Determining the Molecular Weight of Polystyrene in Food-Serving Plates by Thin Film Analysis

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Abstract

The common properties of polystyrene are well known. In this series of experiments we dissolved polystyrene food-serving plates in varying concentrations of toluene, used the spin-casting technique on silicon wafers to create thin films, and measured properties of the food-plate polystyrene by ellipsometry. We have confirmed that the material in the plates is indeed polystyrene by using Fourier Transform Infrared Spectroscopy (FTIR). Using statistical analysis, we have determined the molecular weight (MW) of the polystyrene to be 315242 ± 9735.

Contents

1 Introduction

Polystyrene is an inexpensive, common material used for plastic cups, plates, and Petri dishes. [1] Our goal was to find the molecular weight (MW) of the polystyrene cut from a food-serving plate, and we used Fourier Transform Infrared Spectroscopy (FTIR), spin casting, and ellipsometry to achieve this goal. The FTIR was used to determine the composition of the food-serving plate by, spin casting was used to create very thin films of polystyrene on a silicon wafer, and ellipsometry was used to measure the thickness of these thin films. By doing so, we were able to use statistical analysis to determine the molecular weight of the plate from its thickness.

2 Equipment and Description of Data

To conduct this research project, we used polystyrene food-serving plates dissolved in toluene (Sigma-Aldrich) in concentrations of 5.03 mg/mL, 9.88 mg/mL, 14.70 mg/mL, 20.00 mg/mL, and 24.97 mg/ml. We used 1-0-0 silicon wafers cut to approximately 1 cm by 1 cm dimensions with diamond cutter and tweezers, and used the spin-casting instrument at 2507 RPM for 30 seconds for measurement. The spin-casting instrument was manufactured by Headway Research (model PWM32). Then, we used an ellipsometer manufactured by Rudolph Research (model AutoEL) to determine the thickness of the thin films. Data collected included the thickness of the thin films and the concentrations of the polystyrene-toluene solutions. The FTIR instrument was manufactured by ThermoFisher Scientific (model Nicolet 6700 FTIR).

3 Research Procedures

Two Miller index [100] silicon wafer squares ([100] formed squares instead of triangles from [111]) of 1 cm by 1 cm were cut for each concentration of polystyrene in toluene, for a total of 10 wafers. The tools used were a diamond cutter and tweezers. The cutting was done on large paper towels to ease the clean-up process.

As polystyrene food-serving plates were to be used later in the experiment, it was necessary to confirm that they were indeed polystyrene. Pieces were cut out and pressed into the FTIR instrument for scans. A "blank" run was executed to remove noise and background content. A second run was executed to determine the content of the plate's sample. The data from the blank was subtracted from the second run. Data collected included a percentage match with polystyrene. The software searched libraries of FTIR data and found matches with the collected data.

After cutting the wafers, solutions of polystyrene in toluene were made. Parts were cut from the polystyrene plate and the masses were determined in milligrams. Appropriate volumes of toluene were measured to create approximately 5, 10, 15, 20, and 25 mg/mL concentration solutions of polystyrene in toluene (the exact values were recorded). The toluene was pipetted into the polystyrene and the bottles capped to prevent evaporation.

The silicon wafer squares were placed at the centre of the spin-casting instrument, and 3 drops of polystyrene-toluene solution were quickly pipetted onto the silicon wafers. The instrument was run at 2507 RPM for 30 seconds, and the wafer squares removed from the instrument. This process was repeated 10 times, once for each wafer square.

After completion of spin-casting, the thickness of the films were measured by ellipsometry. The approximate colour of each square was matched with pre-existing samples to ensure accuracy of thickness measurement. After retrieval of Delta and Psi (raw) values from the ellipsometer, the values were entered into the computer for calculation of thickness. The 0th order calculation was used for all wafers, as that was the only reasonable value that matched with the pre-existing samples. The computer had 0th, 1st, 2nd, and 3rd order calculation values available as output. 2 trials for each wafer square were completed for 4 trials in each concentration and 20 trials in total.

After thickness measurements, contact angle properties were tested to determine the hydrophobicity of the wafers, post-coating. The manufacturer of the high-resolution camera was KSV Instruments (model CAM 200). The imaging was done under low-light conditions to ensure maximum contrast. Both the coated and non-coated wafer were dripped one drop of deionized water, and the contact angle on each side measured. Two trials were done for each wafer, for a total of 4 angle measurements on each wafer (2 angles per trial). These results are in the Appendix as they do not pertain to the main goal of the experiments (to obtain the MW of polystyrene in the plates).

