Fabricating Atmospheric Chemical Sensors using Mesoporous Indium Tin-Doped Oxide (ITO) Photonic Crystals

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Introduction

We designed a porous film system based on the *conducting* metal oxide, indium tin-doped oxide (ITO), which is, theoretically, capable of detecting atmospheric chemicals both optically and via electrical resistance measurements. Starting with ethanol solutions of tin (IV) chloride and indium (III) chloride, a surfactant (CTAB) was introduced to form of ITO micelles and the solutions were deposited on silicon and glass substrates via spin-coating. Experiments to create ITO thin films were partially successful as indicated by optical microscopy and spectroscopic ellipsometry. In preparation for preparing stacks of ITO films, alternating stacks of silica and titania were created and subsequently analyzed for their thicknesses and porosity values using ellipsometry.

Abstract

Porous, optically active films have made headway into the field of atmospheric chemical and biological sensors within the past decade. However, reading optical data from a film in real-time requires bulky optical sensors, as well as an external source of light. While recent advances have led to more streamlined and more efficient optical systems, the above problems still exist; thus, a new approach must be taken to create a better system.

To address this issue, we designed a one-dimensional photonic crystal based on indium tin-doped oxide (ITO) capable of simultaneously producing an optical and electrical response. In order to become familiar with the process of producing these thin films and tuning their characteristics (such as thickness and the size of the micelles, and thus the film's overall porosity), we created samples of both single and multiple stack opals of silicon dioxide (SiO_2, silica) and titanium dioxide (TiO_2, titania) and measured their properties via spectroscopic ellipsometry and visible light spectrometry.

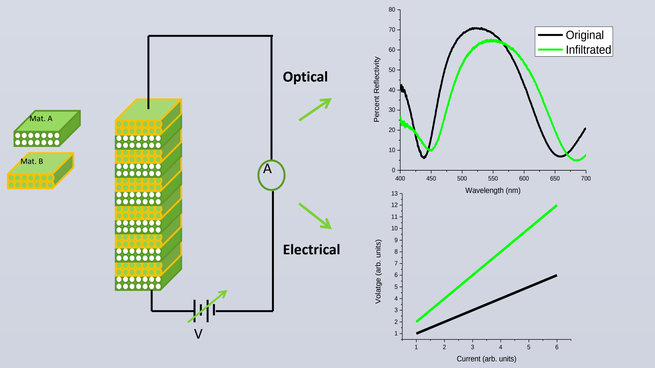


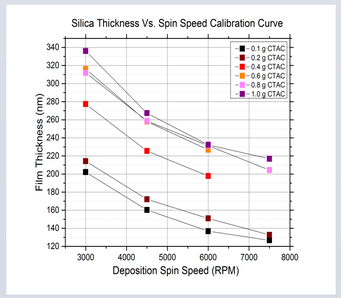
Figure 1. A theoretical view of a stacked inverse opal sensor, along with example output of a benchmark scan, overlaid with measureable respond to a generic chemical signal.

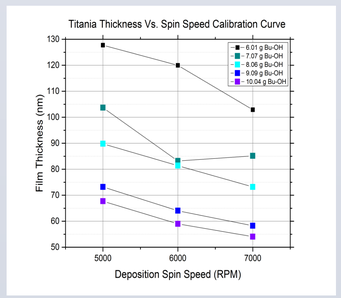
Methods

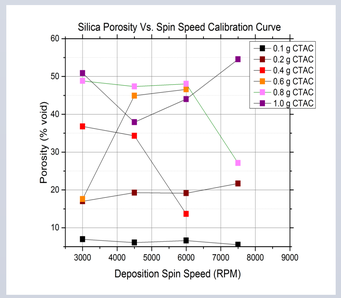
Tin (IV) chloride, indium (III) chloride and a surfactant (CTAB) were dissolved in ethanol and was stirred for several hours to allow for the formation of ITO micelles. All reactions using this technique were performed in an air-free environment. After mixing, the solutions were deposited on silicon and glass substrates via spin-coating. The stacks of silica and titania were created by creating an ethanol solution of silica precursor (Trimethyl Orthosilicate) and a surfactant (CTAC); and a separate 1-butanol solution of titania precursor (Titanium (IV) Ethoxide), a surfactant (P123 copolymer), and a weak solution of HCl to facilitate the formation of the P123 micelles. The solutions were then deposited and analyzed in the same manner as the ITO films.

