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Correlation of silver halide emulsion bar-coater vacuum chamber pressure fluctuations with flow pattern occurrence

Paul Fitzpatrick

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CORRELATION OF SILVER HALIDE EMULSION BAR-COATER
VACUUM CHAMBER PRESSURE FLUCTUATIONS
WITH FLOW PATTERN OCCURRENCE

by

Paul G. Fitzpatrick

A thesis submitted in partial fulfillment
of the requirements for the degree of
Bachelor of Science in the School of
Photographic Arts and Sciences in the
College of Graphic Arts and Photography
of the Rochester Institute of Technology

Signature of the Author Paul G. Fitzpatrick 4/3/82
Photographic Science and Instrumentation Division

Certified by Serafino Cardinali 5/5/82
Thesis Advisor

Accepted by Ronald Francis
Undergraduate Research Coordinator
Rochester Institute of Technology
College of Graphic Arts and Photography

Title of Thesis: Correlation of Silver Haldie Emulsion Bar-Coater Vacuum Chamber Pressure Fluctuations with Flow Pattern Occurrence

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E.I. duPont deNemours and Company, Inc.
Photo Products Department

Date 4/23/82
Correlation of Silver Halide Emulsion Bar-Coater

Vacuum Chamber Pressure Fluctuations

With Flow Pattern Occurrence

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Paul G. Fitzpatrick

Submitted to the Photographic Science and Instrumentation Division in partial fulfillment of the requirements for the Bachelor of Science degree at the Rochester Institute of Technology

ABSTRACT

A study was conducted in which the cause of flow pattern, a repetitive mottle-like defect, was investigated. Results obtained indicate that vacuum pressure fluctuations occur at the start of coating by the vacuum flow port that are 367% greater than at the center of coating. These fluctuations decay in time during the period of flow pattern occurrence, and range in frequency from 0 to 0.10 mm$^{-1}$. These pressure fluctuations correlate to flow pattern density fluctuation frequencies of the same range. Additionally, it was seen that the decay rate of the ME side of the vacuum chamber was three times the rate of the UME side. No significant pressure fluctuation phenomena was observed by the end seals of the vacuum chamber.
ACKNOWLEDGEMENTS

The author wishes to express thanks to the E.I. duPont deNemours Company, of Rochester, New York, who provided the facilities and equipment that enabled the successful completion of this project.

Acknowledgement is also given to Serafino Cardinali of E.I. duPont deNemours Company, Rochester, New York, who acted in the capacity of thesis advisor to this project.

Appreciation is also in order to Donald Gilbert of E.I. duPont deNemours Company, Rochester, New York, who enabled the author to make measurements and obtain samples during regular production hours. The author is indebted to the coater crew members whose assistance in setting up equipment allowed the work to proceed smoothly.
DEDICATION

This thesis is dedicated to the memory of Nicephore Niepce who started us all in the business of image-making and who is sure to have struggled with the task of coating asphaltum on his stone or metal plates.
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LIST OF ABBREVIATIONS AND NOTATIONS

FFT- Fast Fourier Transform
ME- Marked Edge of the coated web
$\text{mm}^{-1}$- cycles per millimeter
psi- pounds per square inch (gauge measurement)
RMS- Root Mean Square
S.P.- Standard Procedure conditions for coating the product
UME- Unmarked Edge of the coated web
**BAR COATING (AJAAB)**

- Coating Roller
- Coating Bead
- Emulsion Flow
- Coated Web
- Bar Lip
- Vacuum Chamber
- Drainage Ports
- Vacuum Force
- To Vacuum Pump
- Uncoated Web
- Coating

Diagram showing the process of coating a bar with an emulsion and the flow of coated and uncoated materials through the coating system.
INTRODUCTION
Bar Coating and Flow Pattern

A coating bar, roller and vacuum chamber are shown in figure One. The emulsion to be coated flows down the slide as indicated in the drawing. When the emulsion reaches the end of the slide, surface tension forces cause the fluid to bead. The web to be coated travels in the indicated direction with the coating roller guiding its position relative to the bar lip. A vacuum chamber is used to provide a downward force on the bead so that its position is maintained and a uniform coating is achieved.

