \) can be found from the MULTILAYER program by trial and error.
2.3.5.2 $r_{\text{eff}}$ and its relation with $\Delta n$

As mentioned earlier, $\Delta n$ is not a true electro-optically induced change of refractive index, because it combines electro-optic and electrostrictive effects. Likewise equations 2-10 and 2-11 define an effective electro-optic coefficient $r_{\text{eff}}$.

\[
-\frac{\partial}{\partial n}\left(\frac{1}{n^3}\right) = r_{\text{eff}} * E = \frac{2\Delta n}{n^3}
\]  \hspace{1cm} (2-10)

\[
r_{\text{eff}} = \frac{2\Delta n}{En^3} = \frac{2t\Delta n}{n^3V}
\]  \hspace{1cm} (2-11)

Where $n =$ the EO material's refractive index
$V =$ the dc applied voltage
$t =$ the thickness of the Electro-optic layer
$\Delta n =$ the effective electro-optic induced birefringence ($n_2-n_1$)
2.4 EXPERIMENTAL RESULTS AND COMPARISON WITH THEORY

2.4.1 ELLIPSOMETRY MEASUREMENTS

In this subsection we present ellipsometric results from two samples: an aluminum mirror and an Indium Tin Oxide (ITO) thin film on glass substrate.

**Beam extinction method (Null Ellipsometry):**

Figure 2-10 shows the measured rotation and ellipticity obtained from an aluminum mirror. The resulting data fit and corresponding analysis show the values of $n$ and $k$ to be 0.88 and 5.70 respectively. These $n$ and $k$ values match very well with the results obtained from a commercial ellipsometer.

![Figure 2-10. Aluminum mirror front-surface ellipsometric rotation and ellipticity measurement. Polarization orientation ($\theta_{pol}$) = 60°. Extinction method observed by naked eye. Analysis results: $n = 0.88$, $k = 5.7$](image)
Figure 2-11 shows the measured rotation and ellipticity of an ITO thin film (nominal thickness = 200nm) on a soda lime glass substrate (n=1.51). The polarization orientation (\theta_{pol}) was 60°. The circles represent the measurements taken by the Ultimate ellipsometer within the incident angular range of 10° to 86°. The squares represent the measurements taken by a commercial multiwavelength ellipsometer with the incident angle ranging from 36° to 70°. The solid line represents the theoretical results assuming n = 2.00 and t = 232nm). Note that the angular range of measurable data points with the Ultimate Ellipsometer is somewhat greater than that with the commercial device.

Figure 2-11. Null ellipsometry measurements for ITO thin film on glass substrate (n=1.51). Polarization orientation (\theta_{pol}) = 60°. Analysis results: Ultimate ellipsometer n = 2.00, k = 0.0001, t = 232nm. Multiwavelength ellipsometer: n = 2.00, k = 0, t = 216nm
Figure 2-12 shows the same ITO sample measured for polarization rotation and ellipticity after the incident polarization orientation was changed to 30°. This new orientation results in an increase in both rotation and ellipticity. The larger rotation and ellipticity signals give a higher signal to noise ratio and thus yield a more accurate estimate of $n$ and $t$.

![Graph showing rotation and ellipticity](image)

**Figure 2-12.** ITO thin film on glass substrate. Rotation and ellipticity extinction method measurements. Polarization orientation ($\theta_{pol}$) = 30°. Analysis results: $n = 1.96$, $t = 236$nm.

The purpose of this example is to show that the amount of rotation and ellipticity can be increased or decreased by simply changing the incident beam’s polarization orientation.
Analyzer rotation method:

Figure 2-13 shows a plot of the normalized differential detector signal \((S_1 - S_2)/(S_1 + S_2)\) versus the angular position of the Wollaston prism \((\theta_w)\) for the same ITO sample. The beam’s angle of incidence \((\psi_{inc})\) was 60° and its polarization orientation \((\psi_{pol})\) was 30°; the quarter-wave plate (FR2) setting was 45°. Analysis of the data gives a rotation of 44° and an ellipticity of -7°. The corresponding extinction method give a rotation of 45° and an ellipticity of -6°.

![Plot of normalized differential detector signal](image)

**Figure 2-13.** ITO thin film analyzer rotation data. ITO thin film on glass substrate. Incident angle \((\psi_{inc}) = 60°\). Polarization orientation \((\psi_{pol}) = 30°\). RS3 setting \((\theta_f) = 45°\). Analysis results: rotation = 44°, ellipticity = -7°.
Figure 2.14 compares the rotation and ellipticity measurement data obtained from the beam extinction method and from the analyzer rotation method with a polarization orientation ($\theta_{pol}$) of 30°.

![Graph showing comparison of rotation and ellipticity results](image)

**Figure 2.14.** Comparison of extinction and analyzer rotation ellipsometric results. ITO thin film on glass substrate. Polarization orientation ($\theta_{pol}$) = 30°. Extinction method results: $n = 1.96$, $k = 0.00$, $t = 236$nm. Analyzer rotation method results: $n = 1.93$, $k = 0.00$, $t = 240$nm.

The purpose of this example is to confirm that both extinction and analyzer rotation methods yield nearly identical rotation and ellipticity results.
2.4.2 REFLECTANCE MEASUREMENTS

Four sets of test results are given in this subsection as examples of the Ultimate Ellipsometer power measurement capabilities. The first set of measurements shows the reflectance of a simple glass microscope slide (first using a red HeNe $\lambda = 632.8\text{nm}$ and then a $\lambda = 780\text{nm}$ semiconductor diode laser). Note the ability to calculate the glass plate’s refractive index by observing the Brewster’s angle (angle at which p-polarized light goes through a minimum) (Born and Wolf, 1980, Hecht and Zajac, 1987). That is, once the Brewster's angle ($\theta_B$) is determined, we know $n_{\text{sub}}$ from $n_{\text{sub}} = \tan(\theta_B)$. The next two measurements show the reflectance data from a five layer mirror of high and low index dielectric materials. The last power measurement example shows the reflectance of a thin film of Indium Tin Oxide (ITO) on a glass substrate. All power measurements shown include both s- and p-polarized incident light.

Figures 2.15(a) and (b) show a glass microscope slide’s reflectance versus angle of incidence using a red HeNe laser ($\lambda=632.8\text{nm}$) and then using a $\lambda=780\text{nm}$ diode laser. The circles show the measured values. The solid lines are the theoretical fits (i.e., Fresnel reflection coefficients). The back facet of the slide was covered with black tape to avoid secondary surface reflections.
Figure 2-15. Glass plate reflectance versus $\psi_{\text{inc}}$ (a) $\lambda=632.8\text{nm}, n=1.51$, (b) $\lambda=780\text{nm}, n=1.51$.

Figure 2.16a shows the reflectance versus angle of incidence for a five layer quarter wave stack. (The term “quarter wave stack” refers to a series of layers each having an optical thickness, $nd$, equal to one quarter of a reference wavelength ($\lambda$), and alternating high and low refractive indices). The light source used was a two milliwatt red HeNe laser ($\lambda = 632.8 \text{ nm}$). The high index material ($n_H = 2.3$) is ZnS, while the low index material ($n_L = 1.35$) is Cryolite(Na$_3$AlF$_6$). The solid line shows the theoretical reflectance for a perfectly constructed five layer stack. The solid circles (sample # 1) show the measured data for a real stack that was annealed at $450^\circ\text{C}$ for three hours. The hollow circles (sample # 2) show the measured data for a stack that was not annealed.

Figure 2-16b shows the theoretical and measured reflectance curves for the five layer stack assuming that the stack’s optical thickness, for each layers, is $0.27\lambda_d$ instead of
0.25\(\lambda_d\) (\(\lambda_d = \) optical design wavelength). This assumption yields a very close match and implies that the stack layers are slightly thicker than the design specification.

**Figure 2-16.** Reflectance versus \(\psi_{\text{inc}}\) for a five layer stack. The stack consists of alternating layers of ZnS and Cryolite (Na$_3$AlF$_6$). \(\lambda_d = 632.8\text{nm}\). (a) layer thickness of 0.25\(\lambda_d\), (b) layer thickness of 0.27\(\lambda_d\).

Figure 2-17 shows the theoretical and measured reflectance of both s- and p-polarized light from a thin film of ITO deposited on a glass substrate. (\(n_{\text{ito}} = 1.96\), \(n_{\text{glass}} = 1.5\), \(t_{\text{ito}} = 236\text{nm}\)). This is the same sample that was analyzed earlier in Figures 2-12, 2-13, and 2-14.
2.4.3 GRATING MEASUREMENTS

2.4.3.1 DIFFRACTION ORDER EFFICIENCIES

The following figures show the measured diffraction efficiencies of a 1.6 micron pitch phase grating milled on a glass substrate. Figure 2-18 shows the $0^{\text{th}}$, $-1^{\text{st}}$, and $+2^{\text{nd}}$ order diffraction efficiencies versus incident angle ($\Psi_{\text{inc}}$). In each case both s- and p-polarizations were used as the incident beam's polarization state. Note that the diffraction efficiencies for the zero order are very nearly the same as Fresnel's reflection coefficients from an air/glass interface.
Figure 2-18. Diffraction order efficiencies in reflection of a 1.6 micron pitch gold plated phase grating. (a) 0 order diffraction, (b) -1st order, (c) +2nd order.
2.4.3.2 Magneto-optic (MO) effects from samples deposited on grooved substrates.

Figures 2-19(a), (b), and (c) show the magneto-optic Kerr hysteresis loops obtained from different diffraction orders (0, -1st, and +2nd), from a 1 micron pitch phase grating coated with the magneto-optic material TbFeCo.

Figure 2-19. TbFeCo coated phase grating Kerr loops obtained from the various diffraction orders. \( \lambda = 632.8\text{nm}, \) grating pitch = 1 micron, groove depth = 200nm, (a) 0 order diffraction, (b) -1st order diffraction, (c) +2nd order diffraction.
2.4.4 MAGNETO-OPTIC MEASUREMENTS

In this subsection we present magneto-optic measurement (Faraday and Kerr rotation) results from samples of bismuth-doped dysprosium iron garnet (B.I.G.) using laser wavelengths of 633nm, 544nm, and 780nm. All the measurements described in this section were made with $\theta_{pol} = 90^\circ$ (s-polarized incident light) and $\theta_F$ (FR2) = 45°. Figures 2-20(a) and (b) demonstrate the use of the analyzer rotation method to measure the Kerr effect ($e_k$ and $\theta_k$). In figure 2-20a the two curves show the normalized differential signals $(S1-S2)/(S1+S2)$ for different magnetic polarities (up/down magnetization states) versus analyzer angular position. These normalized differential signals are referred to as $S_{12up}$ and $S_{12dn}$. Figure 2-20b is a plot of the difference signal obtained from subtracting the two curves in Figure 2-20a ($S_{12up} - S_{12dn}$). From the phase and amplitude of this sinusoidal difference signal, the Kerr rotation and ellipticity are readily obtained (Mansuripur, 1990). Figure 2-20 shows that even when the normalized differential signals are very noisy we still tend to get a smooth $S_{12up}$-$S_{12dn}$ signal.

Figures 2-21(a) and (b) show the significant increase in differential signal amplitude achieved by using a green HeNe laser ($\lambda=543.5$nm). Figure 2-22 demonstrates the use of differential detection method to measure the hysteresis loop using the Faraday effect. Figure 2.23 is a composite of Faraday rotation loops measured on a B.I.G. sample using three different lasers ($\lambda = 543.5$nm, 632.8nm, and 780nm).
Figure 2-20. B.I.G. Kerr rotation measurements using analyzer rotation method. 
\( \lambda = 632.8 \text{nm}, \theta_{\text{pol}} = 90^\circ \) (s-pol), \( \Psi_{\text{inc}} = 10^\circ \). (a) normalized S12up and S12dn signals vs. \( \theta_w \). (b) normalized S12up-S12dn resultant signal vs. \( \theta_w \). Analysis results: ellipticity \( (\varepsilon_k) = 0.037^\circ \); Rotation \( (\theta_k) = 0.06^\circ \).

Figure 2-21. B.I.G. Kerr rotation measurements using the analyzer rotation method with \( \lambda = 544 \text{nm} \). (a) S12up and S12dn signals versus \( \theta_w \). (b) S12up - S12dn signal versus \( \theta_w \). Analysis results: Ellipticity \( (\varepsilon_k) = 0.00^\circ \); Rotation \( (\theta_k) = 0.21^\circ \).
**Figure 2-22.** B.I.G. sample Faraday effect hysteresis loop. $\lambda = 633$nm. Angle of incidence ($\psi_{\text{inc}}$) = 20$^\circ$, film thickness = 250nm. The Faraday rotation angle for this sample is about 1.0$^\circ$ per micron.

