IMPACT OF METALLIZER PROCESS CONTROLS ON OPTICAL AND GAS BARRIER UNIFORMITY

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Abstract

A process study of vacuum metallizer was performed and the results in terms of optical density uniformity, metal adhesion and barrier uniformity were studied. Gas barrier was found to be a complex function of the process parameters studied while adhesion was determined to be sensitive to testing materials as well as process parameters. Visual uniformity was found to depend primarily on metallization pressure.

Introduction

Large volumes of polymer films are commercially vacuum metallized with aluminum every year and the metallization of roll substrates such as polyester, nylon and oriented polypropylene films for packaging applications are wide spread. In addition the metallization of paper is principally a decorative application where holographic or iridescent patterns are intensified by the reflective metal layer. The barrier properties of the aluminum layer are controlled by the base sheet onto which the aluminum is applied[1] but also by the deposition process[2]. In general the films are metallized to add a significant light barrier and to enhance the moisture and perhaps oxygen gas barrier properties of the film.[3] Metal adhesion is also a primary concern.

However, there are few process studies of vacuum metallizers which relate the interaction of the process and the substrate in producing a product with uniform optical and barrier properties. In the current study, a Box-Behnken experimental design was performed on a polypropylene based substrate to determine the principle metallization process variables controlling the development of metallized film properties.

Discussion

In physical vapor deposition the metal is evaporated in a vacuum and deposited directly onto the polymer part. The advantage to PVD is that high evaporation rates may be obtained which yields high production rates. The metal most often used is aluminum due to its balance in cost, developed properties and ease of evaporation.

Figure 1 shows a schematic diagram of a typical commercial metallizer. In this equipment film is unwound in a low vacuum and routed by rollers to a high vacuum evaporation chamber where the aluminum is fed by wire to a heated ceramic boat (Figure 2) where it evaporates and is deposited on the film surface passing above it on a thermally controlled roll (Figure 3). Figure 3 shows the relationship of several evaporation boats in a metallizing chamber to the film and chill roll on which the film is wrapped during aluminum deposition. The aluminum deposit from each boat overlaps to produce a continuous film across the width (Figure 4)[4] and down the length of the film. The film is then wound onto a core.

Film formulations are highly developed by film suppliers to permit excellent metal adhesion and light and gas barrier properties suitable for a wide product range and good film handling and winding properties in vacuum. Speeds in excess of 615 M/min are common. However, for a given film design and formulation the metallization process will cause variations in film properties

[1] Mount III, Eldridge, M., Wagner, John R., "Aroma, Oxygen and Moisture Barrier Behavior of Coated and Vacuum Coated OPP Films for Packaging", *J. Plastic Film & Sheeting,* V**17**(July), 2001, pp 221-237.

[2] R.L. Szoke, "Vacuum metallizing Plastic Films and Papers with Aluminum Control Parameters and Limitations A Converters Perspective", Proceedings of First International Conference on Vacuum Web Coating, R. Bakish, ed., New Orleans, November 29-December 1, 1987, pp149-158

[3] Gavitt, I.,F., "Vacuum Coating Applications for Snack Food Packaging," Proceedings of the 36th Annual Technical Conference of the Society of Vacuum Coaters (93), pp254-258

[4] Kenneth A. Taylor & E. G. Ferrari, "Design of Metallization Equipment For Web Coating", Thin Solid Films, 109(1983) 295-304

Principle metallizing variables are the film optical density, the chill roll temperature at deposition, the evaporation chamber pressure, the film speed, evaporation rate and wire composition. Of these variables the evaporation rate is contained in line speed and the wire composition has been shown to be of minor importance.

Experimental

A Box-Behnken experimental design was conducted on an 80 inch wide commercial vacuum metallizer at a constant line speed using Evaporation chamber pressure, chill roll temperature and final film optical density as the three independent process variables. Table 1 lists the variable range studied and table 2 lists the experimental runs in design order. The experiment consists of 12 treatment combinations with five center points to determine the error in the measurements. During the experiment the order of the treatment combinations was randomized and the random order used to minimize any systematic error in the testing.. The response variables were oxygen transmission rate, optical density transverse variability, metal pick off with Scotch 610 and Scotch 600 tapes.