In the bar coating process, it is periodically necessary to separate the coating roller from the bar lip. When this occurs, the bead formed by the coating fluids is disturbed and broken. When the bead is re-established, a repetitive mottle-like defect occurs for a short period of time. This defect was believed to be the result of coating bead instability and oscillation and was termed "flow pattern". (See a sample in Appendix C)

It was believed that the coating bead instability was caused by a number of factors, the most prominent being pressure fluctuations inside the vacuum chamber. Pressure fluctuations change the magnitude of the stabilizing force, allowing changes in the bead thickness to occur. These thickness changes show as density fluctuations in exposed and processed samples.

This thesis describes studies made to determine the cause of flow pattern. A method was devised that enabled conditions inside the vacuum chamber during the first few minutes of coating to be correlated to the flow pattern density fluctuations. This
information will aid in the understanding of temporary bead instability at parent roll starts, so that a more efficient vacuum system can be designed to minimize the effects of pressure fluctuations at parent roll starts on the coating quality.

Development of Bar Coating

The first published use of a bar or slide hopper for multi-layer coating of photographic emulsions was made by Russell$^{1,2}$ and Mercier$^3$. In these publications, a series of plates is described, each containing a cavity and precisely machined slot, which were used to flow photographic emulsions to a coating roller. A continuously moving web, guided by the coating roller, was used to pick up the emulsion and form the coating. The uniformity of coating was dependent on the formation of a stable coating bead, with the coating bar maintaining this bead.

Wright$^4$ and Padday$^5$ enhanced this coating method to allow thicker coatings to be applied and for emulsions with large percentages of suspended solids to be coated. Their improvement was to increase the size of the emulsion delivery slot, at the exit, to prevent particles and particle agglomerates from becoming lodged in the exit. This reduced losses from streaks caused by these particles disturbing fluid flow at the slot exit and from particles agglomerating inside the bar cavity, and subsequently flowing to the coating bead and causing a streak to form.

Jackson$^6,7$ devised a horizontal extension of the bar lip to enable a larger, more stable bead to be formed. He found the existing coating system to be linespeed limited, with a critical coating speed, above which chatter occurred. By extending the bar lip
FIGURE TWO

A.E. Beguin
Vacuum Chamber

Extrusion Coater

coating roller

Web Travel

to vacuum

Vacuum Chamber

No. 2,681,294
he found that linespeed could be increased without disturbing the coating bead and creating streaks or chattermarks in the coated product.

Use of Vacuum in Coating and Casting

In conjunction with developments in the coating bar to enhance bead stability, vacuum was being used to enhance coating uniformity. Knight\(^8,9\) first described the use of vacuum in a method for coating porous paper sheets with varnishes to provide a good printing surface. His invention was used to remove air from the coating zone on an extrusion-type coater to improve uniformity by forcing the coating solutions into the paper base. Knight's process was used primarily in the coating of paper products, and no mention is made of photographic emulsions coating.

Beguin\(^10\) (Fig. 2) first published the use of vacuum in photographic emulsions coating. He used a vacuum chamber to improve the coating over splices and minimizing material waste at parent roll starts. Beguin stated that the vacuum chamber stabilized the position of the bead after the gap between the coating roller and extrusion coater was re-established after a splice. Additionally, he claimed that the coating speed could be increased two to ten times the existing linespeeds due to the stabilizing vacuum force. Linespeeds of 300 feet per minute were claimed for a two-pass coating of photographic emulsions (coating and drying two layers, one at a time). In Beguin's patent, issued before Russell's and Mercier's, there is no mention of bar coating. Beguin's vacuum chamber was attached to extrusion and skim coating devices. Russell and Mercier both referred to the use of a vacuum chamber on a bar coater, but no details or drawings were given in the
P. Herzhoff
Suction System
Coating Roller

Web travel

A = air flow
E = emulsion flow

FIGURE FOUR
No. 3,690,917
Dieck\textsuperscript{11} described the use of a vacuum chamber in photographic film base casting. In this case, vacuum was used to remove the entrained air in the casting chamber to prevent bubbles and puckers on the film base surface. He also mentioned that the vacuum had a significant effect in minimizing defects caused by vibration of the casting equipment influencing the stability of the casting composition.

Ishiwata\textsuperscript{12}(fig. 3) patented the use of a gas jet that would impinge on the web entering a chamber of a bar coater. In this case, the gas jet (air) was used to remove dust and dirt particles adhered to the web. Additionally, the gas jet created a vacuum through the Venturi effect, enhancing bead stability. Ishiwata mentioned that the coating bead would be interrupted by the layer of air entrained on the moving web surface. The gas jet was used to block the entrance of this air into the coating bar chamber.