4 Results and Data

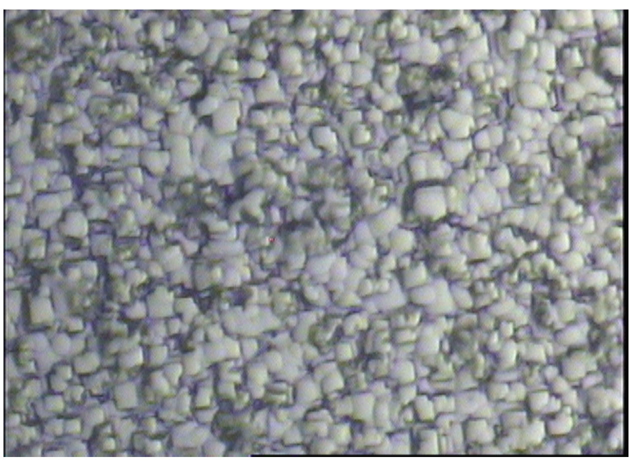

Figure 1: A 50X magnification image of [100] silicon showing the rectangular structures. This image was from group 5 as group 7 did not have both [100] and [111] images.

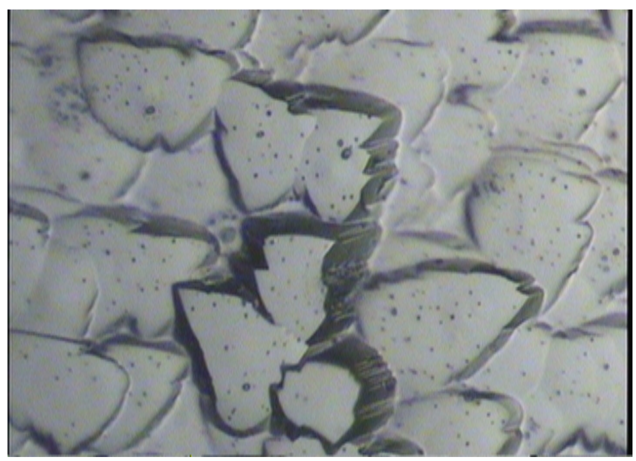

Figure 2: A 50X magnification image of [111] silicon showing the triangular structures. This image was from group 5 as group 7 did not have both [100] and [111] images

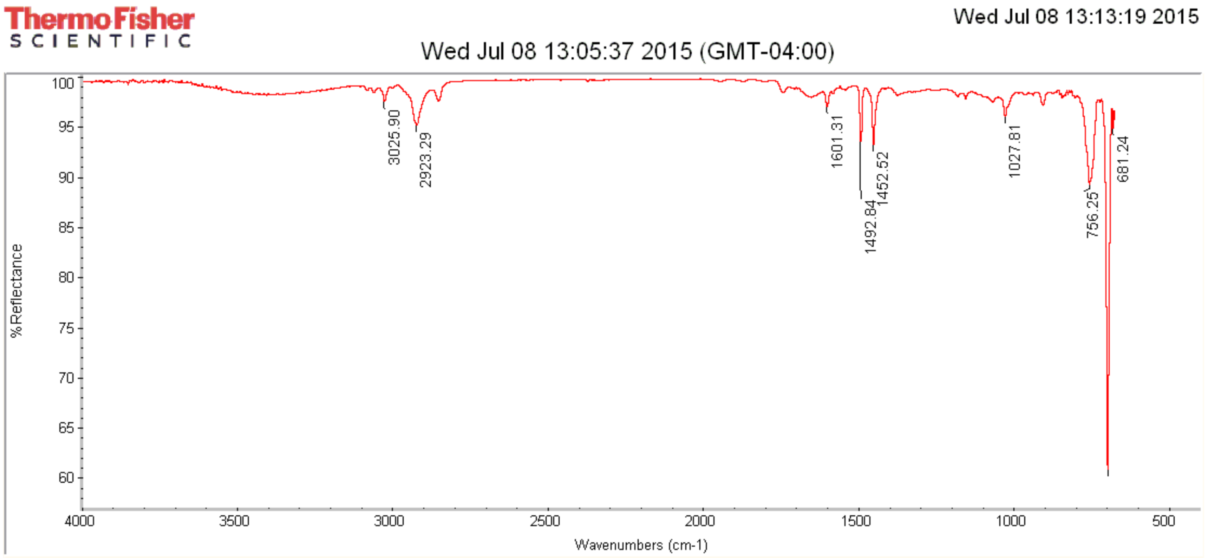

Figure 3: FTIR data from polystyrene plates showing an 80% match with polystyrene. An 80% match allows us to confirm that the polystyrene plate sample was indeed made from polystyrene.

