

Pushing the limits of slot Coating with the application of low viscosity Gases

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Introduction

Slot coating is a pre-metered coating operation, which in principle should be able to deliver a film as thin as required simply by increasing speed except for the fact that above a certain critical speed air entrainment occurs. The classic experimental work of Lee, Liu and Liu [1992] showed that in conventional slot-over roll set-up, film thickness down to the order of 100 to 160 μm can be formed for speeds ranging from 3 to 35 cm/s (1.8 to 21 m/min) using silicone oil of viscosity 50mPa.s as test fluid, a slot gap of 0.25mm and a slot-to-web gap of 200 μm . Lower wet thicknesses down to 30 μm are possible using fluids of viscosity of the order of 10mPa.s for a coating speed of the order of 6cm/s (3.6m/min) using this method. Very low film thickness, 10 microns or less are however unattainable judging from this data. This has led to a number of studies taking up the challenges of reducing the minimum film thickness in slot coating. One approach devised was the application of a bead vacuum. This was shown able to reduce the minimum film thickness by more than 30% when it is sufficiently high [1] as a result of the coating bead in the gap being pulled back by the applied vacuum, allowing the downstream lip of the slot to serve as a doctor blade thus restricting the net flow out of the slot die. The application of this technology however did not drastically reduce film thickness.

A major advance in achieving thinner films was made some years ago by dispensing with the backing roll leading to what is known as tensioned-web-slot coating [2]. This set-up allows the gap between the slot and the substrate to be reduced without danger of clash between the slot and a steel roller. This development has transformed slot coating allowing it to produce films as thin as 0.5 microns but only at very low speeds, typically less than 10 m/min. Although, this is good progress in adapting slot coating to modern needs, increasing the speed is the next step in the development. This is precisely the subject of our research.

The strategy used here centers around the key parameter, air entrainment speed, and how best to postpone it drastically to much higher speeds than it is possible by manipulating the coating flow geometry, the coating fluid properties or the substrate properties. The question is thus: how about manipulating the air or gas properties? Already prior research by Benkreira and Ikin [2010] showed that in dip coating process, replacing air with a suitable gas and reducing the pressure significantly postponed the onset of gas entrainment to higher speeds. The proposition of Benkreira and Ikin [2010] is that the viscosity of the gas played an important role in determining the onset of gas entrainment as considering that the gas becomes entrained as a thin film within a "vee". The coupling force between the gas and liquid consequently increases with gas viscosity leading to more gas being dragged down by the web. If this is true, the same gain should be observed in slot coating and indeed in all other coating flows. This will then allow slot coating to meet the operational challenges imposed by modern applications in the electronics and other high technology industries (solar cells for example) where thickness less than 10 microns are required at speeds higher than 1m/s.

Experimental Set-up

The proposed research was carried out for both slot-over -roll and web-tensioned slot coating. The experimental rig for tensioned web slot coating is shown in Figure 1 where a 50 mm wide slot coater is mounted in a vacuum chamber, within which a web winding rig is set up. The slot over roll mode incorporated a 100 mm diameter precision steel backing roller machined with microgrooves in order to minimize the tendency for air becoming entrained between the web and roller. In the web tensioned slot coating mode, the web was passed downwards directly over the slot exit.

The slot coater was constructed of Perspex and comprised a slot of width 425 μm and length 4.2 mm, the upstream and downstream land-lengths being 0.2 mm and 1.5 mm respectively. The slot

coater was mounted on a cradle as shown in Figure 1. The cradle pivoted about bearings mounted on an upper translator and its lower position was determined by a pin mounted on the lower translator. The upper and lower translators thus served to control slot inclination angle and the slot-to-web gap respectively. The slot-to-web gap was set to 0.2 mm.