**Figure 2-23.** Composite of three B.I.G. Faraday loop measurements at wavelengths of 544nm, 632.8nm, and 780nm. Angle of incidence ($\psi_{\text{inc}}$) = 20$^\circ$. The Faraday rotation for this sample ranged from 0.2$^\circ$ at $\lambda = 780$nm to 1.7$^\circ$ at $\lambda = 544$nm.
2.4.5 ELECTRO-OPTIC MEASUREMENTS

In this subsection we present electro-optic (EO) measurement results from samples of sol-gel derived PLZT and PZT. Differential detector signal (S12) versus applied voltage (V) results are presented at a laser wavelength of 632.8nm. Figures 2-24(a) and (b) show S12 versus applied voltage for thin films of PLZT and PZT respectively.

Figure 2-24. EO measurements on sol-gel derived PZT/PLT the films. Normalized differential detector signal (S12) versus applied voltage. (a) PLZT (typical quadratic EO pattern). Analysis: $\Delta n = 0.020, r_{\text{eff}} = 59\text{pm/volt}$. (b) PZT (classic EO “Butterfly” pattern). Analysis: $\Delta n = 0.051; r_{\text{eff}} = 315\text{pm/volt}$.

Chapter 4 (section 4.3) gives a detailed description of these two samples, the test setup, and the analysis results.
2.5 SUMMARY

This chapter has described a new ellipsometer that goes beyond the conventional capabilities of ellipsometry. In addition to measuring the ellipse of polarization (rotation and ellipticity) in reflection and transmission, this device can measure a sample's reflectance (R), transmittance (T), grating periodicity, diffraction order efficiencies, diffraction order state of polarization, magneto-optic Kerr effects ($\varepsilon_k$ and $\theta_k$) and Faraday effects ($\varepsilon_F$ and $\theta_F$) as well as electro-optic effects ($\Delta n$ and $r_{eff}$). All of this measurement capability is contained in a compact, light weight structure containing high precision achromatic optical elements. The various measurements performed to date have shown a high degree of accuracy and repeatability. With so much measurement capability, the possibility of combining and integrating various procedures and measurement results into a unified optical/magneto-optical/electro-optical analysis tool is very real and constitutes a possible extension of this research for the future. With the addition of automated control devices, (such as stepper motors), and a unified set of analysis software programs the "Ultimate Ellipsometer" holds the promise of being the premier optical, magneto-optical, and electro-optical research measurement device.
CHAPTER 3

CHARACTERIZATION OF SOL-GEL DERIVED BISMUTH DOPED IRON GARNET THIN FILMS

Spin coating is the best technique for coating thin, uniform, organic films on round substrates. This method is widely used to coat photoresist masters. Materials for spin coating must be in a liquid state. The spin coating process is a complex interplay of physical effects (e.g., centrifugal force, viscosity, and solvent evaporation rate). The remarkable result of these physical effects is that the eventual thickness of the thin film layer is nearly uniform and independent of position (radius). A simplistic explanation of this uniformity is that the centrifugal acceleration acting to throw the solution off the disk is countered by an increased viscosity as the film thins. This dynamic balance is the basis of the film thickness uniformity. In this chapter we discuss spin coated thin films of sol-gel derived Bi-doped dysprosium iron garnets. Sol-gel is a processing technique in which a fibrous gel is drawn (spin coated) from a solution at near room temperature and converted to a ceramic by annealing at several hundred degrees Celsius. An advantage of using a sol-gel solution is the near total elimination of organic precursors upon final processing. This yields a homogeneous thin film with high purity. A disadvantage of sol-gel processing is the high annealing temperatures required. This precludes using plastic or plastic-surfaced substrates (Marchant, 1990).
Sol-gel derived Bi-doped dysprosium iron garnet thin films having composition of 
\( \text{Bi}_x\text{Dy}_{3-x}\text{Fe}_5\text{O}_{12} \) are prepared from organic precursors (e.g., alkoxides) dissolved in an 
alcohol with bismuth content, \( x \), up to 1.5. A thin film is formed by spin coating onto a 
glass substrate. The films are amorphous when fired below 500°C. A non-garnet phase 
appears at about 500°C. To obtain single-phase garnet films, firing temperatures of at 
least 650°C are required. X-ray diffraction was performed to elucidate the phase 
development in these films. The phase assembly is highly dependent on composition and 
firing temperature. Dysprosium iron garnet films are easier to crystallize as compared 
with yttrium iron garnet films. With increasing Bi content, the films crystallize more 
readily; however, the garnet phase becomes harder to achieve and non-garnet phases 
proliferate. This chapter gives optical and magneto-optical (MO) characterization results 
(index of refraction, absorption coefficient, thickness, Faraday rotation, and coercivity) 
obtained from bismuth doped dysprosium iron garnet samples prepared under differing 
sol-gel processing conditions. We describe how various processing parameters, such as 
spin speed and annealing temperature, affect dysprosium iron garnet's optical and MO 
properties.
3.1 MEASUREMENTS OF BISMUTH DOPED DYSPROSIUM IRON GARNET (B.I.G.) THIN FILM ON GLASS SUBSTRATE

Two sets of B.I.G. samples were prepared using a sol-gel process, and characterized using the Ultimate Ellipsometer. Both sets of samples were made from the same solution (i.e., the organic solvents and B.I.G. precursor concentrations were the same for all samples). The first set of eight samples was spun at different spin speeds. The samples of this chapter are labeled by appending spin speed (in rpm), annealing temperature (in °C) and annealing duration (in minutes). As an example, BIG-2000-650-60 identifies a B.I.G. sample that was spin coated at 2000 rpm and annealed at 650°C for 60 minutes. We first look at a set of eight samples that were spun at different spin speed (ranging from 1000rpm to 4000rpm) and subsequently annealed at 650°C for three hours. Sample BIG-1250-650-180 was somewhat cloudy and caused excessive scattering. The other seven samples were clear and apparently of good optical quality. These eight samples were used to study how spin speed affects various optical properties of B.I.G. [i.e., refractive index (n), absorption coefficient (k), thickness (t), Faraday rotation (θ_F), and magnetic coercivity (H_c)]. The second set of samples were all spin coated at 1000 rpm. This set of samples was used to study how annealing temperature and annealing duration affects various optical and magnetic properties of B.I.G.
3.1.1 B.I.G. SPIN SPEED STUDY

In this subsection we give the ellipsometric data and optical constant (n, k, t) analysis results of eight B.I.G. samples spun at varying speeds ranging from 1000 rpm to 4000 rpm. After spin coating, each of these eight samples were annealed at 650°C for three hours. Therefore these samples are designated as BIG-xxxx-650-180, where xxxx corresponds to the particular spin speed.

3.1.1.1 ELLIPSOMETRY AND DETERMINATION OF OPTICAL CONSTANTS

Figures 3-1 (a)-(h) show both the measured and fitted (least squares fit) ellipsometric (rotation and ellipticity) data for eight B.I.G. samples at $\lambda = 633\text{nm}$. The assumed refractive index of the 7059 Corning glass substrate was $n = 1.52$. The incident polarization was at $30^\circ$ relative to $p$ direction.
Figure 3-1. Ellipsometric measurements (rotation and ellipticity) and fits from BIG spin speed study. (a) BIG-1000-650-180; Analysis: n = 1.77, k = 0.011, t = 359nm  
(b) BIG-1250-650-180; Analysis: n = 2.27, k = 0.010, t = 341nm  
(c) BIG-1500-650-180; Analysis: n = 1.79, k = 0.020, t = 294nm  
(d) BIG-1750-650-180; Analysis: n = 1.87, k = 0.010, t = 256nm  
(e) BIG-2000-650-180; Analysis: n = 2.10, k = 0.030, t = 217nm  
(f) BIG-2500-650-180; Analysis: n = 2.26, k = 0.010, t = 207nm  
(g) BIG-3000-650-180; Analysis: n = 2.50, k = 0.040, t = 181nm  
(h) BIG-4000-650-180; Analysis: n = 2.64, k = 0.050, t = 179nm
3.1.1.2 MAGNETO-OPTIC (FARADAY ROTATION) MEASUREMENTS

Knowing the optical properties of B.I.G. is very important. However, B.I.G. is of primary importance for its relatively large magneto-optic effects (i.e., its Kerr and Faraday rotation angles) at shorter wavelengths (i.e., blue, green). Therefore, we measured the Faraday rotation (in transmission) for each of the eight B.I.G. samples using the Ultimate Ellipsometer’s green HeNe laser and electro-magnet (a 543.5nm green HeNe laser yields nearly four times the Faraday rotation of a 632.8nm red HeNe laser). Figures 3-2 (a)-(h) show the Faraday rotation hysteresis loops associated with each BIG sample.
3.1.1.3 GENERAL TRENDS OF SPIN SPEED STUDY

After having all eight samples evaluated, we give some general trends. [Note: sample BIG-1250-650-180's optical values will be shown, but due to its rather poor optical quality we will not include it with the general trend fit lines]. Figure 3-3 is a plot of sample thickness versus spin coating speed (in rpm). The trend line is a decaying exponential and indicates that the minimum thickness attainable for this solution is roughly 170nm (i.e., increasing the spin speed above 3000 RPM has little effect on sample thickness). Note that this curve is associated with one particular concentration of solution. For solutions with less viscosity, lower sample thickness is expected. Another
caveat is that a particular sol-gel solution’s viscosity tends to increase over time. (i.e., a newly formed solution produces a thinner film than will an aged solution). Figure 3-4 shows that the refractive index tends to exponentially decrease as the thickness increases (i.e., the refractive index increases with spin speed). These trends of increased index and decreased thickness are explained by the increased densification caused by rapid evaporation of the organic solvents (The faster the spin speed, the greater the evaporation rate). This increased evaporation rate eliminates much of the precursor (organic solvent) material prior to solidification. The solvent material present after solidification is removed by the annealing process. This post solidification solvent removal causes a certain amount of porosity in the sample and thus tends to decrease the film’s index of refraction while maintaining most of its thickness.

Figure 3-5 shows optical path length (OPL is the refractive index times thickness), normalized by wavelength, versus spin speed. (An OPL of unity equals 632.8nm.) Since refractive index (n) tends to be higher for thinner samples (higher spin speeds), any decrease in sample thickness is accompanied by an increase in refractive index. These two effects tend to keep OPL constant at higher spin speeds.

Figures 3-6 and 3-7 show trends from the Faraday rotation measurements using a green HeNe laser (λ=543.5nm). Figure 3-6 shows that sample coercivity quadratically increases with increasing spin speed (decreasing thickness) (Giles and Mansuripur, 1991).
Figure 3-7 shows Faraday rotation peaks of slightly more than two degrees per micron at a spin speed of approximately 2000 rpm.

![Big sample thickness (A-H) vs spin speed](image)

**Figure 3-3.** Thickness vs. spin coating speed for BIG-xxxx-650-180 samples.

![Index of refraction (n) vs sample thickness (t)](image)

**Figure 3-4.** Index of refraction $(n)$ versus sample thickness $(t)$ for BIG-xxxx-650-180 samples $(\lambda = 632.8\text{nm})$. 
Figure 3-5. Optical path length (OPL) versus spin speed for BIG-xxxx-650-180 samples ($\lambda = 633\text{nm}$).

