# ξħ.

- 1 -

# Dr. Anye N. Chifen, Dr. Christopher Schmitt, Dr. Stephan Küper Leybold Optics GmbH, Alzenau, Germany

# Abstract

This paper reports an innovative solution for a masking oil evaporator based on flash evaporation. Particular emphasis is based on oil consumption and free margin quality. The device has been validated by data obtained under experimental and production conditions as a function of total nozzle width (area), metal layer thickness (Ohm/sq) and coating drum speed (m/s). The desired oil thickness on the web was calculated for different coating processes. Furthermore, specific aspects such as process security, oil quality, chamber contamination and cost-savings using the new free margin evaporator are discussed. Regarding these aspects, the flash evaporator offers advantages to the web coater compared to conventional free margin oil evaporators.

# 1. Introduction

Flash evaporators are units designed for an immediate and complete evaporation of the feed material without chemical degradation.<sup>1</sup> On the contrary, the traditional vapor evaporators are more like distillation devices, in which a large amount of material, e.g. oil is heated, and a relatively small amount of vapor is generated. In the case of masking oil evaporators, the oil condenses on the web.<sup>2</sup> The main differences between both processes are the evaporation temperature, amount of material to be evaporated and the oil residue after finishing the coating process. Although both processes operate under vacuum, the flash evaporation process allows for efficient and economical use of the material involved, and provides a high control over the physical and chemical properties of the condensed oil on the web.

With regard to recent advances in pattern metallization for vacuum web coatings, the oldest method used for masking free margins was a metal ribbon that prevented the deposition of metal on the substrate in the coating zone.<sup>3</sup> This method has been replaced widely by using the traditional free margin evaporator, basically a tube filled with oil, which is heated and evaporates through nozzle openings. The device requires large volumes of oil to be heated and the evaporation rate of the oil is controlled mainly by the temperature of the whole device. This poorly controlled process often leads to excess or insufficient amounts of condensed oil on the web, which in turn leads to contaminated product and/or coating machine or blurred free margins. Therefore, in order to create free margins with optimum edge steepness and minimum oil contamination of the product and machine after coating, the amount of oil on the web has to be controlled effectively.

A flash evaporator can deliver precisely the required amount of oil vapor whereby the fractional distillation is completely minimized. The advantages of the new device present economical, quality and environmental advantages.

The FME described here has been developed and tested for the capacitor web coaters at Leybold Optics, however it can be retrofitted to other web coating machines with minor alterations.

# 2. Setup of free margin evaporator

The free margin evaporator consists of three main units:

- oil reservoir and pumping system,
- oil vapor generator and
- main housing and nozzle plate
- 2.1 Oil reservoir / Pumping unit

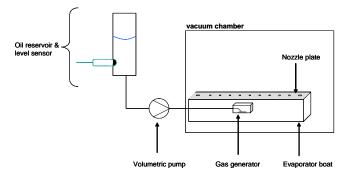


Figure 1: Schematic representation of the flash evaporator. The gas (vapor) generator and main housing are placed in the vacuum winding chamber, while the other units, oil reservoir and volumetric pump are located outside the vacuum chamber.

The oil is conveyed by a precision volumetric micro pump from the reservoir to the gas generator. The reservoir itself is equipped with a level sensor to ensure process security. The volumetric pump unit has been calibrated to deliver precisely the required amount of oil for the respective processes. Both the volumetric pump and oil reservoir are placed outside the vacuum chamber. Therefore, refill of fresh oil can take place at any time, even during process.

### 2.2 Oil Vapor Generator

The gas generator has a regulated heater circuit, and is kept at a constant temperature during all experimental conditions. The temperature is set high enough to flash evaporate the masking oil upon contact with the hot surface of the gas generator. A typical temperature for the gas generator is 230°C. The gas generator is thermally decoupled from the main housing. This unit is also thermally controlled at a lower temperature in order to prevent any condensation of the oil vapor. Both the

temperature of the gas generator and the main housing are <u>not</u> used as a means to control the oil evaporation rate. Their temperatures are set once and then kept constant for all processes. The amount of oil evaporated is controlled only by adjusting the pump rate of the volumetric micro-pump.

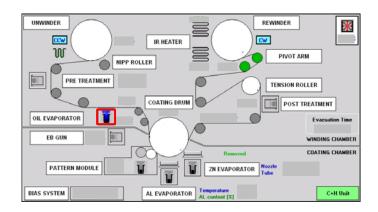


Figure 2: Screen-shot showing the position of the oil evaporator in the winding system of the web coater.