Below is the data from the ellipsometer. Four trials were completed for each concentration of thin film.

Concentration (mg/mL)	Thickness (Å) Trial 1	Trial 2	Trial 3	Trial 4
5.03	271	273	300	276
9.88	545	570	589	577
14.70	914	913	1053	1055
20.00	1340	1344	1361	1357
24.97	1881	1829	1810	1827

Figure 4: All data collected from 4 trials of the ellipsometer.

Mean Thickness (Å)	Standard Deviation	Concentration (mg/mL)	Error
280	13.49	5.03	0.05
570	18.57	9.88	0.05
984	81.12	14.70	0.05
1351	10.08	20.00	0.05
1837	30.71	24.97	0.05

Figure 5: A table of Thickness vs. Concentration.

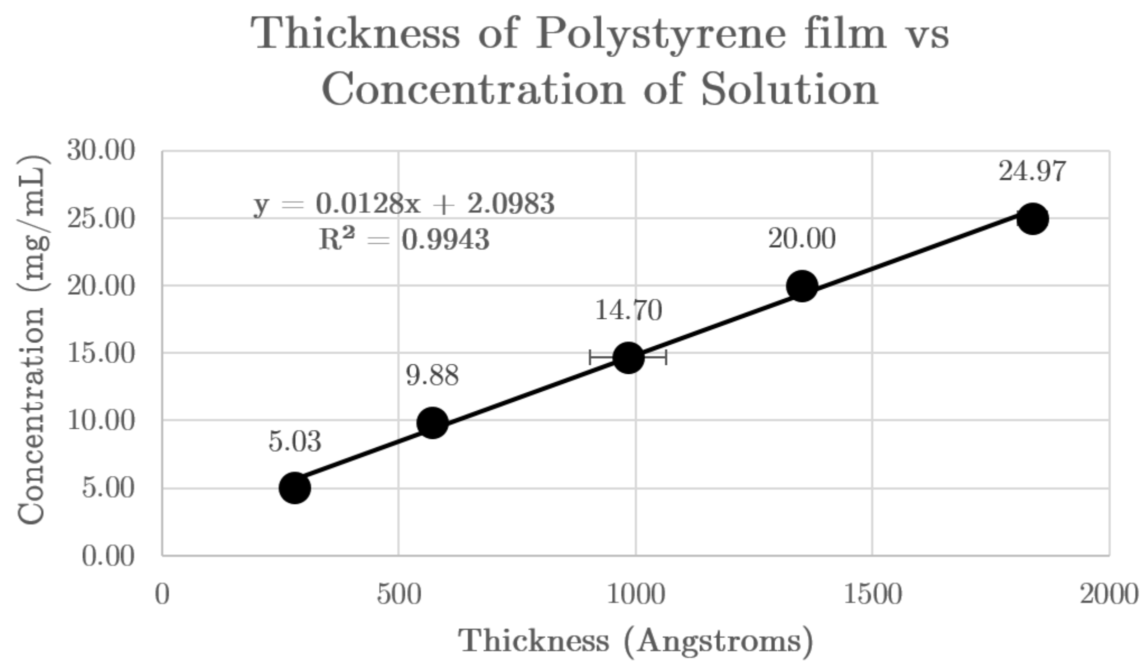

Figure 6: A plot of Thickness vs. Concentration from the table in Figure 5.