Figure 3-6. Magnetic coercivity versus spin speed.
Figure 3-7. Faraday rotation per micron versus spin speed ($\lambda = 543.5$nm).
3.1.2 B.I.G. ANNEALING STUDY

For this study, we took identical samples of B.I.G. that had been spin coated at 1000 rpm and varied the annealing temperature and annealing duration of each sample. The annealing temperature ranged from 600°C to 700°C, while the annealing duration ranged from 1 minute to 5 hours. Table 3-1 summarizes how annealing conditions affect the B.I.G.'s optical and magneto-optical properties.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Duration</th>
<th>Rotation (θp) at λ = 544nm</th>
<th>Coercivity (Oe)</th>
<th>Index (n) at 633nm</th>
<th>Absorption coefficient (k) at 633nm</th>
<th>Thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BIG-1000-600-60</td>
<td>1 hr</td>
<td>0.33°</td>
<td>280</td>
<td>1.80</td>
<td>0.020</td>
<td>320</td>
</tr>
<tr>
<td>BIG-1000-600-120</td>
<td>2 hr</td>
<td>0.22°</td>
<td>270</td>
<td>1.81</td>
<td>0.010</td>
<td>326</td>
</tr>
<tr>
<td>BIG-1000-600-180</td>
<td>3 hr</td>
<td>0.33°</td>
<td>300</td>
<td>1.84</td>
<td>0.020</td>
<td>315</td>
</tr>
<tr>
<td>BIG-1000-600-300</td>
<td>5 hr</td>
<td>0.48°</td>
<td>300</td>
<td>1.81</td>
<td>0.020</td>
<td>309</td>
</tr>
<tr>
<td>BIG-1000-650-30</td>
<td>30 min.</td>
<td>0.47°</td>
<td>275</td>
<td>1.81</td>
<td>0.014</td>
<td>294</td>
</tr>
<tr>
<td>BIG-1000-650-60</td>
<td>1 hr</td>
<td>0.47°</td>
<td>290</td>
<td>1.77</td>
<td>0.010</td>
<td>309</td>
</tr>
<tr>
<td>BIG-1000-650-180</td>
<td>3 hr</td>
<td>0.47°</td>
<td>320</td>
<td>1.82</td>
<td>0.011</td>
<td>300</td>
</tr>
<tr>
<td>BIG-1000-650-300</td>
<td>5 hr</td>
<td>0.48°</td>
<td>384</td>
<td>1.83</td>
<td>0.029</td>
<td>304</td>
</tr>
<tr>
<td>BIG-1000-675-60</td>
<td>1 hr</td>
<td>0.45°</td>
<td>375</td>
<td>1.77</td>
<td>0.005</td>
<td>290</td>
</tr>
<tr>
<td>BIG-1000-675-180</td>
<td>3 hr</td>
<td>0.50°</td>
<td>485</td>
<td>1.81</td>
<td>0.015</td>
<td>275</td>
</tr>
<tr>
<td>BIG-1000-700-1</td>
<td>1 min.</td>
<td>0.47°</td>
<td>300</td>
<td>1.84</td>
<td>0.010</td>
<td>270</td>
</tr>
<tr>
<td>BIG-1000-700-15</td>
<td>15 min.</td>
<td>0.50°</td>
<td>650</td>
<td>2.37</td>
<td>0.013</td>
<td>164</td>
</tr>
</tbody>
</table>

Table 3-1. Summary of optical and magneto-optical measurements from the B.I.G. annealing study.
3.1.2.1 GENERAL TRENDS FROM ANNEALING STUDY

1. Magnetic coercivity increases with both annealing temperature and duration. This is probably due to the decrease in grain size associated with increasing the annealing temperature and/or annealing duration. These small magnetic grains are more resistant to magnetic reversal, as compared with larger grains.

2. Faraday rotation increases with annealing time. This is due to the increased crystallization of single-phase garnet.

3. While material thickness (t) decreases with both annealing temperature and duration, its index of refraction (n) may increase or decrease, depending on the amount of densification that occurs. During the annealing process, the organic solvents are extracted. The materials left behind will becomes more or less porous depending on the amount of densification.

4. The absorption coefficient (k) value is listed in the summary table. But no general trends should be deduced from this data. The values listed yield the minimum analysis error value, but there exists a wide range of k values where the analysis error is still very small [this is not true with index (n) or thickness (t)].
3.2 BISMUTH IRON GARNET IN THIN FILM FABRY-PEROT CAVITY

3.2.1 THIN FILM FABRY-PEROT BASICS

A Fabry-Perot structure consists of two mirrors and a cavity (see Figure 3-8). A thin film Fabry-Perot design uses two quarter wave stacks of alternating high and low index dielectric (non-absorbing) materials as its mirrors (see Figure 3-9), and a thin film cavity material, all deposited on a glass substrate. Quarter wave stacks of 20 or more layers with reflectances greater than 99.99% are not uncommon. A Fabry-Perot cavity has the characteristic of transmitting a well-defined envelope of wavelengths (see Figure 3-10). The center wavelength passed is dependent on the optical path length of the cavity material. Let's refer to this center wavelength as $\lambda_c$. For a particular $\lambda_c$, the optical path length between mirrors is exactly an integer multiple of half wavelengths (i.e., $\text{OPD} = m\lambda_c/2 = nd\cos\theta$, where $m$ is an integer and $\theta$ is the beam angle within the cavity). If the OPL is greater than $m\lambda_c/2$, it is still possible to observe resonant transmission through the Fabry-Perot structure. By tilting the Fabry-Perot cavity, with respect to the beam (i.e., $\theta > 0$), the following changes occur:

1. The mirror reflectance differs for s- and p-polarization components when the mirror is tilted. The tilted reflectance associated with the s-polarized light tends to increase, while the tilted reflectance associated with the p-polarized light tends to decrease at
first (i.e., Fresnel reflectance variations). Figure 3-11 shows the s-pol and p-pol component reflectances for an eight layer quarter wave stack consisting of alternating SiO₂ and TiO₂ thin films. This difference in tilted reflectance causes the transmission envelope width to decrease for s-pol light and increase for p-pol light.

2. OPDs for both components decrease as the angle of tilt (or angle of incidence) increases. This causes the design wavelength ($\lambda_c$) to decrease.

3. The admittance of each polarization component (s- and p-) varies differently with tilt angle (i.e., admittance $\eta_s = n \cos \theta$, while $\eta_p = n/\cos \theta$). Thus, as we tilt the device, the transmission envelope shifts to lower wavelengths and splits into two separate envelopes (see Figure 3-13). The s- component’s envelope narrows while the p- component’s envelope widens.

---

MIRROR

CAVITY

MIRROR

SUBSTRATE

Quarter wave stack (QWS)

Separation = $d = m\lambda/2$

Quarter wave stack (QWS)

---

**Figure 3-8.** Fabry-Perot structure.
Figure 3-9. The structure of quarter wave stack (mirror). In our designs the number of pairs of quarter wave stack layers varied from 8 to 11 to 17.

Figure 3-10. Calculated Fabry-Perot transmittance curve at normal incidence showing a well defined envelope of transmitted wavelengths. R = 99.0%, n = 2.0, d = 633nm.
Figure 3-11. Calculated reflectance curves for s- and p-polarization components of an eight layer quarter wave stack of alternating TiO₂ and SiO₂ thin films as a function of the angle of incidence.

The width of the envelope of transmission versus wavelength is a function of the mirror reflectances (the higher the reflectance, the narrower the envelope of wavelengths). At the extremes: for \( R = 0 \), all wavelengths are passed and the envelope is infinite in extent. For \( R = 99.99\% \) the envelope has shrunk to a minute extent (i.e., 0.1 angstrom at FWHM) allowing only the center wavelength to pass. For \( R = 90\% \) we get a relatively broad envelope of transmitted wavelengths. Figure 3-12 compares the Fabry-Perot's transmission envelope for mirrors with (a) 99.99% reflectance with (b) 90% reflectance.
Figure 3-12. Fabry-Perot calculated transmittance versus wavelength curves. OPL of cavity = 633nm, normal incidence, mirrors have. (a) 99.99% reflectance. (b) 90% reflectance.

Figure 3-13 shows the effect on p- and s-polarized light transmittance curves when the Fabry-Perot device is tilted by 45°. Notice that the transmission envelope splits as each polarization component moves to shorter wavelengths. Figure 3-14 shows that in order to achieve tilted transmission of s-polarization at $\theta = 45^\circ$ for $\lambda = 633$nm, the design wavelength (cavity thickness) must be longer than 633nm (e.g., 678nm).

Figure 3-13. Calculated transmittance of 45° tilted Fabry-Perot. (s- and p-polarization components, $\lambda_e = 633$nm, mirror reflectance at normal incidence (R) = 99%, cavity thickness = 633nm, n= 1.0).
Figure 3-14. Fabry-Perot 45° tilted transmittance calculation of s-polarized light with $\lambda_e = 678$nm. Mirror reflectance at normal incidence = 99% (i.e., 13 layer QWS). Cavity thickness = 678nm, index (n) = 2.0.
3.2.2 B.I.G. THIN FILM CHARACTERIZATION USING FABRY-PEROT STRUCTURE

This section describes the use of a thin film Fabry-Perot structure to determine the optical constants \((n, k, t)\) of two sol-gel processed B.I.G. samples.

3.2.2.1 SAMPLE #1 OPTICAL CONSTANT \((n, k, t)\) DETERMINATION

A spin coated layer of sol-gel derived B.I.G. (same composition and spin rate as sample BIG-2000-650-180 from previous section) was deposited onto a seventeen layer QWS composed of nine layers of TiO\(_2\) and eight layers of SiO\(_2\) on a glass substrate. (Figure 3-15 shows the calculated reflectance versus wavelength for the TiO\(_2\)/SiO\(_2\) QWS. These quarter-wave-thick layers of alternating high and low index materials give a maximum theoretical reflectance of 99.87\%. Another identical seventeen layer quarter wave stack was then deposited onto the B.I.G. material, making a thin film Fabry-Perot structure with B.I.G. as the cavity material. This sample will be referred to as B.I.G. Fabry-Perot sample #1.
Figure 3-15. Calculated reflectance vs. wavelength for a seventeen layer quarter wave stack of TiO₂ and SiO₂ on a glass substrate. Reference wavelength = 633nm. Maximum reflectance ($R_{\text{max}}$) = 99.87%. Coefficient of Finesse ($F$) = $4R/(1-R)^2$ = $2.4 \times 10^6$.

Figure 3-16 shows the measured and calculated spectral transmission versus wavelength curves for B.I.G. Fabry-Perot sample #1. We measured a small transmittance peak of 2% at a wavelength of 705nm. This Fabry-Perot thin film structure was analyzed by the MACLEOD Thin-Film Design Software program (Macleod, 1994). The measured transmittance shape and magnitude was duplicated by setting the cavity material’s optical constants to $n = 2.2$, $k = 0.012$, and $t = 209\text{nm}$.

Figure 3-16. B.I.G. Fabry-Perot #1 spectral transmittance and calculated fit at normal incidence. Fit results: $n = 2.2$, $k = 0.012$, $t = 209\text{nm}$. 
These optical constant values should be quite accurate since any slight deviation from these values causes significant changes in the transmission characteristics.

A second optical constants measurement method employs the tilting of the sample. If the optical thickness (nd cosθ) of the cavity layer is greater than mλc/2 at normal incidence, then tilting the cavity (making θ > 0) can make nd cosθ = mλc/2, and thus setup a resonance condition. Since s-polarized light and p-polarized light have differing admittances when θ > 0, the tilt required for resonance is also different. By measuring the required tilt for each component to achieve resonance (i.e., a transmission peak or reflection dip), we are able to deduce the cavity's optical path length (nd). Since this tilted analysis is done at λ = 632.8nm, we do not necessarily expect the same results for n and k as we obtained earlier at λ = 705nm (both n and k are wavelength dependent). B.I.G. Fabry-Perot sample #1 reflectance and transmittance was measured using a red HeNe laser (λc = 632.8nm). The reflectance measurement provided resonant dips for both p- and s-polarization components. It was observed that s-polarized light required approximately 64° of tilt to achieve resonance, while p-polarized light resonance required approximately 52° of tilt. The sample's s-polarization transmittance was too small to measure, while for p-polarization the transmittance peak was approximately 3% at 52°. Figure 3-17 shows the measured reflectance dips caused by the resonances of s- and p-polarized light, and the theoretical reflectance dips when the values n = 2.2, k = 0.014,
and $t = 209\text{nm}$ were used in calculations. The MACLEOD Thin-Film Design Software program was used to calculate the cavity’s optical constants required to achieve these resonances.

![Figure 3-17](image)

**Figure 3-17.** B.I.G. Fabry-Perot #1 measured and calculated reflectance dips. Calculated curve based on $n = 2.2$, $k = 0.014$, $t = 209\text{nm}$.

These calculated optical constants are quite accurate since any slight deviation causes significant changes in the transmission characteristics. As an example, Figure 3-18(a) shows that a 1% change in thickness (from $t = 209\text{nm}$ to $t = 211\text{nm}$) causes a 2 nanometer spectral shift and a 45% increase in peak transmittance. Figure 3-18(b) shows that a 10% change in refractive index (from $n = 2.2$ to $n = 2.0$) causes a 3 nanometer spectral shift and a 25% increase in peak transmittance. Figure 3-18(c) shows that a decrease in the absorption coefficient from $k = 0.014$ to $k = 0.010$ causes more than a 100% increase in peak transmittance (from $T = 2.1\%$ to $T = 4.7\%$).
Figure 3-18. BIG FP #1 spectral transmittance with small deviations from best-fit parameters. (a) effect of a 1% error in thickness $t$, (b) effect of a 10% error in refractive index $n$, (c) effect of a 28% error in absorption coefficient $k$. 
3.2.2.2 SAMPLE #2 OPTICAL CONSTANT (n, k, t) DETERMINATION

We made a second Fabry-Perot structure using two eight layer SiO$_2$/TiO$_2$ QWS (reference wavelength = 600nm) instead of the seventeen layer QWS for sample #1. This decrease in QWS number of layers increases the transmission at the expense of the Fabry-Perot’s transmission band width (Figure 3-19 shows the calculated reflectance versus wavelength for the eight layer QWS). These eight quarter wave thick layers of alternating high and low index materials give a maximum theoretical reflectance of 92.8%. Two successive layers of sol-gel processed B.I.G. (BIG-2000-650-180) were used as the cavity material for this sample. This sample will be referred to as B.I.G. Fabry-Perot sample #2. The quarter wave layers were deposited using a 600nm reference filter.