Metz\textsuperscript{13} improved on Dieck's invention by providing a vacuum chamber for a film casting device that would allow a uniform pressure profile to be established across the entire width of the chamber. With this device, air leakage through the edges of the vacuum chamber, which caused the non-uniform pressure profile, was eliminated by placing the vacuum suction ports at the edges of the chamber. Metz claimed improved thickness uniformity cross-web by using the vacuum chamber.

Herzhoff\textsuperscript{14, 15, 16}(fig. 4) applied vacuum use to extrusion-type coaters to improve coating uniformity. He designed a two-stage vacuum chamber, with one chamber designed to provide suction and the other to remove excess emulsion. He claimed that this vacuum
chamber minimized the influence of pressure fluctuations on the coating thickness.

The most recent patented improvement in vacuum chambers is described by Bird\(^1\) (fig. 5). A suction knife was used to remove the laminar layer of air entrained on the surface of the web entering the vacuum chamber. In this patent, the suction knife was placed inside the vacuum chamber of a bar coater to allow an increase of the coating speed. This increase resulted from a more effective removal of the laminar layer of air entrained on the moving web as compared to previously designed vacuum chambers. Bird described a critical coating speed above which the coating bead oscillated, resulting in non-uniform coating. Bird claimed increases in coating speed by 50\% or more above existing speed. Bird stated that the suction knife was to be placed 20 to 30 mils from the web surface, and that the width of the suction slot was not to exceed 50 mils. In Bird's publication, no mention is made of coating defects due to bead instability at roll starts or due to splices. Bird further states that the exact cause of the bead instability was not known, but some contributing factors had been identified (laminar layer of air entrained on the web).

Frequency Analysis

With the advent of compact digital processing equipment in the past five to ten years, it has become possible to provide an analysis of oscillatory signals to give an amplitude versus frequency spectrum. This processing, referred to as Fast Fourier Transform Real Time Analysis has been used in conjunction with vibration analysis of mechanical equipment and electronic signals.\(^1\) Lynch and Todd of duPont\(^1\), published a report where chatter, a repetitive coating defect, was traced to air knife coater vibrations by using frequency analysis of air knife acceleration measurements.
The peak vibration frequency correlated to the chatter frequency. The chatter frequency was determined by straight-forward measurement of spacing and known linespeed.

Flow pattern appears much more complex than chatter. Whereas chatter appears as one or two distinct frequencies, flow pattern appears, at first examination, as random mottle. Further study reveals a complex repetitive pattern, but the exact frequency structure cannot be determined through straight-forward measurement. Since the defect is a change in density with position, continuous density measurements were made along the coating direction, and the resultant output voltage of the densitometer Fast Fourier analyzed to determine its frequency structure in the space domain. This space domain spectra was correlated to spectra of pressure fluctuations calibrated to the space domain through linespeed.

This information enabled a comparison of spectra to be made, and revealed the relationship between pressure fluctuations and flow pattern occurrence. This information will be used to redesign the vacuum system to enable a steady-state condition to be achieved rapidly, thus minimizing the amount of coated material with the flow pattern defect.
EXPERIMENTAL

Statement of the Work and Experimental Objectives

The hypothesis tested was that the Fourier analysis of flow pattern density fluctuations could be correlated to the frequency spectrum of pressure fluctuations occurring during the start of coating.

An experimental schematic is shown in figure Six. The objective was to record the output voltage of pressure transducers during the first few minutes of coating to determine the frequency spectrum at known intervals after the coating start. These spectra would be correlated to image density fluctuation spectra of film samples taken at known intervals after the coating start. This objective was successfully completed and enabled a comparison of pressure fluctuation "signatures" to flow pattern samples, to determine the cause of the defect.

A secondary objective was to compare pressure fluctuations at various locations inside the vacuum chamber to determine a pressure fluctuation profile in the cross-web direction. Additionally, various vacuum levels were investigated to determine their effects on pressure fluctuation occurrence and decay. Each of these objectives was successfully completed and the results are displayed in the RESULTS section of this thesis.

