Slot Coating Experiment: steady-state and frequency response analysis of the slot coating flow

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Introduction

Slot coating process is one of the method to produce functional thin film such as battery electrodes for lithium ion battery and optical film for LCD display. It is classified as a *pre-metered* method, where the final wet thickness can be determined by the flow rate and the production speed and is independent of other operating conditions. Therefore, it is desirable for high-precision film productions.

When the coating liquid is fed to the feed slot, the liquid bridge, so called coating bead, is formed between the die lip and the moving web and bounded by two gas/liquid interfaces, or upstream and downstream menisci. At a proper operating condition, different forces, e.g. capillary, viscous, gravity, and inertial, are balanced to maintain the coating bead. Especially, for the high-precision thin film production, vacuum is applied to the upstream to stabilize the coating bead.

The location of upstream meniscus is critical to achieve suitable flow status. Most of the steady-state analyses are designed to construct the *coating window*, or the map with ranges of desirable operating conditions, which can be demarcated by tracking the location that causes defects. Meanwhile, the coating flow is always surrounded by small-scale disturbances generated by rotating units in the coating process, such as flow pump, vacuum pump, and roll. These disturbance may cause instability of the flow, hence destroy film uniformity. Because requirements for uniformity become more severe in industry, the sensitivity analysis under the disturbance is important. Therefore, frequency response analysis should be performed together with steady-state analysis.

Here, we visualize the coating bead flow using a lab-scale slot coating device with the transparent quarts roll. The CCD camera is located inside the roll and directly visualizes menisci facing the die lip faces. From the image, the locations of upstream static contact line (SCL) and dynamic contact line (DCL) are detected for the analysis of the coating flow. From the analysis one can generate the coating window experimentally or check transient behaviors of the coating bead from various external disturbances, such as pump pulsations or roll runout. Particularly, the transient responses are usually considered computationally, and rarely treated in experiments (e.g. Joos(1999)). With an aid of image analysis on automatic detection of contact line locations, the temporal behavior of upstream meniscus can be analyzed.

Experiment set-up & Flow visualization

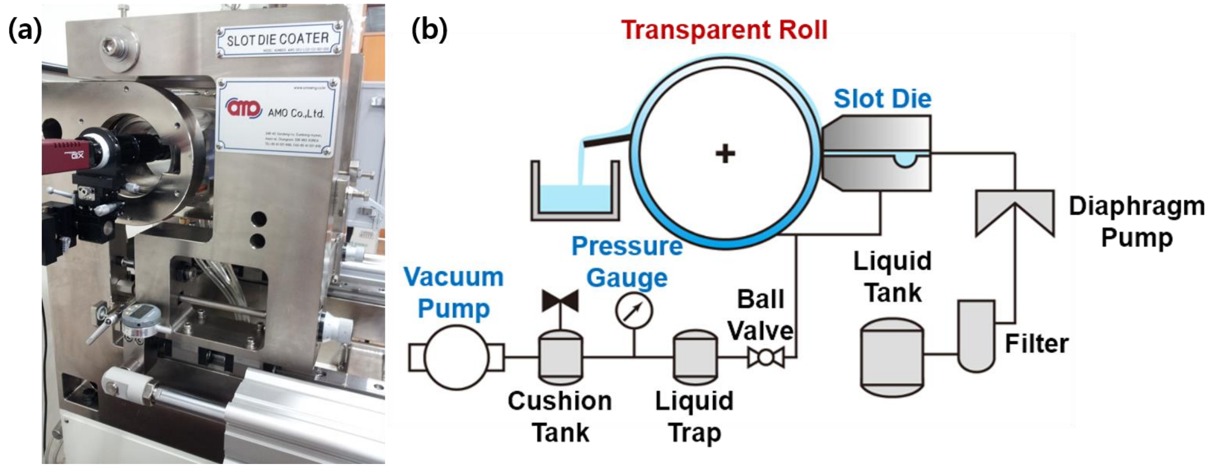

Figure 1. (a) An image of experimental set-up with CCD camera, and (b) a schematic diagram of the slot coating device.