![Figure 3-19. Theoretical reflectance vs. wavelength for an ideal eight layer quarter wave stack of TiO$_2$ and SiO$_2$ materials on a glass substrate. Reference wavelength = 600nm. Maximum reflectance ($R_{max}$) = 92.8%. Coefficient of Finesse (F) = 716.](image)
We measured B.I.G. Fabry-Perot sample #2’s transmittance versus wavelength with a spectrometer. A transmittance peak of 27% was detected at a wavelength of 665nm. Using a red HeNe laser, B.I.G. Fabry-Perot sample #2’s reflectance and transmittance versus tilt angle were also measured. It was observed that s-polarized light required approximately 42° of tilt to achieve resonance, while p-polarized light resonance required approximately 35° of tilt. This Fabry-Perot thin film design was entered into the “MACLEOD Thin Film Design Software” program (Macleod, 1994). The cavity material’s optical constants (n, k, t) were adjusted until all three measurement results were duplicated. Figure 3-20 shows the measured and calculated spectral transmission versus wavelength curve.

![Figure 3-20. B.I.G. Fabry-Perot #2 transmittance versus wavelength at normal incidence. Calculated curve based on n = 2.3, k = 0.013, t = 462nm.](image)
Figures 3-21(a) and 3-21(b) show the measured transmittance peaks and reflectance dips caused by the resonances of s- and p-polarized light. The fitted curves are calculated with \( n = 2.3, \ k = 0.014, \text{ and } t = 462\text{nm} \). Notice that sample BIG-2000-650-180 had a thickness of 209nm while sample BIG-2000-650-180x2 has a thickness of 462nm. Since both samples used the same B.I.G. solution mixture, we would expect the two layer structure to have twice the thickness of the single layer. But given that the B.I.G. mixture was stored for approximately one month between spin coatings, and viscosity tends to increase with time, the increase in thickness is not unexpected.

Figure 3-21. BIG FP #2 measured and calculated reflectance/transmittance of s- and p-polarized components versus angle of incidence. (a) Transmittance peaks. (b) Reflectance dips. Theoretical curves are based on \( \lambda = 632.8\text{nm}, \ n = 2.3, \ k = 0.014, \ t = 462\text{nm} \).
<table>
<thead>
<tr>
<th>Measurement</th>
<th>Index of refraction (n)</th>
<th>Absorption coefficient (k)</th>
<th>Film Thickness (t)</th>
<th>Measurement wavelength (λ)</th>
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<td>k = 0.012</td>
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<td>t = 462nm</td>
<td>λ = 667nm</td>
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<td>k = 0.014</td>
<td>t = 462nm</td>
<td>λ = 633nm</td>
</tr>
</tbody>
</table>

Table 3-2. Optical constant measurement summary of two B.I.G. Fabry-Perot samples.

From the summary table 3-2 we see that B.I.G. has an absorption coefficient of $k = 0.0014$ for both samples at $\lambda = 633$nm. As the wavelength increases, the absorption coefficient tends to decrease (i.e., $k = 0.012$ at $\lambda = 705$nm). The refractive index and thickness differences between samples (note that sample #2 is supposed to be twice as thick as sample #1) are probably due to viscosity differences between precursor materials. Using the thin film Fabry-Perot structure allows accurate determination of a cavity material’s n, k, and t values. The ability to accurately determine the optical absorption (k)
of sol-gel processed B.I.G. is of particular significance. Normal ellipsometry techniques are rather poor at determining B.I.G.'s absorption coefficient.

### 3.2.3 MO EFFECT ENHANCEMENTS

This subsection shows the measured, calculated, and theoretical maximum B.I.G. Faraday rotation enhancements induced by placing a magneto-optic (MO) material (BIG) in the cavity of a thin film Fabry-Perot structure. (B.I.G. Fabry-Perot sample #2).

An interesting feature of a Fabry-Perot structure is its ability to enhance the magneto-optical Faraday rotation. We first measured a B.I.G. sample (on glass) to establish a reference Faraday rotation value. The reference sample and the B.I.G. Fabry-Perot sample #2 were made from the same solution, spin coated at the same speed, and annealed at the same temperature for the same duration. Figure 3-22 shows the measured Faraday loop from the BIG reference sample (incident angle was 36°).

![Faraday Rotation Measurement](image)

**Figure 3-22.** Reference sample (BIG-2000-650-180) Faraday rotation measurement. Sample on glass substrate (n=1.52) using a red HeNe laser with s-polarized incident light. This measurement established a reference Faraday rotation angle of 0.125°. Film optical constants were estimated to be $n = 2.1$, $k = 0.03$, and $t = 217$nm.
The Fabry-Perot sample #2 was used for all MO enhancement measurements reported in this section. From Figure 3-21 we observe that p-polarized red HeNe light is resonant at 36° angle of incidence, while s-polarized light is resonant at 42°. Figures 3-23 show the measured Faraday hysteresis loops for s- and p-polarizations at 36°, 40°, and 42° angles of incidence. These measurements show a maximum enhancement of 1.8° at 36° angle of incidence using s-polarized light. This 1.8° rotation represents a 14 times improvement in rotation over the reference rotation value (of 0.125°).

**Figure 3-23.** Fabry-Perot MO (Faraday rotation) enhancement measurements using red HeNe laser. (a) p-polarized light incident at 36°, (b) p-polarized light incident at 42°, (c) s-polarized light incident at 36°, (d) p-polarized light incident at 40°.
Using the MULTILAYER program (Mansuripur, 1990), we constructed a model of the B.I.G. Fabry-Perot sample #2 structure. The cavity material’s dielectric tensor elements (diagonal and off diagonal) were adjusted to give comparable transmittance and Faraday rotation values to the measured values. [The MULTILAYER program gives results in amplitude transmittance \( t \). All transmittances presented here are power transmittance \( T \), where \( T = |t|^2 \). Therefore all transmittance results from the MULTILAYER program have been converted to power transmittance (\( T \)) before being shown]. Figure 3-24 shows the calculated and measured transmittance and Faraday rotations based on this model. Notice that the rotation enhancement maxima for s- and p-polarizations occur at the resonant conditions of the opposite polarization component [i.e., maximum rotation occurs when non-resonant polarized incident light (low transmittance) generates an internally resonant component]. Unfortunately, this maximum rotation condition is associated with small amounts of transmitted light.

**Figure 3-24.** Modeled BIG Fabry-Perot #2 structure with diagonal dielectric tensor element corresponding to \( n = 2.3 \), \( k = 0.014 \), and off-diagonal element \( \varepsilon_{xy} = -0.00072 + 0.0032i \). (a) Calculated transmittance. (b) Calculated and measured Faraday rotation angles.
3.2.4 MAXIMUM THEORETICAL FARADAY ROTATION ENHANCEMENTS

The previous measured and calculated Faraday rotation enhancements are based on a Fabry-Perot structure consisting of 8 layer QWS mirrors, which gives a coefficient of finesse (F) of 716 at $\lambda = 600$nm. If we increase the finesse of the Fabry-Perot structure, we would expect an even greater rotation enhancement. Using the MULTILAYER program, the theoretical maximum rotation enhancement is readily calculated. For simplicity, we set $k = 0$ (no absorption), and increase the cavity’s coefficient of finesse (F) to $4.0 \times 10^{10}$ (this corresponds to two QWS mirror reflectances of $R = 99.999\%$). Using $\text{TiO}_2$ and $\text{SiO}_2$ as the QWS layer films, each QWS would have 27 layers). Figure 3-25(a) shows that linearly polarized light at normal incidence causes a split in the transmission envelope. Figure 3-25(b) shows a maximum ellipticity approaching $\pm 45^\circ$ at two nearby resonance frequencies. A peak transmittance of less that 100% is due to the index mismatch between the incident medium (air) and the substrate material (glass).
Figure 3-25. Theoretical effects of a high finesse Fabry-Perot resonator with MO cavity material. (a) transmittance at normal incidence versus wavelength. (b) Faraday rotation and ellipticity versus wavelength. Cavity material’s OPL = 598nm, absorption coefficient \( k = 0 \). Off-diagonal element \( \epsilon_{xy} = 0 + 0.02i \).

Explanation of Figure 3-25: Linearly polarized light is the superposition of right circularly polarized (RCP) and left circularly polarized (LCP) light. If linearly polarized light is incident on a Fabry-Perot at normal incidence, the transmitted light will again be a combination of RCP and LCP light. If the cavity material does not couple polarization states (i.e., material’s dielectric tensor has all zero off-diagonal elements), then the RCP and LCP eigenstates are resonant at the same wavelength, and produce a single transmission peak of linearly polarized light. But, if the cavity material does couple polarization (e.g., non-zero off-diagonal tensor elements), as in the case with magneto-optic materials, the RCP and LCP eigenstates have slightly different resonant wavelengths. This difference in resonance wavelength splits the transmittance curve into two peaks (each peak is associated with either the RCP or LCP eigenstate). The greater
the MO coupling (off-diagonal tensor element), the greater will be the split. Figures 3-26(a) and (b) show the transmitted spectral envelope when incident light is RCP and LCP respectively for a particular magnetic state. From these plots we see that RCP and LCP are resonant at different wavelengths. This is a result of non-zero off-diagonal elements, \( \varepsilon_{xy} \) that causes RCP and LCP to see different refractive indices. Figures 3-27(a) and (b) show that when the sign of the off-diagonal elements reverses (i.e., magnetic state is reversed) the spectral shifts associated with RCP and LCP light are reversed (i.e., RCP resonance wavelength trades places with the LCP resonance wavelength). These theoretical results are, of course associated with extremely high finesse of the cavity and zero absorption of the cavity material.

**Figure 3-26.** High finesse, zero absorption Fabry-Perot transmittance versus wavelength. Cavity's dielectric tensor off-diagonal element \( \varepsilon_{xy} = 0.0 + 0.013i \) corresponding to up magnetic state. (a) RCP incident light. (b) LCP incident light.
Figure 3-27. High finesse, zero absorption Fabry-Perot transmittance versus wavelength. Cavity’s dielectric tensor off-diagonal element $\varepsilon_{xy} = 0 - 0.013i$ corresponding to down magnetic state. (a) RCP incident light. (b) LCP incident light.

### 3.2.5 REALISTIC MAXIMUM FARADAY ROTATION ENHANCEMENT

Any real Fabry-Perot thin film structure will contain some optical absorption that will severely limit the maximum amount of MO enhancement. This section tries to give an indication of how much rotation enhancement is achievable (at $\lambda = 633$nm) and at what expense (i.e., how much transmittance to expect). To this end, we modeled a 43 layer thin film structure on glass substrate consisting of 21 layer quarter wave stacks of TiO$_2$ and SiO$_2$ (mirrors, $\lambda_c = 616.5$nm) and cavity layer of B.I.G. material ($n = 2.3$, $k = 0.01$, $t = 280$nm). The off-diagonal dielectric tensor elements were given values corresponding to the largest values measured (to date) for any B.I.G. sample (i.e., $\varepsilon_{xy} = 0.003$, + 0.013i).
We simulated this structure using the MULTILAYER program and obtained the following results. Figure 3-28(a) shows the transmittances associated with RCP and LCP incident light. Figures 3-28(b) and (c) show the simulated structure's Faraday rotation angle and ellipticity at normal incidence.

