Synthesizing and Characterizing Mesoporous Mirrors

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ABSTRACT

Distributed Bragg Reflectors were synthesized by spin coating alternating layers of mesoporous silicon dioxide (SiO_2) and mesoporous titanium dioxide (TiO_2) on a silicon wafer. After annealing, samples were characterized and modeled using ellipsometry and absorption spectroscopy before the next layer was deposited. The samples displayed a single Bragg peak that, due to thin film interference, was dependent on the thickness and index of refraction of the layers of SiO_2 and TiO_2. After all layers were deposited, 5CB liquid crystals were infiltrated into the mesopores and a shift in the Bragg peak was observed.

INTRODUCTION

A Bragg mirror, or Distributed Bragg Reflector, (DBRs) consists of a stack of thin layers of dielectric materials with alternating high and low refractive indices[1]. In the case of this experiment, mesoporous silica (SiO_2, n=1.25-1.45) and titania (TiO_2, n=1.65-1.75) were the materials used. One period of the DBR is described as one layer of silica and one layer of titania. Generally, one needs to increase the reflectivity and the width of the Bragg peak, two parameters that are dictated by the index contrast of the two constituent materials and the number of periods in the DBR.[1,2,3]. DBRs are examples of 1-dimensional photonic crystals[2] which, while simple compared to their 2-D and 3-D counterparts, still offer quite a few applications including optical fibers and lasers.

DBRs display a Bragg peak whose central wavelength, λ_{bragg}, is dependent on the index of refraction and the thickness of the materials used: $m\lambda_{bragg}=2(h_L n_L+h_H n_H)$ where h is the thickness and n is the index of refraction and the subscripts refer to high and low index layers[1]. Index of refraction can be changed by changing the porosity (percentage of air contained within a single layer) of the materials, and thickness can be controlled by altering the conditions in which the layers of the DBR are deposited. Changes in the fabrication process also lead to better structural integrity of the stacks, particularly annealing temperature, and annealing each the samples after each layer[2,3].

Liquid Crystals are materials who, among other things, change their orientation depending on the conditions in which they are placed[4]. When infiltrated into the pores of DBRs the change in index of refraction of the whole sample will have an effect on the wavelength reflected by the DBR. By controlling the orientation of the liquid crystals through either temperature or applied voltage, the wavelength reflected by the DBR could also be controlled [4] .

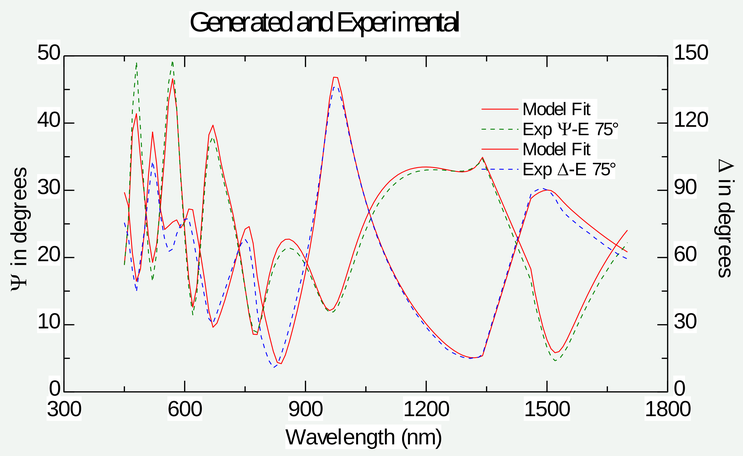

Figure 1. Ellipsometric Data obtained for a sample at 75° incident light. The red line denotes the fit obtained from the model (shown on far right of poster) of the sample.

EXPERIMENTAIL DETAILS

The DBRs were synthesized via spin coating alternating layers of silica and titania solutions onto a silicon substrate. Changing the surfactant levels in the solutions changed the porosity of the resultant layer. Between each deposition, the samples were annealed at 300 ℃. After 3 periods were deposited, 5CB liquid crystals from Merck were infiltrated into the samples by submerging the samples in a liquid crystal-acetonitrile solution for two hours.

A variable-angle spectroscopic ellipsometer from J. A. Woollam was used in a vertical sample mount configuration to gain ellipsometric data to model the samples between layer depositions (after samples were annealed, but before a new layer was deposited). For each sample Ψ and Δ were measured at 75º in the range 200-1700nm. After each period was deposited, absorption spectroscopy was used to view the Bragg peak created. Reflection data was taken after the DBRs were fully synthesized using the ellipsometer, and then again after they were infiltrated with liquid crystals. Liquid crystals were infiltrated by submerging the samples in either an acetonitrile-liquid crystal solution or an ethanol-liquid crystal solution.

Next, the liquid crystal infiltrated samples were placed in a cryostat and ellipsometric and reflection data were taken (using the ellipsometer) at slowly increasing temperatures.