## 2.3 Main Housing and Nozzle

The main housing serves as the means to distribute the oil vapor uniformly to all nozzle openings. It is equipped with a labyrinth system for optimum spread and uniformity of free margins. Nozzle plates are mounted via a quick release mechanism to the top of the main housing and can be exchanged quickly and easily without cooling and dismantling the whole FME unit. The schematic in Figure 2 shows the position of the FME within the winding system. A typical temperature of the main housing and nozzle during operation is 180 °C.

# 3 Experimental

### 3.1 Process Parameters

The evaporator unit was characterized running a series of experiments with different web speeds, layer thickness, nozzle widths and pumping rates. For the experiments, the flash evaporator and main housing were set to constant temperatures of 230°C and 180 °C respectively, sufficient to flash evaporate and distribute the oil vapor to the nozzles without re-condensation inside the main housing. The main parameter varied to influence the evaporation rate of oil was the pumping rate. The nozzle types used are listed in table 1. Masking oil used was Fomblin® Perfluoropolyether due its excellent stable thermal and chemical properties.<sup>4</sup>

The results were evaluated by measuring the free margin width, free margin steepness, evaluation of remaining oil after coating (optical judgment by the operator) and overall oil consumption. If the optimum amount of masking oil was applied, the free margins were steep and clear, virtually no oil remained on the film after coating and the rollers itself were not contaminated.

Nozzle type	No of openings	Size of free margin /mm	Free margin %	Nozzle area /mm <sup>2</sup>
1	22 & 42	2.9 & 1.9	25	314.214
2	9	3.9	6	83.86
3	54	0.5	4.5	58.58

Table 1: Nozzle types, which were used to determine the required oil amount on the web.

The main process parameters influencing the optimum amount of masking oil are:

- web coating speed
- evaporation (pumping) rate
- nozzle type (total width of masking area)
- metal layer thickness

These factors were varied according to the values stated in Table 2 for all the samplings stated here.

After positioning the oil evaporator beneath the web with a precision positioning unit and starting the coating process, the oil flow rate was optimized, i.e. by increasing the oil flow rate until no dark margins were measured. Furthermore, the edge steepness was monitored online, which indicated sharper free margin edges upon increasing oil amount.

### 3.2 Free Margin Analysis

The free margin width was characterized both online and offline using a CCD camera based Free Margin Measuring Sensor (FMMS, Leybold Optics) and a precision measurement projector, (Digital Measuring Projector (JT12A–B)) respectively. The offline measurements were performed at ambient pressure, room temperature and humidity. All values reported here are the averages of three measurements per free margin width for all the nozzles stated above. Both characterization methods were also used to determine the free margin steepness.

#### **Coating Speed**

Film length / m	CD speed [m/s]
	5
	8
60.000	12
	15
	18

### Layer Thickness (Resistivity)

Metallization process	Resistivity [Ohm/ sqr]	
Al	1.5	
AI	4.5	
Al / Zn	6	
Al / Zn	7.5	

Table 2: Process parameters varied to determine the required amount of oil on the web

## 4 Results and discussion

### 4.1 Evaporator & Vapor Generator Temperature

For an effective flash deposition, the operating temperatures of both units were optimized at 180 °C and 230 °C ( $\pm$  0.2 °C) for the evaporator and vapor generator respectively. At these temperatures the oil was flash evaporated reliably for the flow rates the pump is capable of, and no re-condensation of oil inside the housing or nozzle was noticed. There was no need to further change or optimize the temperatures during the test runs.

### 4.2 Calibration of Pump Speed and Flow Rate

The volumetric micro-pump was calibrated to ensure that the actual oil flow rate followed the pumping speed linearly. Figure 3 illustrates the relationship between the volumetric pump speed and the oil flow. The maximum flow rate was measured at 44  $\mu$ l/sec, i.e. 100 % pump rate. The flow rate was reproducible within  $\pm$ 2%. In this report the pump rate is expressed in percentage of maximum rate [%].

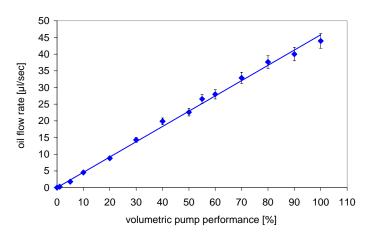


Figure 3: Oil flow rate as a function of pump speed.

### 4.3 Oil Consumption

The oil consumption of a total run could (after successful calibration of the pump) be determined by either integrating the pumping rate over time or by direct measurement of the amount used from the reservoir.