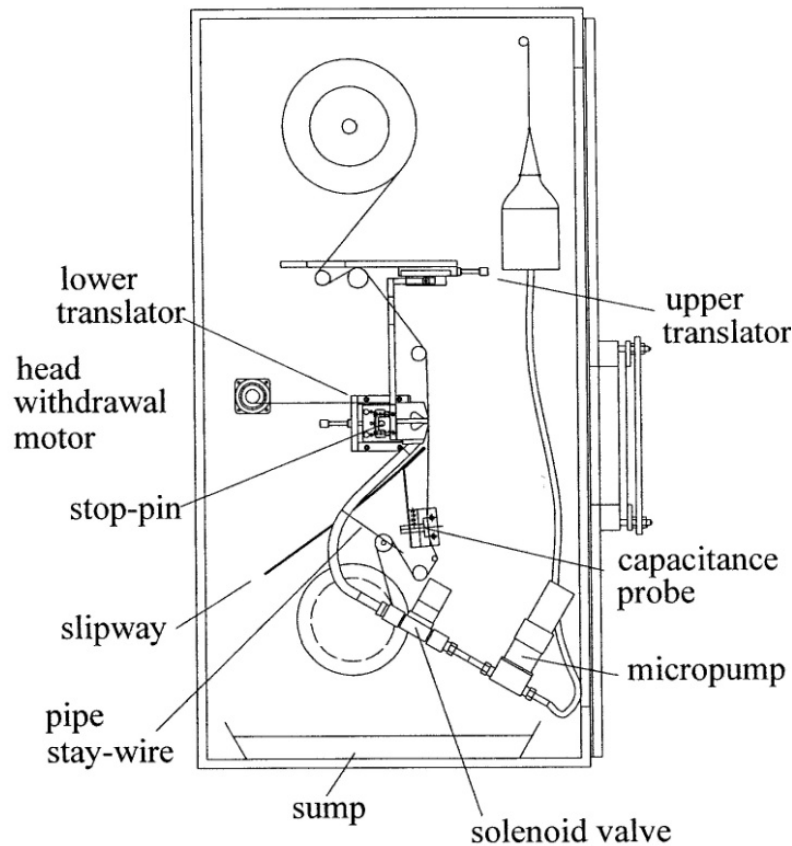


Figure 1: Rig for Tensioned web slot coating

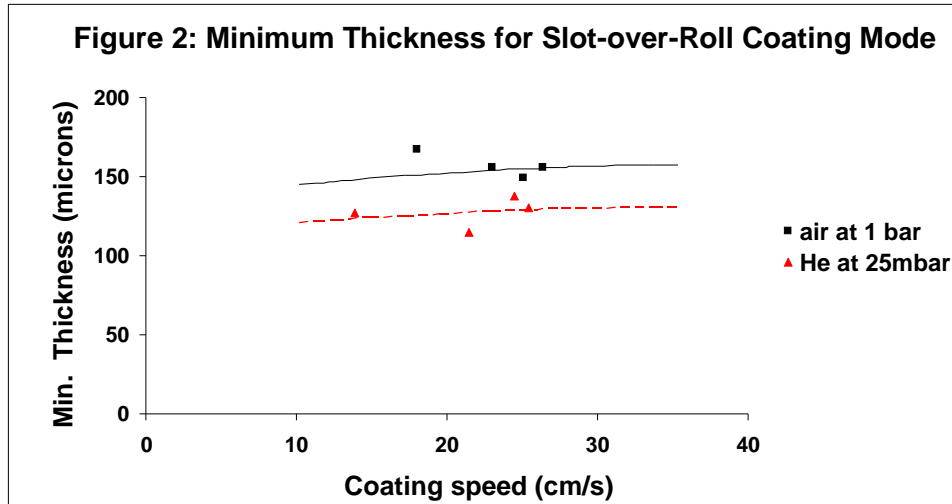
Silicone oil of viscosity 50mPa.s measured at 23°C was used as coating fluid in view of its low partial pressure thus enabling the gas pressure to be significantly reduced without risk of evaporation. In both slot coating modes, the oil was metered to the coating head using a micro-pump supplied from reservoir suspended at variable heights in order to achieve a range of flow rates.

