Aim: This lab serves as an introduction to preparation technologies for organic thin films. Thin film deposition of polymers by spin coating will be studied. Film thickness distribution on the sample should be determined and compared with the theoretical model.
1 Abstract

The goal of this practical experiment should be the preparation of thin polystyrene (PS) films via spin coating. The polystyrene powder has to be dissolved in toluene. Fill out the safety information sheet first. Toluene is harmful (Hazard: Xn) and highly flammable (Hazard: F). You must wear protective clothing and gloves!

The thickness of polymer films depends on different factors:

- viscosity and concentration of the solution
- the evaporation rate of the solvent
- rotational speed
- rotation time

Two polymer solutions with different concentrations shall be prepared and spin-coated using three different angular velocities to get films of different thicknesses. The film thickness will be measured using a Talystep profilometer.

2 Introduction

Spin coating is a fast and easy method to generate thin and homogeneous organic films from solutions. Spin coating is a procedure used to apply uniform thin films to flat substrates. In short, an excess amount of a solution is placed on the substrate, which is then rotated at high speed in order to spread the fluid by centrifugal force. A machine used for spin coating is called a spin coater. This method was first described by Emslie et al. (1958) and Meyerhofer et al. (1978) using several simplifications.

3 Basics

3.1 Spin Coating process

3.1.1 General

Spin coating is a method to produce thin organic films that are uniform over large areas (diameter ≥ 30 cm). There are four different stages of the process:

- deposit a coating fluid on to a wafer or flat substrate
- accelerate the substrate up to its final, desired rotation speed
- spin the substrate at a constant rate and fluid viscous forces will dominate the fluid thinning behavior
- continue spinning at a constant rate and solvent evaporation will dominate the coating’s thinning behavior.

After evaporation of the whole solvent, a solid film will have been generated.
To get a homogeneous film, several different factors are important and have to be considered:

- evaporation rate of the solvent
- viscosity of the fluid
- concentration of the solution
- angular velocity (rotating speed)
- spinning time.

### 3.1.2 Solvent evaporation

**Evaporation rate of the solvent**

For the spin coating process, it is necessary to have a solvent that evaporates quickly at room temperature, such as acetone or toluene.

The evaporation process influences the flow of the solution. If the evaporation of the solvent happens very quickly, the solution will flow slower as the viscosity of the solution increases. The film thickness depends on the evaporation rate of the solvent. There are 3 possible cases:

1. The solvent does not evaporate. This means the film thickness ($d$) depends only on the rotational speed ($\omega$) and spinning time ($t$).

   $$ d : \omega^{-1} \cdot t^{-\frac{1}{2}} $$

2. The solvent evaporates at a constant rate.

   $$ d : \omega^{\frac{3}{2}} $$

3. Solvent evaporation varies with the square root of the angular velocity.

   $$ d : \omega^{\frac{1}{2}} $$
In the spin coating process, the interactions between substrate and solution layer are stronger than the interactions between solution surface and air.

During the spin coating process, the solvent evaporates, which leads to an increasing concentration and therefore increasing viscosity. This changes the rheology of the solution.

The film thickness is dependent on the viscosity and concentration of the liquid. The more concentrated the solution is, the thicker the film gets. The reverse holds for the dependency of film thickness on angular velocity.

The same dependency is obtained for the spinning time. At a constant spinning speed, the longer the spinning time, the thinner the film.

![Figure 2. Film thickness vs angular velocity at varying concentrations of the solution](image)

### 4 Testing

#### 4.1 Equipment, samples and handling instructions

For this experiment the model *Spincoater Model P6700 Series* of the firm *Specialty Coating Systems, Inc.* will be used. It has a maximum speed of 2000 rpm.

**Always use the spin coater with the cover closed!**

First, turn on the “air-pressure” to the left on the left hood (outside). Turn the lever up. Then switch on the vacuum pump on the left hood. The button is next to the spin coater. The spin coater itself does not have to be switched on; this is automatic. Take off the top cover and place the substrate (Si wafer) on the middle of the chuck. Close the cover.