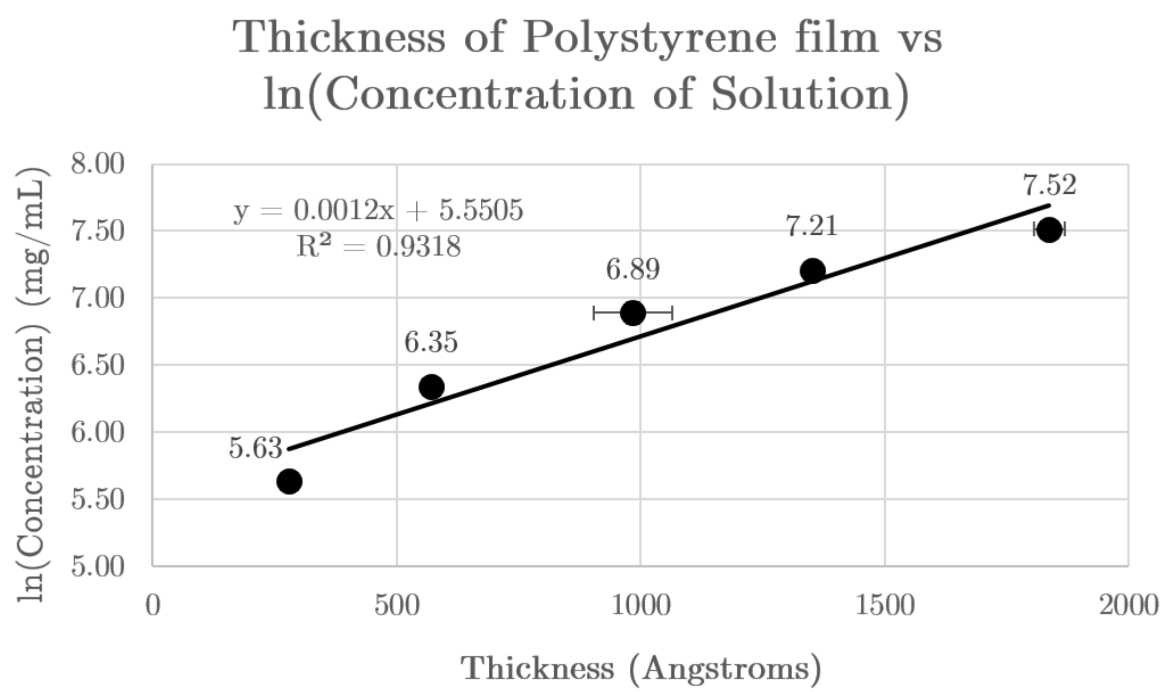

Figure 7: A plot of Thickness vs. ln(Concentration), used for testing regression. However, the R^2 value is inferior compared to the R^2 value from the linear (not ln) plot in Figure 6, so we will be using the linear plot for data extrapolation purposes.

Since the linear line of best fit from Figure 5 is

$$\text{Concentration} = 0.0128 * \text{Thickness} + 2.0983 \tag{1}$$

we can extrapolate the concentration for a film of thickness 300nm (3000Å) by plugging in value for thickness (3000). The extrapolated concentration is 40.50 mg/mL with a maximum error of 0.40. There will be more discussion on error in the Error Analysis section.

After extrapolation of concentration, we used the paper from Dr. Miriam Rafailovich, "Spin Casting Polymer Thin Films", which had an equation on page 15 relating concentration to molecular weight. The equation in the paper [2] was as follows:

$$\log_{10} \text{Concentration} = 3.378 - 0.322 \log_{10} \text{MW} \tag{2}$$

By solving for MW and using 40.50 for concentration, we obtain a MW of 315240 with an error of ±9732. Steps to obtain the error will be discussed in the Error Analysis section. This number is within reason, as the typical MW of polystyrene is 300,000. [3]

A fun fact: using the logarithm of base e (ln) will result in a MW of approximately 0.9.

Below are the images of the high-resolution camera used in determining hydrophobicity of the coated silicon wafer.

5 Error Analysis

Errors were introduced during the experimentation process. When finding concentrations of polystyrene in toluene, an error of 0.05 occurred from the measurement of volume of toluene. The glass pipette used for collecting toluene was accurate to ±.05mL. The error in the mass measurement for polystyrene was insignificant, due to the provided decimal places being far fewer than the number of decimal places offered by the scale.

For extrapolation purposes, the maximum percentage error in the concentration measurements was in the first data point: 0.05/5.03 = 0.994%. Multiplying this by the extrapolated concentration at 300nm (Φ_{300} = 40.50mg/mL) gave an error of ±0.40. We will call this number $d\Phi_{300}$.

The derivation of the error in molecular weight at 300nm using calculus is as follows:

5.1 The Calculus Method

Recall that the equation relating concentration and MW was

$$\log_{10} \text{Concentration} = 3.378 - 0.322 \log_{10} \text{MW} \tag{3}$$

We will replace Concentration with Φ and MW with M. If we solve for M, we get the following equation:

$$\text{M} = 10^{\frac{3.378-\log_{10}\Phi}{0.322}} \tag{4}$$

Recall that $d\Phi_{300}$ is the error in concentration at 300nm. Similarly, the error in MW at 300nm would be dM_{300}. To obtain dM_{300}, we must first take the derivative of equation 4 with respect to Φ.

$$\frac{d}{d\Phi}(\text{M}) = \frac{d}{d\Phi}(10^{\frac{3.378-\log_{10}\Phi}{0.322}}) \tag{5}$$

The operation on the RHS is difficult and time-consuming for a human to perform, so Wolfram Mathematica was used to perform the operation. Below is a screenshot of the Mathematica screen.

```
In[3]:= Simplify[
         D[10^((3.378 - Log10[Φ]) / .322), Φ
         ]]

Out[3]= - 9.6123×10^10 / Φ^4.10559
```

Figure 8: Screenshot of the Mathematica code used to perform the derivative in the RHS of equation 5.