The thickness of oil on the web depends on the oil flow rate  $F_{flow(t)}$ , the web speed  $v_{(t)}$  and the total nozzle width *w*. The nozzle width *w* is a constant for a given coating run, whereas  $F_{flow(t)}$  and  $v_{(t)}$  are not. To attain a given thickness of oil, web speed and oil flow rate (pumping rate) must be balanced.

$$D_{oil(t)} = \frac{\dot{F}_{flow_{(t)}}}{v_{(t)} \cdot w}$$
 eqn 1

The web speed is normally regulated to keep the layer thickness constant, as the evaporators (e.g. zinc oven or aluminum boats) change their evaporation rate over time. Therefore, in order to keep the oil thickness on the web constant, the oil flow

rate must immediately follow the web speed, i.e. proportionally and consistently. For the new oil evaporator, this is realized by a regulator which adjusts the oil pump rate proportionally and without delay to the web speed. The total oil volume used during the coating run is then the integral of the flow rate over the time of the coating run.

$$V_{oil} = \int_{t=0}^{t=x} F_{flow(t)}$$
eqn. 2

whereby  $V_{oil}$  is the total volume of oil consumed,  $t_0$  and  $t_x$  are the time at the start and the end of the run and  $F_{flow(t)}$  is the flow rate at a given time t.

### 4.3.1 Oil Flow Rate vs. Web Speed

At constant web speed, above a certain amount of oil flow rate, no significant changes with respect to free margin width or the edge steepness could be noticed. However, a further increase of the pump rate poses the danger of depositing excess oil on the web, which would contaminate the machine. This aspect was always taken into consideration for determining the optimal (minimum) oil thickness on the web.

While the resistivity was held constant at 1.5  $\Omega$ /sqr for aluminum deposition, the coating drum speed was varied between 5 – 18 m/s. These settings were used for all the nozzles mentioned.

At constant coating drum speed, 60.000 m web was metalized using each nozzle. Figure 4 shows the relationship between the web speed and the pump rate. The optimum oil amount required increases linearly with the coating speed with good approximation. This can be observed very clearly for the "*nozzle 1*", which has 25 % free margin over a 650 mm width of web. At a web speed of 18 m/s, 65 % of the pump rate is required to yield excellent steep free margin edges. Nozzles with smaller fraction of free margin (Nozzles 1 & 2) require less oil, but follow the same linear relationship between coating speed and pump rate. Figure 4 also shows that 35 % of the pump rate is still available to mask even larger free margin areas.

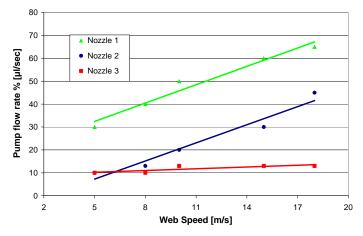


Figure 4: Pump flow rate as a function of coating speed during deposition of 1.5  $\Omega$ /sqr Al using different nozzles.

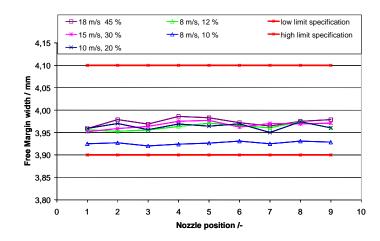


Figure 5: free margin width of *"nozzle 2"* obtained for different coating process conditions. If the pump rate is adapted properly, the free margin width can be held within tight tolerances. If the pump rate becomes insufficient, the free margin width and edge steepness deteriorates.

Figure 5 shows the free margin width across the web using "nozzle 2" for various process conditions. It is obvious that the free margin width was limited within the 3.95 and 3.99 mm for varying web speeds from 8 to 18 m/s, if the pump rate is adapted according to the relationships outlined in the equations above. For all these samples the edges were clear and steep. Figure 5 also indicates that, if the oil flow rate becomes insufficient (like at 8 m/sec and 10 % pumping rate), the free margin width decreases significantly. For this case the edge steepness was also unacceptable. The result was similar for other nozzle types. We observed the free margin width to be within  $\pm 1$  % of the mechanical nozzle opening width. No offset of the mechanical nozzle opening width was necessary.

### 4.3.2 Pump Rate as a Function of Metal Layer Thickness

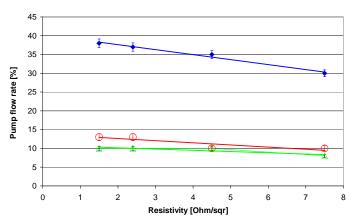


Figure 6: Oil flow rate as a function of metal layer thickness (resistivity), at constant web speed, using different set of nozzles.

Figure 6 shows the relationship between the pump flow rate required to create optimum free margins and the metal layer thickness. As already mentioned above, all the three nozzles were used. As expected, the required pump rate for good free margins decreases linearly with the metal layer thickness deposited.

### 4.3.3 Free Margin Quality

The quality of the free margins was measured with respect to their width and their uniformity across the web. Additionally, the respective edge steepness for films coated at different resistivity was measured.