![Graphs of transmittance, Faraday rotation, and ellipticity](image)

**Figure 3-28.** Maximum practical Faraday rotation enhancement from a 43 layer Fabry-Perot thin film structure (Finesse = 2.5 x 10^7) versus wavelength. Cavity's dielectric tensor values: \( \varepsilon_{xx} = \varepsilon_{yy} = 5.29 + 0.02, \varepsilon_{xy} = 0.003 + 0.013, t = 280\text{nm} \). (a) RCP and LCP transmittances (b) Faraday rotation, (c) ellipticity.
Key features of Faraday rotation enhancement results are:

1. The two spectral envelopes have partially separated and are distinguishable.

2. The transmittances are very small (i.e., $T = 0.08\%$) and would be difficult to measure without special equipment.

3. The maximum Faraday rotation is about $33^\circ$. This is substantially larger than the $2^\circ$ rotation measured from the B.I.G. Fabry-Perot #2 sample, but much less than the $90^\circ$ theoretical maximum rotation.
3.3 SUMMARY

This chapter has dealt with various optical and magneto-optical characteristics of spin coated, sol-gel derived bismuth doped dysprosium iron garnet (B.I.G.) thin films. With a spin speed study we showed that B.I.G. thin film thickness exponentially decreases with spin speed while refractive index increases. In the same study, it was shown that magnetic coercivity quadratically increases with spin speed while Faraday rotation quadratically decreases. From an annealing study, we showed that as the annealing temperature or duration is increased B.I.G. thin film thickness tends to decrease while refractive index tends to increase (annealing temperatures ranged from 600°C to 700°C, while annealing durations ranged from 1 minute to 5 hours). It was also pointed out that stored sol-gel solutions will become more viscous over time. After explaining the basic characteristics of thin film Fabry-Perot structures, we demonstrated how to accurately determine the optical constants (n, k, t) for a Fabry-Perot cavity material (in this case B.I.G.) by combining spectroscopic transmission measurements and analysis with tilted reflectance and transmittance measurements and analysis. Finally, we presented measured and theoretical maximum enhancements of magneto-optical effects by placing magneto-optical materials in the cavity of a thin film Fabry-Perot structure.
CHAPTER 4

CHARACTERIZATION OF SOL-GEL DERIVED PZT/PLZT THIN FILMS

This chapter shows various optical and electro-optical characterization results (using the Ultimate Ellipsometer) from PZT and PLZT thin films prepared by a spin coating sol-gel process. The optical properties (n and t) of these films are readily determined by depositing them on a glass substrate and making a set of ellipsometric beam extinction measurements. We used a red HeNe laser (λ = 632.8nm) for all ellipsometric (polarization rotation and ellipticity), reflectance (R), and transmittance (T) measurements. Since refractive index is a function of wavelength, all references to refractive index (n) in this chapter are understood to be at λ = 632.8nm unless otherwise stated. Beam extinction ellipsometry gives good estimates for index of refraction (n) and film thickness (t). Unfortunately, it does not give consistently good estimates of the material’s absorption coefficient (k). To get a good estimate of k, we have constructed a Fabry-Perot thin film structure with PZT as the cavity material. We then measured the transmittance (T) as a function of wavelength. This gives us a fairly good estimate of the cavity’s optical constants (n,k,t). By combining the spectroscopic results with transmittance (T) and reflectance (R) versus angle of incidence measurements, we are able to get a good estimate of all three optical constants (n,k,t). Measuring the electro-optic (EO) properties requires a rather complicated sample structure. Since an electric
field must be present across the material to generate an EO effect, a pair of electrodes were deposited on each side of the EO film (i.e., sandwiching the EO material between electrodes). The additional complexity of having electrodes in the test setup, requires an analysis of these electrodes as well. Appendix B shows the ellipsometric measurement data and analysis results for the transparent electrodes (ITO and SnO).

Our collaborators at Donnelly Corporation prepared several sets of samples of both PZT(53/47) and PLZT(28/0/100) on Corning 7059 glass substrates using their sol-gel process. Since all PLZT samples studied here were devoid of zirconate, these samples are designated as PLT. The PZT and PLT samples of this chapter will be designated as follows: type(PZT or PLT)-spin speed(rpm)-annealing temperature(°C)-duration (minutes) e.g., PZT-2000-600-30.

4.1 PLT AND PZT ON GLASS

This subsection gives results from process variation studies of PZT and PLT sol-gel derived thin film samples. The first study looks at how PZT film thickness (t) and index of refraction (n) vary with spin coating speed. The next study looks at how PZT and PLT’s optical constants (n,k,t) are affected by different annealing temperatures. Figure 4-1 shows the basic structure of PZT or PLT thin film samples on glass substrate.
4.1.1 PZT SPIN SPEED STUDY

In this subsection we show the measured ellipsometric data and optical constant (n,k,t) analysis results from five PZT samples spun at varying speeds ranging from 1000 rpm to 3000 rpm. After spin coating, each sample was annealed at 600°C for 30 minutes. Therefore these samples are designated as PZT-xxxx-600-30 where xxxx corresponds to the particular spin speed.
Figures 4-2a through 4-2e show both the measured and fitted (least squares fit) ellipsometric (rotation and ellipticity) data for five PZT samples. The assumed index of the 7059 Corning glass substrate was $n = 1.52$. The incident polarization ($\theta_{\text{pol}}$) was at $30^\circ$. Also included are the analysis results assuming an absorption coefficient ($k$) of zero. Except for sample PZT-1000rpm-600C-30min, the fitted values of $k$ can range from 0.00 to 0.10 without any significant effects on the analysis results. This lack of absorption coefficient sensitivity prevents us from reliably determining this quantity ($k$) using null ellipsometry. Sample PZT-1000rpm-600C-30min is somewhat cloudy which indicates excessive scattering. Excessive scattering would explain this sample’s high fitted value of $k$ (i.e., $k = 0.40$).
Measured rotation
○ Measured ellipticity
Fitted data
PZT-1000-600-30

Rotation / Ellipticity (degrees)

Incident Angle

Rotation / Ellipticity (degrees)

Incident Angle

Rotation / Ellipticity (degrees)

Incident Angle

Rotation / Ellipticity (degrees)

Incident Angle

Measured rotation
○ Measured ellipticity
Fitted data
PZT-1500-600-30

Measured rotation
○ Measured ellipticity
Fitted data
PZT-2000-600-30

Measured rotation
○ Measured ellipticity
Fitted data
PZT-2500-600-30
Figure 4-2. Ellipsometric measurements (at $\lambda = 632.8$nm) and fits from PZT spin speed study samples.

(a) PZT-1000rpm-600C-30min; Analysis results: $n = 1.80$, $k = 0.40$, $t = 310\text{nm}$, $\text{err} = 0.60^\circ$

(b) PZT-1500rpm-600C-30min; Analysis results: $n = 2.03$, $k = 0.04$, $t = 291\text{nm}$, $\text{err} = 0.46^\circ$

(c) PZT-2000rpm-600C-30min; Analysis results: $n = 2.03$, $k = 0.07$, $t = 266\text{nm}$, $\text{err} = 0.45^\circ$

(d) PZT-2500rpm-600C-30min; Analysis results: $n = 2.00$, $k = 0.06$, $t = 256\text{nm}$, $\text{err} = 0.55^\circ$

(e) PZT-3000rpm-600C-30min; Analysis results: $n = 2.25$, $k = 0.08$, $t = 245\text{nm}$, $\text{err} = 0.55^\circ$

4.1.1.2 GENERAL TRENDS OF SPIN SPEED STUDY

Figures 4-3a and 4-3b show that in general, index of refraction ($n$) increases and thickness ($t$) decreases with increasing spin speed. As explained in chapter 3 (section 3.1.1.3) these trends are to be expected. Since the optical absorption coefficient ($k$) obtained by
ellipsometric measurements is unreliable, no absorption trends are considered in this study.

Figure 4-3. PZT spin speed study general trends (a) index of refraction (at $\lambda = 632.8$nm) versus spin speed. (b) sample thickness versus spin speed.

4.1.2 PZT / PLT ANNEALING STUDY

This study looked at how annealing temperature affects PZT and PLT’s optical constants ($n$, $t$). The absorption coefficient ($k$) is assumed to be zero for analysis purposes. Three samples of PZT(47/53) and three samples of PLT(28/0/100) were spin coated on Corning 7059 glass substrates. All six samples were spun at 2000 rpm and annealed for three hours. The PZT samples are identified as PZT-2000-xxx-180, while the PLT samples are referred to as PLT-2000-xxx-180 (where xxx = 500, 550, or 600).
4.1.2.1 ELLIPSOMETRIC MEASUREMENTS AND ANALYSIS

Figures 4-4 and 4-5 show the measured and fitted rotation and ellipticity of the PZT and PLT samples respectively. The assumed index of refraction of the 7059 Corning glass substrate is $n = 1.52$. The incident polarization ($\theta_{\text{pol}}$) was again set to $30^\circ$.

![Figure 4-4. PZT annealing study ellipsometric measurements at $\lambda = 632.8$nm): (a) PZT-2000-500-180; $n = 2.19$, $k = 0.00$, $t = 360$nm, err = $0.52^\circ$; (b) PZT-2000-550-180, $n = 2.13$, $k = 0.00$, $t = 303$nm, err = $0.75^\circ$; (c) PZT-2000-600-180, $n = 2.03$, $k = 0.00$, $t = 272$nm, err = $0.92^\circ$.}
Figure 4-5. PLT annealing study ellipsometric measurements (at $\lambda = 632.8\text{nm}$):  
(a) PLT-2000-500-180; $n = 2.26$, $k = 0.00$, $t = 320\text{nm}$, err $= 0.46^\circ$;  
(b) PLT-2000-550-180, $n = 2.33$, $k = 0.00$, $t = 303\text{nm}$, err $= 0.51^\circ$;  
(c) PLT-2000-600-180, $n = 2.50$, $k = 0.00$, $t = 277\text{nm}$, err $= 0.91^\circ$. 
In general, as the annealing temperature increases, thickness decreases. PZT’s index (n) decreases slightly while PLT’s index (n) increases with temperature. PZT’s slight decrease of refractive index with temperature implies that increased porosity caused by precursor extraction has a greater effect than heat induced densification. Again optical absorption coefficient (k) trends are not reliable and are therefore omitted. Table 4-1 summarizes the annealing temperature results, and Figures 4-6a and 4-6b plot the trends.

<table>
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<tr>
<th>Sample</th>
<th>Index of Refraction</th>
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<td>t = 360nm</td>
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<td>t = 303nm</td>
</tr>
<tr>
<td>PZT-2000-600-180</td>
<td>n = 2.03</td>
<td>t = 272nm</td>
</tr>
<tr>
<td>PLT-2000-500-180</td>
<td>n = 2.26</td>
<td>t = 320nm</td>
</tr>
<tr>
<td>PLT-2000-550-180</td>
<td>n = 2.33</td>
<td>t = 303nm</td>
</tr>
<tr>
<td>PLT-2000-600-180</td>
<td>n = 2.50</td>
<td>t = 277nm</td>
</tr>
</tbody>
</table>

Table 4-1. Summary of PZT/PLT annealing study results.
Figure 4-6. PZT/PLT annealing study general trends. (PZT-2000-xxx-180 and PLT-2000-xxx-180). (a) Refractive Index (n) at $\lambda = 632.8$nm versus annealing temperature. (b) Film thickness (t) versus annealing temperature.

4.2 PZT OPTICAL CONSTANT CHARACTERIZATION USING A THIN FILM FABRY-PEROT DEVICE

This section is very similar to section 3.2.2 in that we use a thin film Fabry-Perot structure to characterize a thin film’s (PZT) optical constants ($n$, $k$, $t$). A main feature of this characterization technique is the ability to accurately determine a material’s optical absorption coefficient ($k$). As in chapter 3, we use quarter wave stacks (QWS) of titanium dioxide ($\text{TiO}_2$) and silicon dioxide ($\text{SiO}_2$) to construct the Fabry-Perot mirrors. In this chapter we use two eleven layer quarter wave stacks ($\lambda_c = 632.8$nm) as the mirrors. Figure 4-7 shows the calculated reflectance versus wavelength for one of the eleven layer stacks.
The maximum calculated reflectance of the mirrors is 98.64%. The Fabry-Perot cavity material consists of two layers of PZT spun at 2000 rpm and annealed at 600°C for 3 hours (i.e., PZT-2000-600-180).

Figure 4-7. Calculated reflectance vs. wavelength for an eleven layer quarter wave stack of TiO₂ and SiO₂ on a glass substrate. Reference wavelength (λ₀) = 633nm. Maximum reflectance R_{max} = 98.64%. Coefficient of Finesse (F) = 21,300.

Figure 4-8 shows a schematic diagram of the composite thin film PZT Fabry-Perot structure (i.e., QWS on PZT on QWS on glass substrate) considered in this section.