The resulting full equation is

$$\frac{\mathrm{dM}}{\mathrm{d}\Phi} = -\frac{9.6123 \times 10^{10}}{\Phi^{4.10559}} \tag{6}$$

After multiplying both sides by $\mathrm{d}\Phi$, we have

$$\mathrm{dM} = -\frac{9.6123 \times 10^{10}}{\Phi^{4.10559}}\mathrm{d}\Phi \tag{7}$$

By evaluating for dM at 300nm, we have

$$\mathrm{dM}_{300} = -\frac{9.6123 \times 10^{10}}{40.4983^{4.10559}} \times 0.4026 = -9732. \tag{8}$$

Since dM is an error, we can take the absolute value of -9732 and obtain an error of ± 9732.

5.2 The Range Method

An alternative method, without calculus, is to take the minimum and maximum values of Φ_{300} (equal to 40.50 ± 0.40) and use them in equation 4. One obtains values for M_{300} of 324973 and 305510. The average distance between these values and the original M_{300} is 9735. This ”range method” confirms that the ”calculus method” is correct. Taking the larger error value from the range method, the MW of 300nm polystyrene is 315242 ± 9735. Methods to decrease error in the future would include using more accurate pipettes.

6 Conclusion

Through this series of educational laboratory experiments, we have learned the purposes of FTIR, ellipsometry, spin-casting, silicon wafers, contact angle measurements, and laboratory safety. We have learned to use calculus to find error propagation and statistical analysis to derive values. We have found that polystyrene coatings make silicon surfaces more hydrophobic, and that the molecular weight of polystyrene in food-serving plates is 315242 ± 9735.

7 Appendix

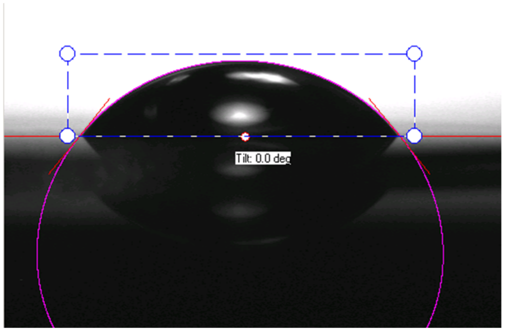

Figure 9: Image of the non-coated silicon wafer with water droplet, with contact angles of 69.66°, 69.22°, 66.15°, and 67.41°. The average contact angle was 65.61°.

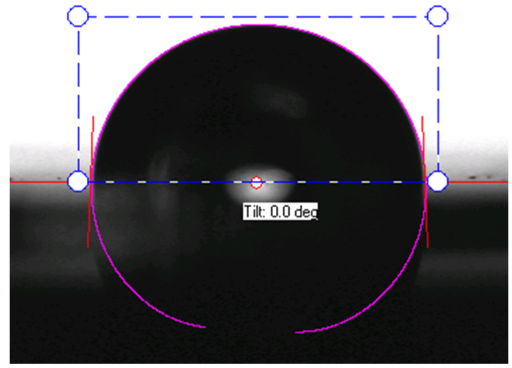

Figure 10: Image of the coated silicon wafer with water droplet, with contact angles of 88.99°, 89.24°, 89.40°, 89.52°. The average contact angle was 89.26°.

From the two images above, we can see that the contact angle was significantly larger for the polystyrene coated silicon wafer, showing that it is more hydrophobic than the uncoated silicon wafer.

8 Acknowledgements

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Yuan Xue, Stony Brook University

Dr. Miriam Rafailovich, Stony Brook University Materials Science

Dr. Adriana Pinkas-Sarafova

9 References

[1] American Chemistry Council. "History of Polymers & Plastics for Teachers." American Chemistry Council. N.p., 2015. Web. 12 Jul. 2015.

[2] Rafailovich, Miriam. "Spin Casting Polymer Thin Films." Garcia Research Program. N.p., Summer 2013. Web. 12 Jul. 2015.

[3] Vollmert, Bruno. "Polymer Chemistry." Springer-Verlag New York Inc. p. 174. 1973. Book. 12 Jul. 2015.