**Operating Instructions**

**Measurement of Liquid Viscosity**

The viscosity is measured using the Brookfield Viscometer (see Figure 1a).

![Image of Brookfield Viscometer](image1a)

![Image of Viscometer Spindles](image1b)

Figure 1. (a) Top view of the Brookfield Viscometer. The viscosity and level indicators are circled by red and blue, respectively; (b) Viscometer spindles. The red arrow points to the bottleneck of the spindle to be used in the experiments. Make sure that this bottleneck is immersed into liquid before performing the viscosity measurements.

1. If necessary, adjust the base of the viscometer. Make sure to maintain its level which is indicated by bubbles in the area circled by blue in Figure 1a. The bubbles should remain in the center of the indicator.

2. Make sure that the viscosity indicator (the red mark circled by red in Figure 1a) points to zero.

3. Choose the biggest spindle (see Figure 1b) and keep it clean and dry before the experiment.

4. Pour about 300 mL of liquid into the beaker.

5. Slowly place the spindle at an angle into the sample. Avoid generating bubbles.

6. Attach the spindle to the mandrel of the viscometer (circled by red in Figure 2). For this, rotate the spindle while holding the mandrel in place.

7. If necessary, add more liquid to the beaker to make sure that it covers the bottleneck of the spindle (see Figure 1b).

8. Make sure that the spindle does not touch the beaker walls and bottom during the measurement.

9. Choose the speed of rotation, turn on the power switch, and wait until the system reaches a steady state.

10. Simultaneously pull down the read switch and flip the power switch to *Pause*. The viscosity is indicated by the red marker (see Figure 1a):

\[
\mu = \frac{60N_0}{\omega}
\]
where $N_0$ is the reading of the red maker, $\omega$ is the rotation velocity (the same units as on the viscometer controls), and $\mu$ is the fluid viscosity in cP.

**Important. The numerical factor in the formula is different for different spindles. The factor of 60 shown in the formula above is applicable only to the biggest spindle.**

11. Record the value of viscosity and flip the switches back.

12. Repeat the measurement with at least three different rotation speeds. For each speed, repeat the measurement at least three times to obtain the average and the error estimate of the measured viscosity.

13. Clean the spindle and beaker with ethanol after the measurement.

### Preparation for Spin Coating Experiments

Slowly open the main cylinder valve V1 of **nitrogen tank** (see Figure 3) to allow $N_2$ to enter the system. Use the regulator to keep the outlet $N_2$ pressure at 80 psi. The outlet gas pressure is indicated by the pressure gauge PG2 (the gauge PG1 shows the pressure inside the tank).

![Figure 3. Gas cylinders.](image)

### Spin Coating Procedure

**IMPORTANT: Do not touch silicon wafers with your hands, use tweezers!**

1. Prepare a silicon wafer.
   a. Clean the mirror side of silicon wafer using ethanol. Absorb the residual ethanol with Kimwipe. Make sure that there is no dust on the wafer surface.
   b. Carefully place the clean wafer in the center of the spin coater. Make sure that the polished side of the wafer is facing up.
   c. Dispense ~1 mL of liquid on the wafer.
Figure 4. Spin-Coater: (a) Overview; (b) Screw-in reservoir.

2. Program and operate the spin coater:
   a. Press the EDIT key and type in the rotational speed and time.
   b. Press the RUN key.
   c. The screen should say “NEED CDA”. Open valve V2 on the air tube (see Figure 3).
   d. The screen should say “NEED VACCUM”. Turn on the pump and press the VACCUM key.
   e. Wait until the screen says READY and press the START key.

   IMPORTANT: Drain the screw-in reservoir (see Figure 4b) after the experiment under the guidance of TA.

   **Recommended values of control parameters**
   
   - Spinning velocity: 3000 to 8000 rpm
   - Spin-coating time: 30 to 240 seconds

   It is recommended to start with high spinning velocity and gradually decrease it in consequent experiments. This is because higher velocity yields a more uniform coating layer which makes it easier to learn the thickness measurement procedure (see below).
Film Thickness Measurements Using *Filmetrics F20*

**NOTE:** The cables connected to the Filmetric device are very fragile. Avoid moving and/or disconnecting them!

**Start-Up**

1. Push the button to turn on the light source.
2. The arrow on the light source should point to *Std* (see Figure 5).

![Filmetrics F20](http://www.filmetrics.com/thicknessmeasurement/f20)

Figure 5. *Filmetrics F20*\(^1\). Make sure that the circled arrow on the light source points to *Std.*

3. After the first wafer is coated, acquire baseline the *FILMeasure* software:
   a. Acquire sample (see Figure 6a):
      - Place a coated wafer below the light source
      - Start the *FILMeasure* software
      - Click the *Baseline* button
      - Click the *Acquire Sample* in a pop-up menu
   b. Acquire reference (see Figure 6b):
      - Place a clean silicon wafer under the light source
      - *Reference Standard* should be Si
      - Click *Acquire Reference*
   c. Acquire background (see Figure 6c):
      - Remove the silicon wafer.
      - Click *Acquire Background*
      - Click *Finish*

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\(^1\) [http://www.filmetrics.com/thicknessmeasurement/f20](http://www.filmetrics.com/thicknessmeasurement/f20)
Figure 6. Acquiring baseling in *FILMeasure* software: (a) Acquiring sample, (b) Acquiring reference, and (c) Acquiring background.
Thickness Measurement

Note: The liquid layer on a silicon wafer may shrink due to surface tension. Therefore, make sure to measure its thickness as soon as possible after the coating.

1. Place a sample under the light source
2. Click *Edit Recipe*. This will bring up a pop-up menu as shown in Figure 7.
3. For Medium 1, choose “Generic, n=1.4” and substrate is Si.
4. Make sure that the *Vary* checkbox on the left side of the column is unchecked.
5. Enter an initial guess for the film thickness (in nm) under *Nominal*.

![Figure 7. Selection of medium and substrate and entering an initial guess for the film thickness.](image)

6. Click on the *Analysis Options* tab and select a method of fitting the data (see Figure 8). There are two methods available:
   a. FFT (Fast Fourier Transform) and
   b. Spectrum matching method.

You can use either of these methods.
Figure 8. Selection of analysis method.

**Spectrum Matching Method**

A typical result of the spectrum matching method is shown in Figure 9. The blue curve shows experimental data and the red curve shows fit results. The panel on the right-hand side of the window (highlighted by the red square in Figure 9) shows the obtained film thickness and goodness of fit. Make sure that goodness of fit is higher than 0.95 (1.0 corresponds to a perfect fit). If goodness of fit is too small, you will see a substantial difference between the red and blue lines in the plot. In order to improve the fit, it is necessary to adjust the initial guess for the film thickness (Nominal box in Figure 7). If the frequency of the calculated spectrum (red curve) is higher than that of the experimental spectrum (blue curve), you need to decrease the initial guess for the thickness.

**FFT Method**

A typical result of the FFT method is shown in Figure 10. The bottom plot shows the Fourier transform of the experimental data (blue line) and the fit results (red line). Similarly to the spectrum matching method, you should make sure that these lines are in good agreement and the goodness of fit is larger than 0.95.
Notes

- For each sample, you need to perform at least three measurements of thickness at different spots. Use these data to compute the average and the standard deviation of the film thickness.

- If you want to change the name that appears next to the line on the graph, just type it in *Sample ID* box on the left of the *FILMeasure* window.