**Figure 4-8.** Schematic diagram of a thin film PZT Fabry-Perot structure.
We first measured the sample's transmittance (T) versus incident wavelength (\(\lambda\)). Based on this spectroscopic measurement, we used MACLEOD's thin film software to estimate the cavity's optical constants (n,k,t). We then measured the transmittance and reflectance at \(\lambda = 633\text{nm}\) versus angle of incidence. This gives us three separate sets of measurements from which to determine the cavity's optical constants. Adjusting the optical constants, we were able to obtain a consistent fit for all three sets of measurements. Figure 4-9 shows the measured spectroscopic transmittance and calculated fit. The calculated fit is based on a refractive index (n) and an absorption coefficient (k) that are independent of wavelength. In reality, both of these optical constants (n,k) change somewhat with wavelength variations. Since the tilted reflectance and transmittance measurements are taken at \(\lambda = 632.8\text{nm}\), we matched the spectroscopic transmittance (T) around \(\lambda = 633\text{nm}\). The spectroscopic transmittance curve shows a transmission peak at 635nm with a transmittance of 14.5%. This is the transmission peak used to fit the absorption coefficient (k = 0.003). It is worth noting that using this value of k, the fitted transmission peak at \(\lambda = 738\text{nm}\) (T = 66%) also agrees closely with the measured transmittance (T = 65%). While, the calculated peak transmittance at \(\lambda = 555\text{nm}\) (T = 55%) is about 20 percent larger than the measured transmittance (T = 44%). This indicates that absorption is fairly constant (k = 0.003) at wavelengths above 600nm. At wavelengths below 600nm, the coefficient of absorption begins to increase. Figure 4-10 shows the results of transmittance versus angle of incidence measurements. Figure 4-11
shows the results of reflectance versus angle of incidence measurements. All calculated fits are based on cavity material’s optical constants of \( n = 2.10, k = 0.003, t = 603\text{nm} \).

**Figure 4-9.** PZT Fabry-Perot spectroscopic transmittance measurement and fit \( (n = 2.10, k = 0.003, t = 603\text{nm}) \).

**Figure 4-10.** PZT Fabry-Perot angular transmittance measurement and fit \( (p\text{-polarization}, \lambda = 632.8\text{nm}, n = 2.10, k = 0.003, t = 603\text{nm}) \).
The Fabry-Perot structure is very sensitive to optical constant changes (i.e., n,k,t), and especially so for thickness changes. A one percent change in thickness will significantly change all three sets of measurements. For instance, Figures 4-12(a) and 4-12(b) show the effect on transmittance (spectral and angular) of changing the thickness from 603nm to 610nm (i.e., a 1% change). Figure 4-13 shows the effects on transmittance of a ten percent change in refractive index. A 10% change of refractive index from $n = 2.10$ to $n = 2.31$ causes a 38% change in peak spectral transmittance (from 13% to 18%) at $\lambda = 633$nm.
Figure 4-12. PZT Fabry-Perot transmittance measurement and 1 percent thickness fit error (i.e., \( n = 2.10 \), \( k = 0.003 \), \( t = 610\text{nm} \)). (a) spectral transmittance. (b) angular transmittance (p-polarization at \( \lambda = 632.8\text{nm} \)).

Figure 4-13. PZT Fabry-Perot spectral transmittance measurement and 10 percent refractive index fit error (i.e., \( n = 2.31 \), \( k = 0.003 \), \( t = 603\text{nm} \)).
Figure 4-14 shows that a 17% change in absorption coefficient from $k = 0.003$ to $k = 0.0025$ produces a 30% change in peak spectral transmittance (from 13% to 17%).

![Figure 4-14](image)

**Figure 4-14.** PZT Fabry-Perot spectral transmittance measurement and absorption fit error of $\Delta k = 0.0005$ (i.e., $n = 2.10$, $k = 0.0025$, $t = 603$ nm).

Using a thin film Fabry-Perot structure to measure a material's optical absorption ($k$) may be impractical in many cases. But, it is an accurate method, and in some cases may be the only reliable method of estimating the value of $k$. 
4.3 PZT/PLT ELECTRO-OPTIC COEFFICIENT ($\Delta n$, $r_{\text{eff}}$) MEASUREMENTS

To measure a material’s EO coefficient ($r_{\text{eff}}$), an electric field must be applied across the material. To establish this electric field, we have added electrodes to our thin film samples (see Figure 4-15). The EO material is sandwiched between these two electrodes. When a voltage is applied to the electrodes, an electric field is developed across the material. The electric field strength is proportional to the voltage difference across the electrodes and inversely proportional to the EO film thickness. Figure 4-15 shows the thin film structure used to measure PZT and PLT’s electro-optic coefficient.

Figure 4-15. Schematic diagram of PZT/PLT thin film EO measurement structure.
About the structure:

PZT and PLZT are used as EO materials because of their high electro-optic coefficients. ITO is used for its electrical conductivity and optical transparency (Salam, 1990, Gupta et al., 1989). A transparent oxide layer supplied by Donnelly Corporation acts as a barrier to indium migration during the sol-gel annealing process. If a barrier layer is not present, the indium migrates through the PZT/PLZT layer during the annealing process and shorts the two electrodes (a short prevents an electric field from forming across the EO material). Doping the tin oxide (Donnelly Corp. selected dopant material) makes it conductive (Stjerna and Granqvist, 1990), thus preventing any substantial voltage drop. Since the EO films (PZT and PLT) have extremely large dielectric constants (K = 1000 to 8000) as compared to ordinary insulating substances (K = 5 to 100) (Buchanan, 1986, Agulllo et al., 1994), any non-conducting barrier layer with a normal dielectric constant value would drop virtually all the voltage across itself and prevent any substantial field from developing across the EO material. This structure is very similar to that used by J.S. Schildkraut (Schildkraut, 1990), except that we do not use an ac voltage signal generator in conjunction with a lock-in amplifier. We simply use a dc voltage source and rely on differential detection of the polarization state of the reflected beam to measure the EO coefficient. Figure 4-16 shows the measured Δn versus applied voltage for two samples, PLT-2000-600-180 and PZT-2000-600-180. The amount of Δn is derived from the normalized differential detector signal (S12).
Note that the barrier electrode layer is an "n" type semiconductor material, while the PZT/PLZT layer is a "p" type semiconductor material. The resulting pn junction creates a space charge that explains the asymmetry of the EO curves.

Figure 4-16. Electro-optical measurements (i.e., Δn (i.e., n – nₐ) versus applied voltage (Ψᵢnc = 60°, θₚ₀l = 40°, θᵢ = 45°, θₜₚ = 45°, λ = 632.8nm). (a) PLT-2000-600-180, (b) PZT-2000-600-180.

4.3.1 EO MEASUREMENTS AND CALCULATIONS

Using the MULTILAYER program and Jones calculus as described in chapter 2 (section 2.3.5), we can readily estimate the amount of Δn required to achieve the measured ΔS12 value. Table 4-2 shows the assumed structure parameters (n,k,t), Ultimate Ellipsometer test setup (Ψᵢnc, θₚ₀l, θᵢ, θₜₚ) and calculated results (Δn, rₑff).
(a) **Sample structure:**

<table>
<thead>
<tr>
<th>Material</th>
<th>Description</th>
<th>n</th>
<th>k</th>
<th>t</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate</td>
<td>Corning 7059 glass; n = 1.52, k = 0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Transparent electrode</td>
<td>ITO; n = 2.033, k = 0.011, t = 200nm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Barrier electrode</td>
<td>SnO; n = 1.81, k = 0, t = 200nm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EO material 1</td>
<td>PLT-2000-600-180; n = 2.50, k = 0.00, t = 600nm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EO material 2</td>
<td>PZT-2000-600-180; n = 2.10, k = 0.003, t = 600nm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Top electrode</td>
<td>Gold/Palladium; n = 1.00, k = 4.00, t = 158nm</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(b) **Ellipsometer Setup**

<table>
<thead>
<tr>
<th>Ellipsometer setup</th>
<th>λ = 632.8nm, Ψ_{inc} = 60°, Θ_{pol} = 40°, Θ_{F} = 45°, Θ_{w} = 45°</th>
</tr>
</thead>
</table>

(c) **Resultant Δn and r_{eff} calculations**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Applied voltage</th>
<th>ΔS_{12}</th>
<th>Δn</th>
<th>r_{eff}</th>
</tr>
</thead>
<tbody>
<tr>
<td>PLT-2000-600-180</td>
<td>26 volts</td>
<td>0.008</td>
<td>0.015</td>
<td>59 pm/volt</td>
</tr>
<tr>
<td>PZT-2000-600-180</td>
<td>21 volts</td>
<td>0.026</td>
<td>0.051</td>
<td>315 pm/volt</td>
</tr>
</tbody>
</table>

Table 4-2. PZT/PLT EO measurement summary (a) sample structure (b) test setup, (c) calculated results (Δn, r_{eff}). The measured samples contain either PLT or PZT but not both.

The values for Δn (and consequently for r_{eff}) are a combination of an electro-optic (Δn) effect and an electrostrictive (Δt) effect. Our sample composition of PLT is PLZT(28/0/100). This particular composition seldom shows any significant electrostrictive effect. Thus our Δn and r_{eff} values for PLT are primarily electro-optics values. This is not necessarily the case with PZT. PZT typically exhibits both a large
electro-optic and a large electrostrictive effect. We have not attempted to separate these
effects and caution against misinterpreting these PZT values of $\Delta n$ and $r_{\text{eff}}$. 
4.3.2 CALCULATED $\Delta n$ SENSITIVITIES

The previously shown calculated values of $\Delta n$ for PZT and PLT (Table 4-2) are based on a multilayer structure (Figure 4-15) where each thin film layer has an assumed set of optical constants (n,k,t). These assumed values are based on analysis fits of PZT, PLT, ITO, SnO, and Gold/Palladium ellipsometric measurements. To see how sensitive $\Delta n$ is to thin film refractive index and thickness variations, we recalculated $\Delta n$ after making various thickness and refractive index changes to the PZT sample structure. Table 4-3 summarizes these sample layer variations on $\Delta n$ calculations. We see that $\Delta n$ is insensitive to thickness variations (in the non EO layers), and only slightly sensitive to refractive index changes.

<table>
<thead>
<tr>
<th>PZT sample variation</th>
<th>Normalized differential signal ($\Delta S_{12} = 0.026$)</th>
<th>$\Delta n$</th>
</tr>
</thead>
<tbody>
<tr>
<td>PZT-base structure (from Table 4-3)</td>
<td>0.026</td>
<td>0.051</td>
</tr>
<tr>
<td>ITO 10% thickness change from $t = 200$nm to $t = 180$nm</td>
<td>0.026</td>
<td>0.050 (2% change)</td>
</tr>
<tr>
<td>SnO 10% thickness change from $t = 200$nm to $t = 180$nm</td>
<td>0.026</td>
<td>0.050 (2% change)</td>
</tr>
<tr>
<td>ITO 5% refractive index change from $n = 2.03$ to $n = 2.13$</td>
<td>0.026</td>
<td>0.052 (2% change)</td>
</tr>
<tr>
<td>PZT 5% refractive index change from $n = 2.10$ to $n = 2.20$</td>
<td>0.026</td>
<td>0.056 (10% change)</td>
</tr>
</tbody>
</table>

Table 4-3. Calculated $\Delta n$ and percent change from sample layer thickness and index ($n$) variations.
4.4 SUMMARY

This chapter has dealt with various optical and electro-optical characteristics of spin coated sol-gel derived PZT and PLZT thin films. With a spin speed study we showed that PZT thin film refractive index increases and the film thickness decreases with increasing spin speed. From an annealing temperature study, we showed that as the annealing temperature is increased (from 500°C to 600°C) PLT and PZT thin films tend to decrease in thickness, while PLT films increased their refractive index and PZT films show a slight decrease in refractive index. We demonstrated the ability to accurately measure the absorption coefficient (k), of a EO material (PLT/PZT) when deposited as the cavity of a thin film Fabry-Perot structure. Finally, we presented results of dc applied voltage electro-optic measurements. These electro-optic (Δn and r_eff) calculations are based of the measured differential detector signal (ΔS12), a MULTILAYER model of the sample under test, and a Jones calculus analysis of the Ultimate Ellipsometer optics.
APPENDIX A

JONES CALCULUS ANALYSIS OF THE ULTIMATE ELLIPSOMETER

The Ultimate Ellipsometer is a polarization sensitive device, in that a beam’s polarization state changes as it propagates through the system. To analyze these changes, we need a simple but powerful polarization analysis tool. To this end, we will use Jones calculus.

JONES CALCULUS:

Jones calculus uses column vectors (Jones vectors) to describe the state of polarization at a given cross-section of the system. Equation A-1 is a Jones vector representation of a coherent, monochromatic light beam with electric field amplitude components $E_x$ and $E_y$ at a location associated with subscript $0$.

\[
E = \begin{bmatrix}
E_{0x} e^{i\phi_x} \\
E_{0y} e^{i\phi_y}
\end{bmatrix}
\]  

(A-1)

The following are examples of Jones vectors representing various polarization states (all vectors have been normalized).
**State of polarization** | **Jones vector**
---|---
Horizontal p-polarization | $\begin{bmatrix} 1 \\ 0 \end{bmatrix}$
Vertical s-polarization | $\begin{bmatrix} 0 \\ 1 \end{bmatrix}$
Right circular polarization (RCP) | $\frac{1}{\sqrt{2}} \begin{bmatrix} 1 \\ -i \end{bmatrix}$
Left circular polarization (LCP) | $\frac{1}{\sqrt{2}} \begin{bmatrix} 1 \\ i \end{bmatrix}$

**TRANSFORMATION OF POLARIZATION STATES** (transformation matrices):

The laser beam’s state of polarization changes as it propagates through the ellipsometer. To account for these changes, the Jones vector is multiplied by a set of two-by-two transformation matrices. In general, the transformation matrix elements associated with a particular optical component depend on both the component’s optical properties and its relative orientation. To simplify matters, we will treat these two conditions separately. Each optical component will have two simple transformation matrices. The first of these matrices will be a rotation matrix that will account for the relative orientation of the component. The second matrix will account for the component’s effect on the state of
polarization at a standard orientation (we will usually assume a horizontal or “x” standard orientation).