Now take up the prepared solution into a syringe. Press the start button of the spin coater after you have placed the syringe in the middle of the top cover where there is a small hole. Drop one milliliter of the solution when the substrate begins to rotate. After you have dropped one milliliter, take the syringe away from the spin coater quickly and calmly.
When the substrate and polymer solution have spun for one minute, press the stop button. Wait until “check vacuum” is displayed before you take the freshly coated Si wafer out of the spin coater.

Use a spinning speed of 1000 rpm. The solution should be dropped on the substrate when it has just begun to rotate.

To change the rotational speed:
- press the button "CHG PRE"
- press "+" ONCE to get to the setting “rpm”
- press „ENT“ to change the rpm to higher or lower values
- press the arrows "←" or "→" until you reach the right position
- press button "+" or "-" to set the right number (max. 2000 rpm)
- press "ENT"
- press "MSG".

At the next „Start“, the new settings will be shown on the display.

**Make sure you fill out the safety information sheets for Acetone and Toluene.**

Perform the following:
1. Produce 3 different concentrated homogeneous solutions of PS in toluene (one solution that can be diluted down) to get thin films of between 50nm and 1500nm thickness. The Si wafers have to be cleaned with a cetone in the ultrasonic bath before use.
2. Use the solution with the highest concentration of PS and deposit films at 3 different spinning speeds: 500, 1000 and 1500 rpm.
3. Deposit 3 PS films by using the different solutions at the same rotational speed of 1000 rpm.

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**Figure 3. Dependence of the polymer film thickness on the solution concentration at constant rotational speed**
Measure the thickness by using a Dektak 8000 profilometer. To do this, make a scratch through the film with metal tweezers or a scalpel. Do not apply too much pressure, otherwise you will scratch the substrate.

**Profilometry** is a method used to measure height profiles. A diamond tip is moved towards the sample until it touches the sample surface. Then, a defined amount of pressure is applied and the diamond tip is drawn horizontally over the sample to record the height profile. The sample surface should not be too smooth to avoid trespassing of the diamond tip. This could lead to changes in the surface topography.

To define the film thickness, an edge is needed where the surface of the substrate is not covered with film.

**Instructions for use:**
- Place the sample in the middle of the sample holder inside the profilometer.
- Close the cover and turn on the monitor.
- Use the trackball to move the pointer to the "red arrow pointing up". Activate this by using the left mouse button.
- Wait until the noise stops, then go to the button "moving tower down, leaving the stylus up". Click with the left mouse button.
- To increase the lighting, click as long as necessary on the left "light bulb button" until the correct brightness is reached.
- To start a measurement, click on the monitor anywhere in the illuminated field.
- Scroll to the right of the scratch. Mark this position by clicking the left mouse button.
- Press "Edit" and "enter scan length".
- Click somewhere left of the scratch, but not too far. Press "ok".
- Now go to the button "switch to scan routine window". Mark "scan parameters".
  - Parameters can be changed:
    - Scan duration: ~10s
    - Profile: hills and valleys
    - Stylus force: minimum inertia force
    - Tower speed: medium.
- Lastly, click the button with the black square surrounded by three thin arrows to get back to the measurement.
- Start with "scan".
- After the scan has finished, click either onto the red or greed line and take in further to the right. Then push the button where the profile is sloped as well as straight.
- To get back, press the button with the three arrows and the black square.
- To remove the sample decrease the illumination and then click onto the red arrow showing down.
- Turn off the monitor and sign the list.

**5 Evaluation**

1. Determine the film thickness distribution of each sample using three different positions and calculate the average value.
2. Determine a theoretical model which can be used to describe the behavior of the 1 solution used at 3 different spinning speeds (500, 1000, 1500 rpm).
3. Dilute the prepared solution so that you have three different concentrations. Now spin coat these 3 solutions at the same spinning speed. Determine the thickness dependence of the film on the concentration.

6 Bibliography

http://www.ece.gatech.edu/research/labs/vc/packaging/theory/spin_theory.html