Figure 1 shows the slot coater device with transparent quartz roll. We put the CCD camera (Prosilica GX 1050, AVT, USA) with a cam follower bearing at the left-side of the roll to visualize the coating bead flow directly. Here, we use glycerin/water solution. The coating liquid is supplied to the slot die by an industrial diaphragm pump (TBLIME-008-6T6T-CWS, TACMINA Co., OSAKA, Japan) after the bubble or dust in the liquid is filtered. Then, the liquid is directly coated on the roll surface, where we can observe if the coating result is fine, and is removed by a Teflon scraper. The vacuum chamber is installed underneath the slot die and the space between the roll and the die is sealed to apply the vacuum to the upstream of the flow.

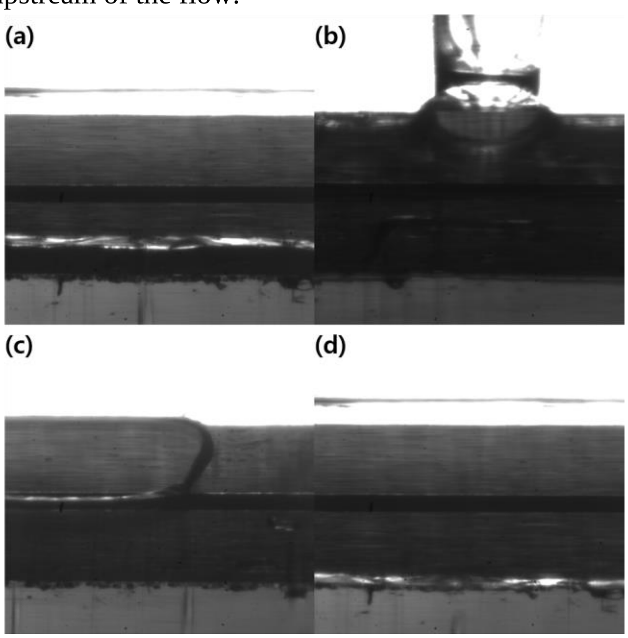

Figure 2. Examples of the visualized coating bead flow at: (a) stable condition, (b) low-flow limit, (c) bead break-up limit, and (d) weeping limit.

Examples of the coating bead images are shown in Fig. 2. During the steady-state analysis, the upper and lower vacuum limits are determined based on the location of the upstream SCL location. The pressure value is found from the pressure gauge before the coating defects occur (e.g. Fig. 2 (c)) with decreasing flow rate, or increasing gap to thickness ratio, at fixed roll speed. The process is repeated to find the coating window at different roll speed. On the other hand, for the transient or frequency response analysis, a sequence of the images is recorded at a stable flow condition, i.e., the upstream meniscus is located at the middle of upstream die lip (e.g. Fig. 2(a)). The ranges of operating conditions are 1.5-5.0mpm for the roll speed, 0-10kPa for the gauge vacuum pressure, and 200-800rpm for the flow pump. The slot die follows the uniform configuration as describe in Lee and Nam (2015). The width of feed slot is 200μm, the length of upstream and downstream die lip is 800 μm, and the gap height is fixed to 180 μm.

Image analysis procedure

In this study, a custom-designed image analysis algorithm is used to estimate the upstream SCL and DCL locations. The brief description on the algorithm is described here. First, pre-processing techniques are used for enhancing images, such as adjusting contrast and removing specs and noises. Such image enhancement allows a simple edge detector, such as Canny method (Canny, 1986), to detect contact lines and feed slot corners as shown in Figure 3. However, the detector may also capture unnecessary linear features, which need to be removed by several post-processing methods. To convert the pixel coordinate for contact lines into the physical one, we exploit the fact that the distance between feed slot corners, so called feed slot height, was fixed to 200 micron. We are currently working on writing a manuscript on the detailed algorithm.