**ROTATION MATRIX**

We define the rotation matrix $R$ as a matrix that compensates for component orientation differences. Assume that the $xy$ coordinate system rotates in a counter clockwise direction ($+\theta$) to make a new coordinate system ($x'$-$y'$). Then the relationship between the two coordinate systems is represented by the following rotation matrix.

**Figure A-1.** Rotation of the $xy$ coordinate system. Positive $\theta$ corresponds to a counterclockwise rotation. (the beam is propagating out of the plane of the page).
\[ R(\theta) = \begin{bmatrix} \cos\theta & -\sin\theta \\ \sin\theta & \cos\theta \end{bmatrix} \]

Note: \[ \begin{bmatrix} x' \\ y' \end{bmatrix} = R(\theta) \times \begin{bmatrix} x \\ y \end{bmatrix} \]

**Jones Matrices for the different Ultimate Ellipsometer optical elements**

<table>
<thead>
<tr>
<th>Optical Element</th>
<th>Jones Matrix ((M_i))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Horizontal (p-pol) linear polarizer</td>
<td>[ \begin{bmatrix} 1 &amp; 0 \ 0 &amp; 0 \end{bmatrix} ]</td>
</tr>
<tr>
<td>(e.g., ASP, PBS(p-pol), Wollaston((\xi)-pol))</td>
<td></td>
</tr>
<tr>
<td>Vertical (s-pol) linear polarizer</td>
<td>[ \begin{bmatrix} 0 &amp; 0 \ 0 &amp; 1 \end{bmatrix} ]</td>
</tr>
<tr>
<td>(e.g., PBS(s-pol), Wollaston((\eta)-pol))</td>
<td></td>
</tr>
<tr>
<td>Linear Polarizer at (+45^\circ)</td>
<td>[ \frac{1}{\sqrt{2}} \begin{bmatrix} 1 &amp; 1 \ 1 &amp; 1 \end{bmatrix} ]</td>
</tr>
<tr>
<td>Quarter wave plate (fast axis horizontal)</td>
<td>[ \begin{bmatrix} 1 &amp; 0 \ 0 &amp; i \end{bmatrix} ]</td>
</tr>
<tr>
<td>(e.g., Fresnel rhombs FR1 &amp; FR2)</td>
<td></td>
</tr>
</tbody>
</table>
To find the resultant state of polarization we multiply an initial Jones Vector by a rotation matrix \((R_1)\) and then by an optical component matrix \((M_1)\). The result is a second Jones vector representing the state of polarization at that particular location. With this new Jones vector we can repeat the process for the next optical component. At each step the resultant Jones vector will give the beam’s current state of polarization. To find the resultant power at a particular location we square the magnitude of the Jones vector.

Resultant Jones Vector \((V_r) = M_4 R_4 M_3 R_3 M_2 R_2 M_1 R_1 V_0\) Where \(V_0 = \) initial Jones Vector

Resultant Power \(= |V_r|^2\)

**Jones Calculus Analysis of Ultimate Ellipsometer Laser Arm**

The logical starting point for polarization analysis is the state of polarization exiting the laser. We will begin our analysis assuming the presence of a linearly polarized laser with its transmission axis at \(+45^\circ\) from horizontal. This transmission axis defines our initial coordinate axes. Therefore we will start with a Jones vector \((V_0)\) that represents linearly polarized light. Keep in mind that our reference axis is at \(+45^\circ\) from the horizontal. The light first propagates to an air spaced polarizer (ASP) which also has its transmission axis rotated \(+45^\circ\) from the horizontal. Since the relative rotation angle is equal to zero \(\theta_{ASP} = 0\), our first rotation matrix is simply an identity matrix (i.e., \(V_1 = V_0\)). In fact,
linearly polarized light aligned along the transmission axis of a linear polarizer should not have its polarization state affected at all. (Note: this linear polarizer does have a purpose. It ensures "clean" linearly polarized light even if the laser is slightly elliptically polarized).

\[ V_1 = M_1 \quad R_1 \quad V_0 \]
\[
\begin{bmatrix}
1 \\
0
\end{bmatrix} =
\begin{bmatrix}
1 & 0 \\
0 & 0
\end{bmatrix} \cdot
\begin{bmatrix}
1 \\
0
\end{bmatrix} \cdot
\begin{bmatrix}
1 \\
0
\end{bmatrix}
\]

The next component encountered is a Fresnel rhomb (FR1) which acts as a quarter wave phase retarder. The Fresnel rhomb's fast axis is fixed in a vertical position. This corresponds to a relative rotation angle of $+45^\circ$. The resulting state of polarization ($V_2$) is left circularly polarized as shown below.

\[ V_2 = M_2 \quad R_2 \quad V_1 \]
\[
\frac{1}{\sqrt{2}}
\begin{bmatrix}
1 \\
i
\end{bmatrix} =
\begin{bmatrix}
1 & 0 \\
i & i
\end{bmatrix} \cdot
\begin{bmatrix}
1 & -1 \\
\sqrt{2} & -\sqrt{2}
\end{bmatrix} \cdot
\begin{bmatrix}
1 \\
0
\end{bmatrix}
\]

Figure A-2 shows the optical components and their angular relationships as the laser beam propagates through the Ultimate Ellipsometer.
Figure A-2. Angular relationship among the various components of the Ultimate Ellipsometer.

This left circularly polarized light ($V_2$) next encounters a polarizing beam splitter cube (PBS) which is mounted on a rotation stage (RS2) that can rotate a full $360^\circ$. A graduated scale on RS2 shows the value of $\theta_{pol}$ upon its counterclockwise rotation. The PBS cube splits the beam into two orthogonal components, corresponding to s-polarization and p-polarization. The s-pol and p-pol components are with respect to the PBS wedge surface. The s-pol component is diverted to the photodetector PD0, while the p-pol component passes straight through the PBS cube. Note that both component beams are linearly polarized. Since the incident light is circularly polarized, each linearly polarized
output beam has the same amplitude. The relative rotation angle $\theta_{pol}$ is a variable parameter that can be chosen according to the needs of each particular experiment.

Beam 1 (p-pol):

$$
\begin{align*}
V_3 &= M_2 \ R_2 (CW) \ V_2 \\
&= \frac{1}{\sqrt{2}} \begin{bmatrix}
\cos \theta_{pol} + i \sin \theta_{pol} \\
0
\end{bmatrix} \begin{bmatrix}
1 & 0 \\
0 & 0
\end{bmatrix} \begin{bmatrix}
\cos \theta_{pol} & \sin \theta_{pol} \\
-\sin \theta_{pol} & \cos \theta_{pol}
\end{bmatrix} \frac{1}{\sqrt{2}} \begin{bmatrix}
1 \\
i
\end{bmatrix}
\end{align*}
$$

Beam 2 (s-pol):

$$
\begin{align*}
V_3 &= M_2 \ R_2 (CW) \ V_2 \\
&= \frac{1}{\sqrt{2}} \begin{bmatrix}
0 \\
-\sin \theta_{pol} + i \cos \theta_{pol}
\end{bmatrix} \begin{bmatrix}
0 & 0 \\
0 & 1
\end{bmatrix} \begin{bmatrix}
\cos \theta_{pol} & \sin \theta_{pol} \\
-\sin \theta_{pol} & \cos \theta_{pol}
\end{bmatrix} \frac{1}{\sqrt{2}} \begin{bmatrix}
1 \\
i
\end{bmatrix}
\end{align*}
$$

Relative irradiance of emerging beam $= |V_3|^2 = 0.5$ for each beam.

Notice that the beam gets equally divided by the PBS cube.

Beam 1 is deflected to the detector (PDO), while beam 2 propagates to the sample holder and then on to the Detector Arm. For simplicity we will normalize $V_3$ and set the angle $\theta_{pol}$ for beam 2 to correspond to the PBS (p-pol) fast axis (i.e. $\theta_{pol} = 0^\circ$).

Thus $V_3 = \begin{bmatrix} 1 \\ 0 \end{bmatrix}$ = Linearly polarized light (Horizontal: p-pol).
**Interaction of linearly polarized beam with sample material**

To analyze the polarization effects caused by the sample material we define a transformation matrix \( \mathbf{M}_s \) for the sample. (For simplicity, we assume no cross-polarization between \( p \) and \( s \) components upon interaction with the sample, although such cross-polarization as caused by birefringence or optical activity can easily be taken into account by adding off-diagonal elements to \( \mathbf{M}_s \).

\[
\mathbf{M}_s = \begin{bmatrix}
V_x & 0 \\
0 & -V_y
\end{bmatrix}
\]

where \( V_x = r_x e^{i \delta_x} \) and \( V_y = r_y e^{i \delta_y} \)

\( V_x \) and \( V_y \) represent the Fresnel reflection coefficients of the \( p \)- and \( s \)-polarization components. \( \delta_x \) and \( \delta_y \) specify the phase shifts associated with each component. Matrix element \( V_y \) is negative due to a reversal in sign after reflection.

\[
\mathbf{V}_s = \mathbf{M}_s \mathbf{R}_{\text{pol}}(\text{CW}) \mathbf{V}_3
\]

\[
\begin{bmatrix}
V_x \cos \theta_{\text{pol}} \\
V_y \sin \theta_{\text{pol}}
\end{bmatrix} = \begin{bmatrix}
V_x & 0 \\
0 & -V_y
\end{bmatrix} \begin{bmatrix}
\cos \theta_{\text{pol}} & \sin \theta_{\text{pol}} \\
-\sin \theta_{\text{pol}} & \cos \theta_{\text{pol}}
\end{bmatrix} \begin{bmatrix}
1 \\
0
\end{bmatrix}
\]
Analysis of the Detector Arm

The first optical component the light encounters on the Detector Arm is a Fresnel rhomb (FR2), which once again, acts as a quarter wave plate (M3). Unlike the Laser Arm’s Fresnel rhomb, which was rigidly mounted, this Fresnel rhomb is mounted on a 360° rotation stage (RS3) and has a graduated scale that corresponds to a clockwise rotation for positive scale readings. The state of polarization emerging from FR2 ($V_4$) is calculated below.

$$V_4 = M_3 R_3 (\text{CW}) V_5$$

$$\begin{bmatrix} V_x \cos \theta_{pol} \cos \theta_F + V_y \sin \theta_{pol} \sin \theta_F \\ -iV_x \cos \theta_{pol} \sin \theta_F + iV_y \sin \theta_{pol} \cos \theta_F \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ 0 & i \end{bmatrix} * \begin{bmatrix} \cos \theta_F & \sin \theta_F \\ -\sin \theta_F & \cos \theta_F \end{bmatrix} * \begin{bmatrix} V_x \cos \theta_{pol} \\ V_y \sin \theta_{pol} \end{bmatrix}$$