The product design was a three layer oriented propylene film with a treated copolymer metallizing surface layer and a heat sealable surface. The film was commercially manufactured and wound on 6 inch cores to a diameter of 20 inches. One roll was used for each treatment combination tested to insure uniformity of the process variables.

Film properties were measured and then used to curve fit against the design model (Equation 1) using design variables in place of actual values (i.e. -1, 0, 1 in place of the physical magnitudes of the variables) to the determine the relative significance of each variable for the dependent variable.

$$y = B_0 + B_1 X_1 + B_2 X_2 + B_3 X_3 + B_4 X_1 X_2$$
$$+ B_5 X_1 X_3 + B_6 X_2 X_3 + B_7 X_1^2 + B_8 X_2^2 + B_9 X_3^2$$

The results of the curve fit of the experimental results to Equation 1 are tabulated in Table 3.

Results and Conclusions

Examination of Table three shows the values of the various coefficients for the independent variables P, pressure, T, chill roll temperature and OD, optical density the various interactions, P*T, P*OD and T*OD and the second order terms P^2, T^2 and OD^2. The B_0 coefficient term represents the mean value of the experimental measurements. The coefficients of each variable are compared to the ½ minimum significant effects at a 90% confidence level, ½[MIN_{90}], to determine if a significant change has been made to the dependent variable by varying the independent variable which it precedes. For instance comparing the value of B1 for TO2 shows a value of 2.88 for the change due to varying pressure. This is compared to the value of ½[MIN_{90}] =1.9 which signifies that a change in chamber pressure has a significant effect on film oxygen transmission (TO2). This comparison is made for each variable and each test result.

It is clear from table 2 that the TO2 behavior of the film is very complex. While it is expected that the OD would have a strong effect on TO2 the complex dependence on chamber Pressure was unknown as was the interaction of the pressure and the chill roll temperature. This indicates that film barrier properties are very sensitive to the metallization process and that chamber pressure and deposition temperature are critical factors in insuring a high quality barrier is developed and maintained.

Metal adhesion was found to be insensitive to tape pick off for all conditions using Scotch 610 tape. However, the use of a more aggressive adhesive tape in the Scotch 600 Tape showed a dependence on chill roll temperature and an interaction with chamber pressure and chill roll temperature. This may help explain the existence of adhesion problems in commercial laminations in spite of good apparent adhesion based upon Scotch 610 tape pick off results.

Optical density variation as measured by the number of peaks in the transverse optical density profile of the film shows a strong correlation with chamber pressure and a second order chill roll temperature dependence. Lower optical density diminishes the visibility of the peaks due to diminished contract between the average OD and the OD of the peaks. This shows that control of chamber pressure will have an impact on the OD uniformity of the film and therefore on the film properties associated with OD such as light barrier and gas barrier properties.

Therefore, based on the results of the designed experiment on metallization process conditions, we can conclude that the metallization process has a strong and complex effect on the development of barrier and adhesive properties of metallized films.

Variable	Low (-1)	Mid point (0)	High (+1)
Chamber Pressure, torr	**1x10^{-4}**	**1x10^{-3}**	**1x10^{-2}**
Chill roll Temp, ^{o}C	-15	0	15
Film Optical Density	1.6	2.3	3.0

Table 1: Independent variable ranges for Box-Behnken Experiment.

Treatment combination	Vacuum	Roll Temperature	Optical Density
1	1	1	0
2	1	-1	0
3	-1	1	0
4	-1	-1	0
5	1	0	1
6	1	0	-1
7	-1	0	1
8	-1	0	-1
9	0	1	1
10	0	1	-1
11	0	-1	1
12	0	-1	-1
Center point	0	0	0
Center point	0	0	0
Center point	0	0	0
Center point	0	0	0
Center point	0	0	0

Table 2: Experimental Design used to determine variable levels.