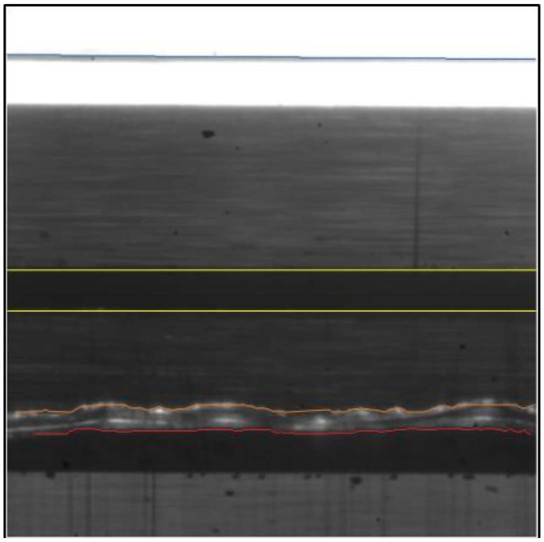

Figure 3. The result of image analysis. The red, orange, yellow and blue lines are SCL, DCL, feed slot and downstream meniscus location, respectively.

RESULTS

Steady-state analysis: Coating window

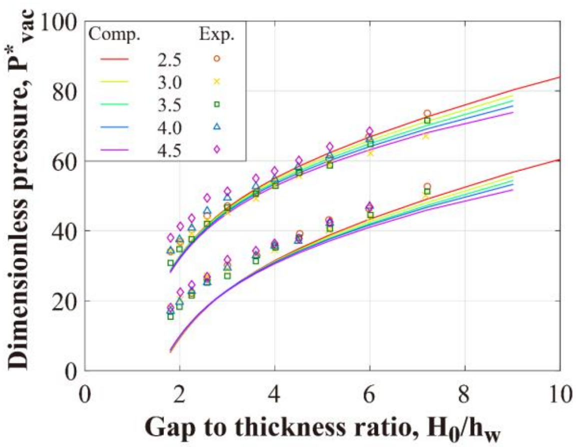

Figure 4. Computational (line) and experimental (marker) coating window at different roll speed (unit: mpm).

The computational model for slot coating flow is described in Lee and Nam (2015). We use the automatic generation method to find the coating window proposed by Nam et al. (2009) and Nam and Carvalho (2010). The coating window by the computation and the experiment is shown in Fig. 4. Here, we use the dimensionless numbers: Gap to thickness ratio ($\mathrm{R_{gt}} \equiv \frac{H_0}{h_w}$, where H_0 and h_w are the gap height and the wet film thickness, respectively) and dimensionless pressure ($\mathrm{P}_{vac}^{*} \equiv \frac{\mathrm{P_{vac}}}{\mu V_w / H_0}$, where $\mathrm{P_{vac}}$ is the vacuum pressure, μ is the liquid viscosity, and V_w is the roll speed). Note that the vacuum pressure should be measured near the vacuum chamber, and is different from the gauge pressure because of the additional unit between the vacuum chamber and the pressure gauge (Fig. 1(b)). The experimental results mostly agree with the computational predictions, except for the low-flow limit. Although the location of downstream contact line and the shape of the downstream meniscus are critical in determining the low-flow limit, it is pinned to the downstream die corner for the computational model.