The last optical component encountered is a Wollaston prism (WP) which is very similar to a polarizing beam splitter cube, in that the light splits into two mutually orthogonal polarization components ($\eta, \xi$). From an analysis point of view, we will treat the two components separately. The $\xi$-pol component is treated as if it were emerging from a horizontal linear polarizer ($M_{PD2}$). The $\eta$-pol component is treated as if it were emerging from a vertical linear polarizer ($M_{PD1}$). We will define $V_{PD2}$ to be the resultant state of polarization of the Wollaston’s $\eta$-pol component (this component is directed from the Wollaston prism to photodetector PD2). We will define $V_{PD1}$ to be the resultant state of
polarization of the Wollaston’s $\xi$-pol component (this component is directed from the Wollaston prism to the photodetector PD1).
\[ V_{PD1} = M_{PD1} \quad R_{PD1} \text{(CCW)} \quad V_4 \]

\[ V_{PD1} = \begin{bmatrix} 0 & 0 \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \cos \theta_w & -\sin \theta_w \\ \sin \theta_w & \cos \theta_w \end{bmatrix} \begin{bmatrix} V_x \cos \theta_{pol} \cos \theta_F + V_y \sin \theta_{pol} \sin \theta_F \\ -iV_x \cos \theta_{pol} \sin \theta_F + iV_y \sin \theta_{pol} \cos \theta_F \end{bmatrix} \]

\[ V_{PD1} = V_x \{ \cos \theta_{pol} \ [\sin \theta_w \cos \theta_F - i \cos \theta_w \sin \theta_F] \} + V_y \{ \sin \theta_{pol} \ [\sin \theta_w \sin \theta_F + i \cos \theta_w \cos \theta_F] \} \]

\[ V_{PD2} = M_{PD2} \quad R_{PD2} \text{(CCW)} \quad V_4 \]

\[ V_{PD2} = \begin{bmatrix} 1 & 0 \\ 0 & 0 \end{bmatrix} \begin{bmatrix} \cos \theta_w & -\sin \theta_w \\ \sin \theta_w & \cos \theta_w \end{bmatrix} \begin{bmatrix} V_x \cos \theta_{pol} \cos \theta_F + V_y \sin \theta_{pol} \sin \theta_F \\ -iV_x \cos \theta_{pol} \sin \theta_F + iV_y \sin \theta_{pol} \cos \theta_F \end{bmatrix} \]

\[ V_{PD2} = V_x \{ \cos \theta_{pol} \ [\cos \theta_w \cos \theta_F + i \sin \theta_w \sin \theta_F] \} + V_y \{ -\sin \theta_{pol} \ [-\cos \theta_w \sin \theta_F + i \sin \theta_w \cos \theta_F] \} \]

\[ V_{PD1}\text{'s photodetector signal (S1) is proportional to} \quad |V_{PD1}|^2 \]
\[ V_{PD2}\text{'s photodetector signal (S2) is proportional to} \quad |V_{PD2}|^2 \]

**General Expressions for photodetector signals (S1 and S2):**

\[ S_1 \propto |V_x \cos \theta_{pol} \ [\sin \theta_w \cos \theta_F - i \cos \theta_w \sin \theta_F] + V_y \sin \theta_{pol} \ [\sin \theta_w \sin \theta_F + i \cos \theta_w \cos \theta_F]|^2 \]

\[ S_2 \propto |V_x \cos \theta_{pol} \ [\cos \theta_w \cos \theta_F + i \sin \theta_w \sin \theta_F] - V_y \sin \theta_{pol} \ [-\cos \theta_w \sin \theta_F + i \sin \theta_w \cos \theta_F]|^2 \]
When the beam is unaffected by the sample (e.g., sample is removed in transmission
$V_x = V_y = 1$) $V_{PD1}$ and $V_{PD2}$ simplify to:

$V_{PD1} = \sin \theta_w \cos(\theta_{pol} - \theta_F) - i \cos \theta_w \sin(\theta_{pol} - \theta_F)$

$V_{PD2} = \cos \theta_w \cos(\theta_{pol} - \theta_F) - i \sin \theta_w \sin(\theta_{pol} - \theta_F)$

$S_1$ (no sample) $\propto 1 V_{PD1}^2 = \sin^2 \theta_w \cos^2(\theta_{pol} - \theta_F) + \cos^2 \theta_w \sin^2(\theta_{pol} - \theta_F)$

$S_2$ (no sample) $\propto 1 V_{PD2}^2 = \cos^2 \theta_w \cos^2(\theta_{pol} - \theta_F) + \sin^2 \theta_w \sin^2(\theta_{pol} - \theta_F)$

**EXAMPLES:**

Testbed setups 1 - 4 are without any samples in sample holder. For each of these four examples we will assume $V_x = V_y = 1$.

**Testbed setup #1: (No Sample)**

On the Detector Arm we orient the Fresnel rhomb (FR2) at 45° ($\theta_F = 45°$). We next position the inner rotation stage for an angular reading of zero degrees (i.e., $\theta_w = 0°$). Since the inner rotation stage couples to the Fresnel rhomb’s rotation, a setting of zero degrees indicates that the Wollaston’s angular orientation with respect to the horizontal matches that of the Fresnel rhomb. The following expression shows the Jones calculus derivations and results for the normalized signals $S_1$ and $S_2$ as a function of polarization orientation angle $\theta_{pol}$. This setup is designed to maximize the $S_1$ and $S_2$ signal variations.

Figure A.3 compares the theoretical results and the measured values.
\[ \theta_F = +45° \quad \text{V}_x = \text{V}_y = 1 \]
\[ \theta_w = 0° \]

\[ S1 \propto |V_{PD1}|^2 = \sin^2\theta_w \cos^2(\theta_{pol} - \theta_F) + \cos^2\theta_w \sin^2(\theta_{pol} - \theta_F) = \sin^2(\theta_{pol} - 45°) \]

\[ S2 \propto |V_{PD2}|^2 = \cos^2\theta_w \cos^2(\theta_{pol} - \theta_F) + \sin^2\theta_w \sin^2(\theta_{pol} - \theta_F) = \cos^2(\theta_{pol} - 45°) \]

**Figure A-3.** Testbed setup # 1. Theory and measurement. Test setup gives maximum signal (S1 and S2) variations. \( \theta_F = +45°, \text{V}_x = \text{V}_y = 1, \theta_w = 0° \).
Testbed setup #2: (No Sample)

This test setup is the same as setup #1 except that we have changed $\theta_w$ from $0^\circ$ to $45^\circ$.

This set of conditions is designed to minimize the $S_1$ and $S_2$ signal deviations.

Figure A-4 compares the theoretical results and the measured values.

\[ \theta_F = +45^\circ \quad V_x = V_y = 1 \]
\[ \theta_w = +45^\circ \]

\[ S_1 \approx 1 \cdot V_{PD1}^2 = \sin^2 \theta_w \cos^2(\theta_{pol} - \theta_F) + \cos^2 \theta_w \sin^2(\theta_{pol} - \theta_F) = 0.5 \]
\[ S_2 \approx 1 \cdot V_{PD2}^2 = \cos^2 \theta_w \cos^2(\theta_{pol} - \theta_F) + \sin^2 \theta_w \sin^2(\theta_{pol} - \theta_F) = 0.5 \]

Notice that for this test setup $S_1$ and $S_2$ are independent of the PBS cube’s orientation.

Figure A-4. Testbed setup #2. Theory and measurement. Setup gives minimum signal ($S_1$ and $S_2$) variations. $\theta_F = +45^\circ$, $V_x = V_y = 1$, $\theta_w = +45^\circ$
Theory has $S_1$ and $S_2$ being constant (at 0.5) versus polarization orientation angle. The slight deviations observed in $S_1$ and $S_2$ values reflect component imperfections and/or alignment errors of the ellipsometer.

**Testbed setup #3: (No Sample)**

On the Laser Arm we orient the PBS cube at $45^\circ$ (i.e., $\theta_{\text{pol}} = +45^\circ$). We next positioned the outer rotation stage (RS3) for an angular reading of $+45^\circ$ ($\theta_F = +45^\circ$). The following expression shows the resultant $S_1$ and $S_2$ signals as a function of the inner rotation stage’s (RS4) angle ($\theta_w$).

\[\begin{align*}
\theta_{\text{pol}} &= +45^\circ \quad V_x = V_y = 1 \\
\theta_F &= +45^\circ \\
\theta_w &= \text{Variable}
\end{align*}\]

\[\begin{align*}
S_1 &\propto |V_{\text{PD1}}|^2 = \sin^2 \theta_w \cos^2 (\theta_{\text{pol}} - \theta_F) + \cos^2 \theta_w \sin^2 (\theta_{\text{pol}} - \theta_F) = \sin^2 \theta_w \\
S_2 &\propto |V_{\text{PD2}}|^2 = \cos^2 \theta_w \cos^2 (\theta_{\text{pol}} - \theta_F) + \sin^2 \theta_w \sin^2 (\theta_{\text{pol}} - \theta_F) = \cos^2 \theta_w
\end{align*}\]

**Testbed setup #4: (No Sample)**

On the Laser Arm we orient the Polarizing Beam Splitter (PBS) cube at $0^\circ$ to horizontal (i.e., $\theta_{\text{pol}} = +0^\circ$). We next position the outer rotation stage (RS3) for an angular reading of $+45^\circ$ ($\theta_F = +45^\circ$). The following expression shows the Jones calculus results for the normalized signals $S_1$ and $S_2$ as functions of the inner rotation stage’s (RS4) angle ($\theta_w$).
\[ \theta_{\text{pol}} = +0^\circ \quad V_x = V_y = 1 \]
\[ \theta_F = +45^\circ \]
\[ \theta_w = \text{Variable} \]

\[ S_1 \propto |V_{PD1}|^2 = \sin^2 \theta_w \cos^2(\theta_{\text{pol}} - \theta_F) + \cos^2 \theta_w \sin^2(\theta_{\text{pol}} - \theta_F) \]
\[ = 0.5[\sin^2 \theta_w + \cos^2 \theta_w] = 0.5 \]

\[ S_2 \propto |V_{PD2}|^2 = \cos^2 \theta_w \cos^2(\theta_{\text{pol}} - \theta_F) + \sin^2 \theta_w \sin^2(\theta_{\text{pol}} - \theta_F) \]
\[ = 0.5[\cos^2 \theta_w + \sin^2 \theta_w] = 0.5 \]

**Testbed setup #5: Beam Extinction using Corning 7059 glass sample:**

This sample material will not cause a phase shift between the p and s reflected beam components, so we can set \( \delta_x \) and \( \delta_y \) to zero. Thus, \( V_x \) and \( V_y \) are equal to the Fresnel reflection coefficients \( r_x \) and \( r_y \) respectively. In addition, \( \theta_w \) is equal to zero since the beam will remain linearly polarized upon reflection (in beam extinction method, the value of \( \theta_w \) is a direct measurement of the ellipticity of the beam). For this example we set \( \theta_{\text{pol}} \) to 30°. The amount of rotation (value of \( \theta_F \)) required to extinguish the beam (say on detector PD1) is a function of the angle of incidence (\( \psi_{\text{inc}} \)).
Given:

\[ \theta_{\text{pol}} = 30^\circ \]
\[ \theta_F = \text{variable} \]
\[ \theta_w = 0^\circ \]
\[ \psi_{\text{inc}} = 0^\circ \text{ to } 85^\circ \text{ by } 5^\circ \text{ increments} \]

We calculate \( \theta_F \) by setting the general expression for \( S1 \) to zero and solving for \( \theta_F \). Figure A-5 compares the theoretical and measured values of \( \theta_F \) required for beam extinction, versus angle of incidence (\( \psi_{\text{inc}} \)).

**Figure A-5.** Measured and calculated values of \( \theta_F \) (PD1 beam extinction) versus angle of incidence (\( \psi_{\text{inc}} \)) for Corning 7059 glass.
APPENDIX B

ITO/SnO ELLIPSOMETRIC MEASUREMENTS AND ANALYSIS RESULTS

Donnelly Corporation provided us with samples of indium tin oxide (InSnO) deposited on Corning 7059 glass substrates. Figure B-1 shows the measured rotation and ellipticity (and analysis fit) of one such sample. The analysis of this representative sample yields an ITO refractive index ($n_{ITO}$) of 2.00, an absorption coefficient ($k$) of zero, and a thickness of 200nm. The measured conductivity of these samples was on the order of $2 \times 10^{-2}$ Mho's.

![Figure B-1](image)

**Figure B-1.** Measured rotation and ellipticity with analysis fit of a thin film ITO sample on Corning glass ($n_{glass} = 1.52$). Polarization orientation ($\theta_{pol}$) was 30° from horizontal (p-pol). Laser wavelength ($\lambda$) = 632.8nm. Analysis results: $n_{ITO} = 2.00$, $k_{ITO} = 0$, $t_{ITO} = 200$nm.
We measured seven sol-gel spin coated samples of tin oxide (SnO) on 7059 glass. Figure B-2 shows the measured rotation and ellipticity of one such sample. The analysis of this sample yields an SnO refractive index \( n_{TO} \) of 1.81, an absorption coefficient \( k \) of zero, and a thickness of 27nm. The conductivity of the SnO film was dependent on the annealing temperature and duration. Conductivity ranged from \( 10^{-5} \) Mho’s (no annealing) to \( 2 \times 10^{-3} \) Mho’s (450°C for 30 minutes).

**Figure B-2.** Measured rotation and ellipticity with analysis fit of a thin film SnO sample on Corning glass \( n_{glass} = 1.52 \). Polarization orientation \( \theta_{pol} \) was 30° from horizontal (p-pol). Laser wavelength \( \lambda = 632.8\text{nm} \). Analysis results: \( n_{TO} = 1.81 \), \( k_{TO} = 0 \), \( t_{TO} = 27\text{nm} \).
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