Experimental Design

To test the experimental hypothesis, pressure fluctuation spectra were obtained at given points after the start of coating. Since the flow pattern occurred only for the first few minutes of coating, the best method to obtain the data was to record the
Pressure Measurement Schematic

Transducer Locations:
1  ME side End Seal
2  ME side Flow Port
3  UME side Flow Port
4  UME side End Seal

FIGURE SEVEN
voltage output of pressure transducers during the time of flow pattern occurrence. This recording was then played back and analyzed with a digital spectrum analyzer. To enable an accurate rendition of low frequency voltage signals, an FM tape recorder was used. In FM recording, the voltage signal is recorded by having it vary the modulation of a carrier wave frequency. The recorders have very stable drives, and by varying the recording speed, the recording frequency range is controlled from DC to 10 kHz. Using a slow speed, accurate recordings of signals in the DC to 500 Hz range can be made. Additionally, since the analog signal is indirectly recorded, tape dropout and aging do not affect the recording quality.

Density measurements were made with a scanning densitometer modified to produce an analog output. Samples obtained from the experimental coating were cut into thin strips and scanned with the densitometer. This enabled a Fourier spectrum computation for each sample, through FFT analysis of the densitometer voltage output.

Experimental Specifics, Part I

For the first experiment, the hypothesis was tested by recording pressure fluctuations at coating starts under standard coating conditions. Refering to figure Seven, for the first experiment, the pressure transducers were placed at locations 1 and 2 inside the vacuum chamber. Signals from these transducers were put into channels 1 and 3 of the FM recorder. Channel 2 was used to provide a reference signal for the recorder's built-in flutter compensation electronics which provided an improved signal-to-noise ratio for low frequencies. Voice notes were made on channel 4 to indicate
FIGURE EIGHT

SAMPLE ANALYSIS MATRIX

<table>
<thead>
<tr>
<th>Scan #, Time After Coating Start (seconds)</th>
<th>1</th>
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<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
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<td></td>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>5,56.0</td>
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<td></td>
<td></td>
<td></td>
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<td>*</td>
</tr>
<tr>
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<td></td>
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<td>7,78.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
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</table>

* these samples were not taken due to lack of material
the coating start and other information. Recordings were made for the first two minutes after the coating start, and an equivalent amount of coated material was obtained to be used to correlate flow pattern to the recordings. Then recordings were played back and analyzed in eleven second increments, using the spectral summation averaging mode of the FFT analyzer. This time period was chosen based on the sampling rate of the analyzer that gave the most information in the shortest period with the lowest noise level.

To obtain a reference of steady-state conditions, a recording was made during coating, approximately 30 minutes after the start. The data analysis consisted of obtaining a spectral average over the eleven second period of the reference recording and placing the spectrum obtained in memory "B" of the analyzer, and then making an eleven second sampling of the start recordings and placing in memory "A" of the analyzer. The ratio of memory "A" to memory "B" was then displayed. This method determined which pressure fluctuations at the start were significantly different from the pressure fluctuations at steady-state conditions. An error estimate was obtained by taking two averages of the reference recording and ratioing them to determine a noise band. This band was then drawn on each plot to determine significant pressure fluctuations.

To correlate the film samples with the pressure fluctuation recordings, a sample analysis matrix was devised (fig. 8). The film sample was cut into seven "scans" which corresponded to the time after coating start for the first seven pressure fluctuation spectra. Each scan was then cut into six "lanes", corresponding to locations on the web referenced to the ME side of the vacuum
chamber. Pressure transducer locations corresponded to lanes 1 and 5. Each sample was cut into thin strips that could be scanned with the densitometer. A reference sample was obtained from the coating at steady-state conditions, and cut into the six lanes. These samples were scanned and stored in memory "B" and an analysis similar to the pressure fluctuation recording analysis was made. This completed the work done for the first experiment.

Experimental Specifics, Part II

For the second experiment, pressure fluctuations were recorded at locations 3 and 4 inside the vacuum chamber, and at vacuum levels of standard and ± 16% of standard. A reference recording was made at each vacuum level, and an analysis similar to that used for the first experiment was completed. It was decided not to sample the web for this experiment due to the large number of samples necessary to complete an analysis in the time required. This experiment enabled a comparison of three vacuum levels and four locations inside the vacuum chamber to be made. This completed the work done in experiment two.

Results of each experiment are contained in the RESULTS section of this thesis. Due to the large number of plots obtained from each experiment, only a summary of the significant results obtained from the first two experiments is displayed.
RESULTS

The results obtained in this section are a summation of raw data obtained from each individual plot. They are intended to show the general trend of pressure and image density fluctuations as measured in experiment One and Two. A brief description of each figure number follows below.