Figure 7 shows the free margin as a function of nozzle position across the web for various metal layer thicknesses. There is virtually no variation of free margin width across the web. The average width obtained was 0.493 mm with a maximum variability of +/- 0.01 mm. With the specified upper and lower width limits of 0.55 mm and 0.46 mm respectively a process capability ratio  $c_{pk}$  of 4.88 was achieved for the free margin width.

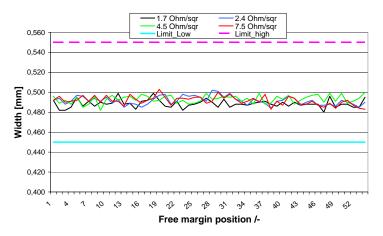


Figure 7: Free margin width distribution across the web for films deposited with different resistivity. Virtually no variation across the web is observed. The specification can be held with a cpk of 4.88.

Figure 8 shows the free margin steepness at oil flow rate optimized for respective resistivity (as explained in Fig. 6) versus metal layer thickness. At a constant CD speed of 8 m/s, *"nozzle 2"* was used to verify the free margin edge steepness. No significant trend could be concluded with respect to the steepness for the all nozzle areas presented here.

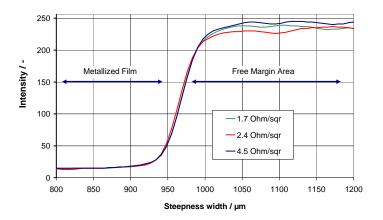


Figure 8: steepness of free margin left edge characterized for different metal layer thickness coated at constant web speed (8 m/s) at optimized pump rate using the *"nozzle 2"*.

## 5 Cost savings calculation

The new free margin oil evaporator can save costs due to its lower consumption of oil. Since the pump rate is correlated to the web speed, see equation 1, an automatic regulation of the pump rate is taking place, if the web speed and/or film resistivity change/s during coating. The oil flow rate is following the boundary conditions without delay to always apply precisely the minimum amount of oil needed. The flash evaporator can therefore be set to a pump rate just sufficient to create accurate and steep free margins. Unlike this, the traditional evaporator always has to be set to a flow rate to cover ALL process conditions (i.e. to the highest flow needed within a run). Due to the regulation, the new evaporator saves up to 35 % of oil compared to the traditional evaporator. On the contrary, in traditional evaporators, the total amount of oil has to be exchanged frequently, because the heavier residues of the deteriorating oil are collecting in the remaining oil. From time to time, the complete content of the oil reservoir has to be scrapped (and therefore wasted). The flash evaporator does not require any oil changes, as it is always supplied with fresh, nondeteriorated oil. Finally, the flash evaporator starts evaporating only, when masking is actually needed. No oil is wasted during pumping and start up as well as during the venting procedure. This provides the additional benefit of preventing contamination of the coater.

A comparative study of the standard (traditional) and the new oil evaporator based on real production data is presented in Table 3. Assuming a saving due to smaller consumption and no waste due to cleaning of the oil evaporator, savings up to  $68.000 \in$  per year can be achieved. Furthermore, this aspect prevents the contamination of rollers, which requires cleaning before each process and decreases the uptime of the machine.

Parameter	Unit	Standard	NEW
Web length	m	40 000	40 000
Price/liter	€	250	250
Consumption/run	ml	26,7	17,30
Filling	ml	500	
Refills before oil change		30	
Rolls/day		30	30
Oil consumption/day	ml	801	519
Oil waste/day	ml	500	
Cost of masking oil/day	€	200	130
Cost of wasted oil/day	€	125	
Working days/year		350	350
Cost of masking oil/year	€	70 088	45 412
Cost of wasted oil/year	€	43 750	
Saving (through regulator)			35,2%
Total cost per year	€	113 838	45 412
Savings per year	€		68 425

Table 3: Comparison of the masking costs using the new evaporator, compared to the standard (traditional) free margin evaporator.

# 6 Conclusion

A new free margin oil evaporator for capacitor film metallizers has been presented. The new unit comprises a regulated volumetric pump, which always supplies the optimum (minimal) amount of oil for masking. The development presents numerous advantages for web coating customers such as better, more precise free margins, less oil consumption and easier operation than the standard free margin evaporators. Due to the regulated oil supply and no need for total exchange of the oil reservoir, significant cost savings can be realized.

The new unit has been qualified in extensive trials and is commercially available for 650 mm and 900 mm web coating width machines.

### **REFERENCES**:

- 1. Beam W. R. & Takahashi T 1964 Rev. Sci. Instrument. 35, 1623-4
- 2. M. Silberberg, 2006 Chemistry 4. Ed., McGraw-Hill
- 3. C. A. Bishop, 2007, Vacuum Deposition, William Andrew Publishing, NY
- 4. Technical Data, product data sheet, SolvaySolexis.com