As for film thickness measurement, it was carried out using a capacitive sensor supplied by Physik Instrumente GmbH. The sensor was mounted opposite an ultra-flat earthed plate against which the back of the coated web passed. The sensitivity to oil film thickness was first calibrated by mounting the assembly on a cantilever suspended over a pool of oil in a dish.

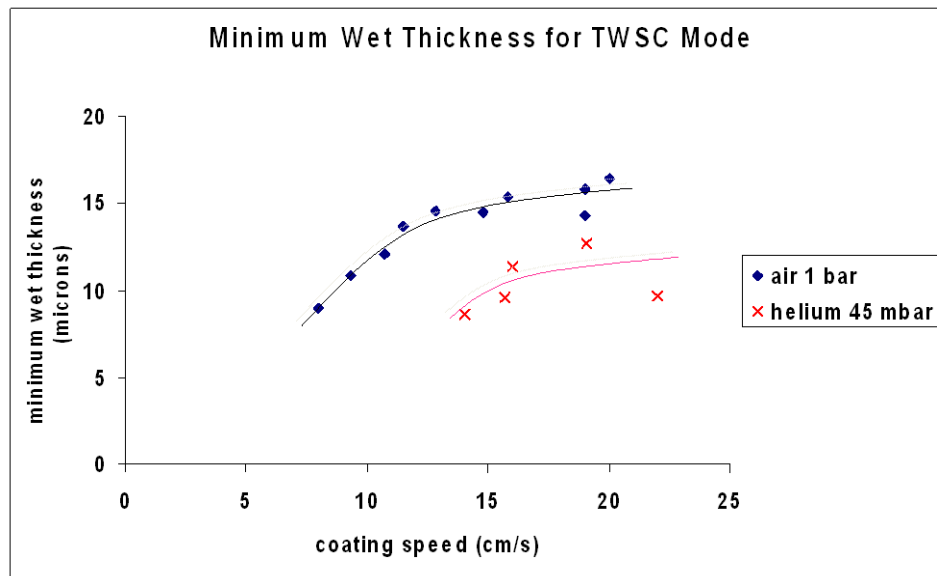
When running a coating experiment, the sensor output was recorded on one of two channels of a Picoscope coupled to a computer while ramping the web speed up at a preset acceleration for typically 20 seconds. The second channel was used to record the web speed profile obtained using a laser tacho set up opposite holes in a disk mounted at the end of a web transport roller. CCD cameras were used for recording images of the instantaneous web speed as displayed by an oscilloscope monitoring the laser tacho output, a view of the coater as observed through a chamber side port and the coating uniformity. A multiplexer was used for enabling all images to be displayed on the computer monitor and for recording a composite video file in memory.

Results and Discussion

Slot-over-Roll: Figure 2 compared our data with those obtained by Lee, Liu and Liu [1992] using the same silicone oil for fluid. We found that replacing the surrounding air with helium maintained at 25 mbar pressure results in typically a 17% reduction in the minimum achievable thickness.



Tensioned Web Slot: Figure 3 below compares data obtained with air at atmospheric and with helium at 50mbar. A very significant reduction in the minimum achievable wet thickness is observed, typically a two fold increase in the maximum coating speed for a given minimum achievable wet thickness. The cause of the lower thickness limit when air was present was air entrainment whereas the cause of the limit when helium was present and maintained at low pressure was ribbing.



Conclusions

These experiments, while limited to much higher coating thickness than ultimately sought within industry, confirms our reasoning drawn from our previous work. It is thus possible to reduce the minimum achievable wet thickness or alternatively increase coating speed by replacing air at atmospheric pressure with helium when coating a flexible substrate using the slot-over-roll process and also when using the tensioned-web slot coating process. The work should therefore be of interest to industry involved for example in developing systems for manufacturing OPVs within an oxygen-free atmosphere.

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