Transient analysis: Frequency response analysis

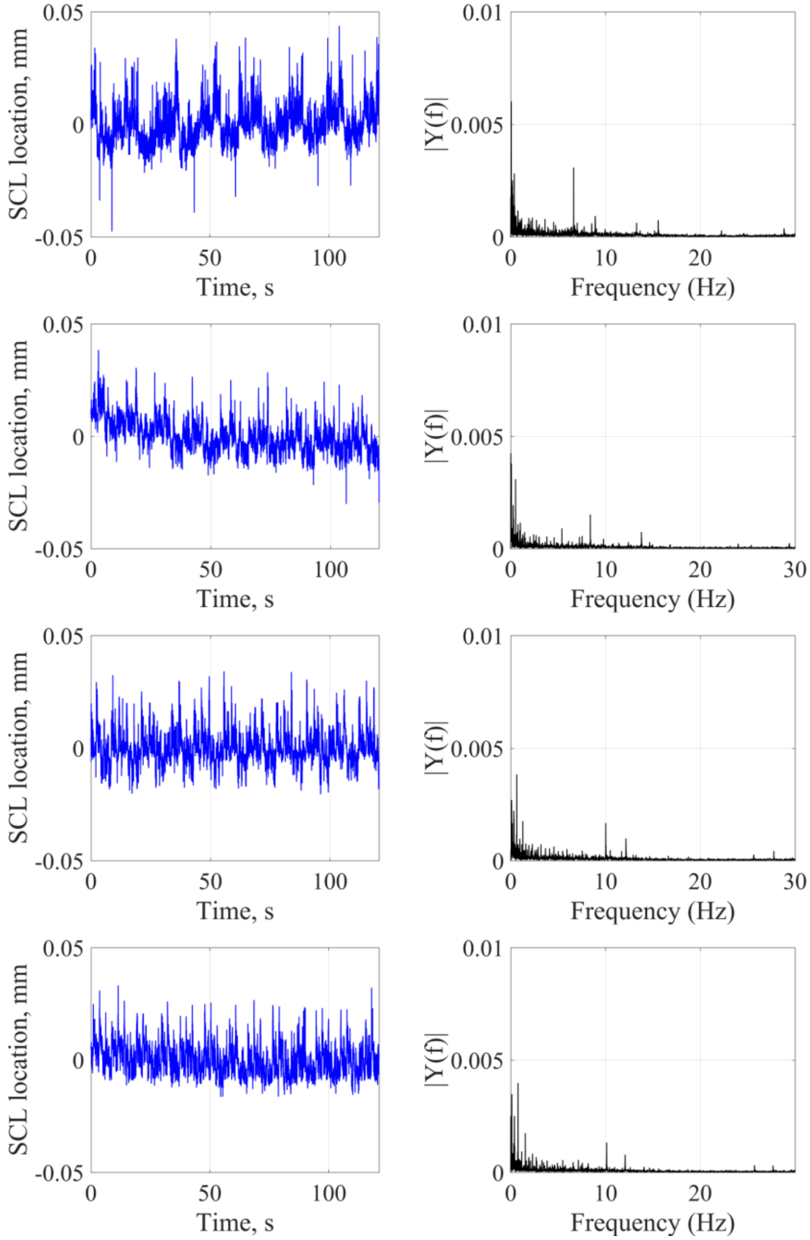

Figure 5. Relative SCL location to the initial location with respect to time (left) and FFT results (right). The roll speed is 1.5, 2.0, 2.5, and 3.0mpm, respectively in downward direction.

For a given steady operating condition, the upstream SCL and DCL coordinates are estimate with respect to time. Here, we focus on the SCL, because the DCL is sensitive to the roll speed. The movement of SCL is plotted over time as shown in Fig. 5 (left). The results can be transformed into a frequency domain by Fast Fourier Transform (FFT), as shown in Fig. 5 (right). Since a modified diaphragm-type pump, which does not show any significant pulsations, roll runout could be a major source for disturbances.

The roll, whose diameter is 15 cm, rotates about 1.7-3.5 times per second. The frequency peak at a low frequency range (< 5Hz) probably comes from the uneven roll surface. This fact is also supported by the positive peak shift (toward right) as the roll speed increases. At a higher frequency range (from 10Hz to 30Hz), several small peaks are observed, which are independent of roll speeds. We are currently working on analyzing the causes for such peaks and identifying various vibrations generated in the current slot coating set-up.

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