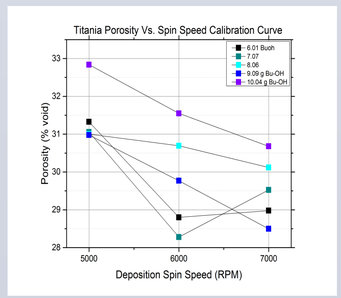
Results

Our experiment, as previously mentioned, was conducted in several parts. A first, proof-of-concept experiment was conducted in two stages. Initially, we performed tests to create several calibration curves which served as a guide on how to accurately create films with specific porosities and thicknesses. Then, the task became to design films with specific thicknesses and porosities that consisted of several periods of alternating silica and titania films with similar physical parameters. Below are graphs of these calibration curves (Scheme 1), as well as an ellipsometric scan and an absolute reflectivity scan of a representative silica/titania film stack.

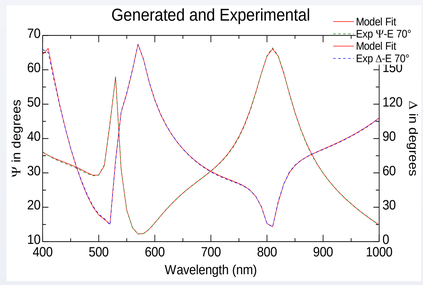








Scheme 1. The calibration curves used to design the stacked samples of silica and titania. IT is evidenced above that thickness of a film is much more precisely tunable than porosity by the high level of variance in the bottom two graphs.



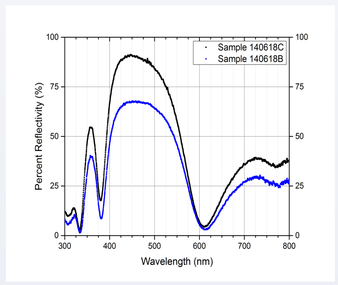


Figure 2, 3. The ellipsometric scan (left, figure 2) is representative of a generalized 3 period stack of thin silica/titania films. The reflectivity scan (right, figure 3) was performed via Xenon lamp reflection on two different, yet similarly designed, thin film samples.

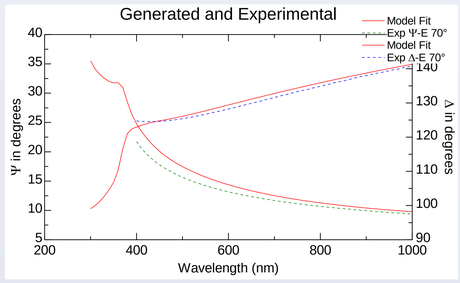


Figure 4. Ellipsometric scan of an ITO deposition on silicon, with an overlaid model of pure silicon substrate. This evidences our inability to accurately model the present film.

Conclusions

In reference to the problems of current atmospheric chemical sensors, no viable solution could be produced by our lab. When our synthesized ITO samples were measured via ellipsometry, the films could not be conclusively modeled by the program, and instead it appeared that the layer in which our ITO was deposited was composed of simply air. One theory, for which there is considerable evidence, is that our acidic solution of ITO etched away the natural oxide layer of the silicon and deposited ITO micelles much closer to the substrate than our equipment was able to precisely model. This inability to accurately comprehend the structure of our samples, or scan them, lead to our inability to produce a working optical model of a thin mesoporous ITO film.

References

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