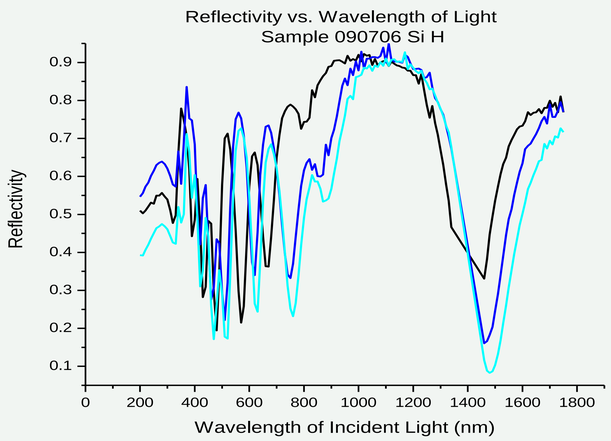

Figure 2. Reflectivity vs. Wavelength of Light for a sample.
Black : Reflectivity vs. Wavelength pre-liquid crystal infiltration.
Purple : Reflectivity vs. Wavelength 75° incident light post infiltration.
Light Blue : Reflectivity vs. Wavelength 70° incident light post infiltration.

RESULTS AND ANALYSIS

The ellipsometry data for a DBR is shown at left [Fig. 1], along with the fit from the model. A cauchy model was used to determine the index of refraction $n(\lambda)$ for each layer [Fig. 3], and the thickness of each layer [Fig. 4]. An effective medium approximation (EMA) was used to estimate the percent porosity of each layer (not shown). The goal of alternating layers of silica and titania with similar indices of refraction and thickness for every other layer was achieved in this case, as figure 4 shows. The thickness of layer 4 is slightly lower than that of the others, and could therefore explain some of the extra quasi-peaks seen in figure 2.

The thickness of the samples was effectively controlled via the spin coating process. The higher the revolutions per minute of the spin coater, the thinner the sample. However, other factors also played a role, including the porosity of the mesoporous layer (directly proportional to the level of surfactant used in the solution), and the annealing temperature.

After synthesizing the DBRs, liquid crystals were infiltrated by submerging the samples in either an acetonitrile-liquid crystal solution or an ethanol-liquid crystal solution. It was found that the acetonitrile-liquid crystal solution offered better reflectivity. The liquid crystal infiltrated samples showed a red shift in central wavelength of the Bragg peak of about 100nm [Fig. 2], but also a decrease in the width of the Bragg peak. The sample was then placed in a cryostat and reflectivity and ellipsometry data were taken at room temperature, 30 ℃ and 45 ℃. There was no apparent shift in the Bragg peak due to temperature changes.

This experiment will be continued via further research into temperature vs. Bragg peak changes, and voltage vs. Bragg peak experiments. More periods will also be added to the DBRs for higher reflectivity.

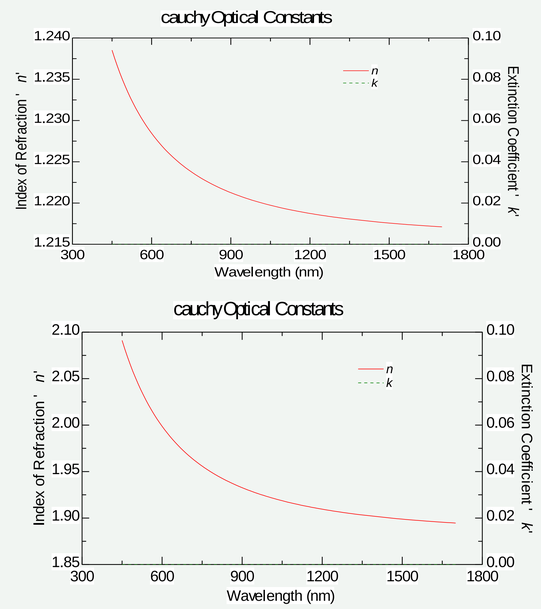

Figure 3. Index of Refraction Curves for two layers of the Cauchy Model (shown below) for a DBR. Top: Layer 6. Bottom: Layer 5.

7	cauchy	129.739 nm
6	cauchy	343.228 nm
5	cauchy	124.328 nm
4	cauchy	282.991 nm
3	cauchy	119.315 nm
2	cauchy	338.566 nm
1	sio2	5.000 nm
0	si	1 mm

Figure 4. Cauchy Model obtained from ellipsometric data. The right hand side denotes the thickness of the layer modeled. Even layers are silica, odd layers are titania (excluding layers 0 and 1, which are the substrate and the native oxide layers respectively).

CONCLUSION

It was found that Distributed Bragg Reflectors synthesized from silica and titania produced a Bragg peak between 90 and 95 percent reflectivity. It was also found that infiltrating liquid crystals into these DBRs shifts the Bragg peak to a higher wavelength. However, changes in temperature to the liquid crystal infiltrated DBRs produced no recognizable shift in the Bragg peak.

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