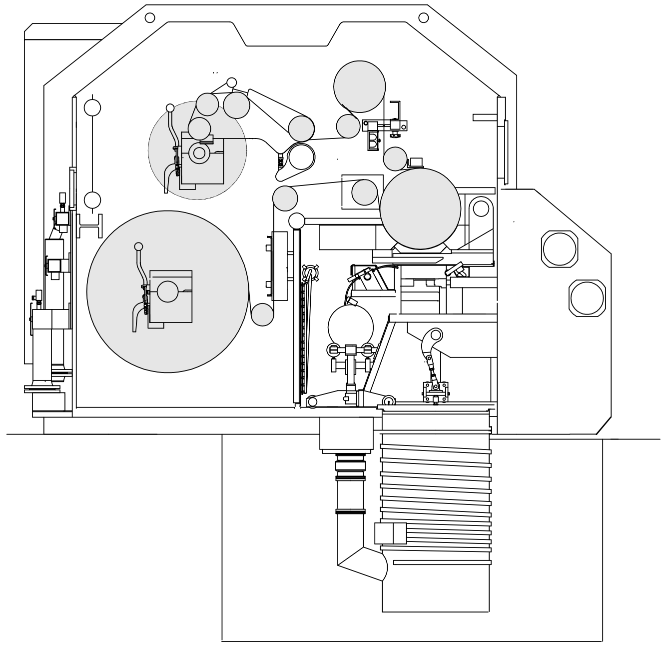

Figure 1: Schematic view of modern web Metallizer for aluminum on films, courtesy of Applied Films

Figure 2: Aluminum wire melting and evaporating on a resistance heated ceramic metallization boat, courtesy of Applied Films

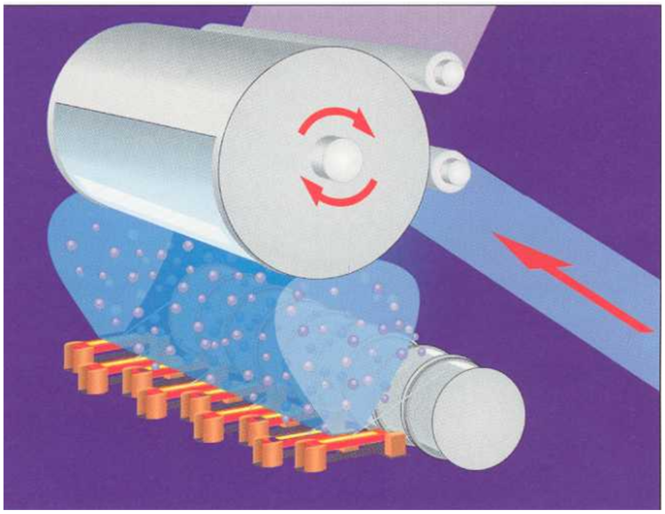

Figure 3: Schematic diagram of metallization process showing evaporation form a series of boats to produce a continuous Aluminum film coating, courtesy of Applied Films

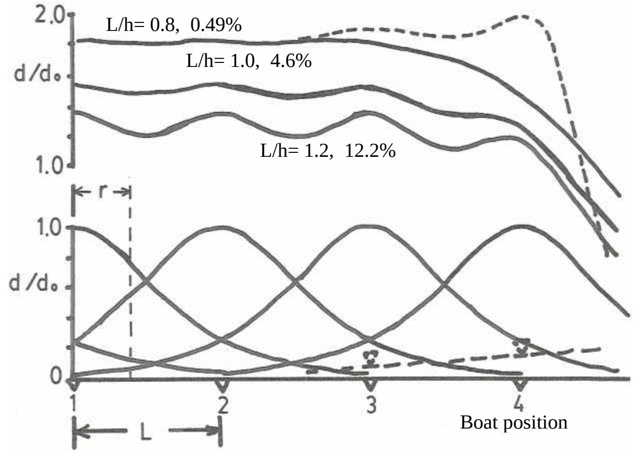

Figure 4: Figure 4 of reference 4, Schematic diagram of continuous metal layer formed from overlap of deposit from each boat

Property	½[MIN_{90}]	Mean	P	T	OD	P*T	P*OD	T*OD	P^2	T^2	OD^2
		B_0	B_1	B_2	B_3	B_4	B_5	B_6	B_7	B_8	B_9
TO2	1.9	9.67	2.88	2.06	-11.96	2.06	3.28	-0.74	-5.69	7.66	8.07
# OD peaks	1.37	5.67	2.12	0.0	-1.88	-0.25	0.0	.25	2.17	1.42	0.17
Pick off 600 tape	8.42	16.7	-3.12	-10.3	-5.3	8.75	2.50	-1.88	2.60	1.98	-0.52
Pick off 610 tape	none	none	0	0	0	0	0	0	0	0	0

Table 3; Curve fit results from Box-Behnken experiment

Key Words: Metallization, Adhesion, Barrier, Experimental Design