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<thead>
<tr>
<th>Figure Number</th>
<th>Description</th>
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<td>RMS Vacuum Pressure Fluctuation vs. Time After Roll Start, Experiment One</td>
</tr>
<tr>
<td>10</td>
<td>Ratio Pressure Fluctuation RMS at Start to RMS at Center vs. Time After Roll Start, Experiment 1</td>
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<tr>
<td>11</td>
<td>Cut-Off Frequency vs. Time After Roll Start</td>
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Table One

<table>
<thead>
<tr>
<th>Table One</th>
<th>Description</th>
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<td>18-23</td>
<td>Sum of Ratio RMS Peaks Below .10 mm$^{-1}$ and Flow Pattern Rating vs. Time After Roll Start, Lanes #1 to #6</td>
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<td>24-30</td>
<td>Sum of Ratio RMS Peaks Below .10 mm$^{-1}$ and Flow Pattern Rating vs. Lane Number Scans #1 to #7</td>
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<td>31</td>
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</tr>
<tr>
<td>32</td>
<td>RMS Vacuum Pressure Fluctuation vs. Time After Roll Start for Vacuum Series Trial, By UME End Seal</td>
</tr>
</tbody>
</table>
RMS Vacuum Pressure Fluctuation vs. Time After Roll Start--Fig. 9 --

KEY for Figs. 9 and 10
- • By port @ start
- • By edge @ start
- • By port @ center
- • By edge @ center

Time After Roll Start (sec.)

RMS Vacuum Pressure Fluctuation Level (x10^{-3} PSI)
Ratio RMS Start/Rms Background vs. Time After Roll Start -- Fig. 10 --
Flow Port Cut-off Frequency vs. Time After Roll Start -- Fig. 11 --

Fluctuations by ME flow port

Fluctuations (mm$^{-1}$ x 10$^{-1}$) vs. Time After Roll Start (sec.)

0-120 sec.
### TABLE ONE

**Flow Pattern Rating**

<table>
<thead>
<tr>
<th>Scan #</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
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<tr>
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<td>0</td>
<td>0</td>
<td>5</td>
<td>2</td>
<td></td>
</tr>
</tbody>
</table>

Code Description:

0  none  
1  just visible  
2  very very light mottle  
3  very light mottle  
4  very light mottle with comb lines  
5  light mottle with comb lines  
6  mottle with comb lines  
7  heavy mottle with comb lines  
8  extremely heavy mottle with comb line—worst case
RMS Density Fluctuation vs. Time  Lane #1  Figure 12

Time After Roll Start (sec.)

Reference Level

0  20  40  60  80  100  120

10 Squares to the Inch

4  8  12  16  20  24  28  32
RMS Density Fluctuation vs. Time  Lane #2  Figure 13

Reference Level
RMS Density Fluctuation vs. Time  Lane #3  Figure 14

Time After Roll Start (sec.) 0 20 40 60 80 100 120

Reference Level

0 4 8 12 16 20 24 28 32

RMS Density Fluctuation (UX - VOLTS)

Squares to the Inch
RMS Density Fluctuation vs. Time  Lane #5  Figure 16

Reference Level
RMS Density Fluctuation vs. Time  Lane #6  Figure 17

Time After Roll Start (sec.)

Reference Level
Lane #1
Sum of Peaks Below .10 mm⁻¹ and Flow Pattern Rating vs. Time  Fig. 18

Lane #1
Sum of Peaks Below .10 mm⁻¹ and Flow Pattern Rating vs. Time  Fig. 18
Sum of Peaks and Flow Pattern Rating vs. Time  Lane #3 Figure 20

Time After Roll Start (sec.)

Flow Pattern Rating

Sum of Peaks Below .10 mm⁻¹ (volts)

Time After Roll Start (sec.)
Sum of Peaks and Flow Pattern Rating vs. Time Lane #4  Figure 21

Sum of Peaks Below 10 mm⁻¹ (volts)

Flow Pattern Rating

Time After Roll Start (sec.)

0 20 40 60 80 100 120
Sum of Peaks and Flow Pattern Rating vs. Time
Lane #5  Figure 22

Time After Roll Start (sec.)

Flow Pattern Rating

Sum of Peaks Below .10 mm \text{min}^{-1} (volts)
Sum of Peaks and Flow Pattern Rating vs. Time  Lane #6  Figure 23

- **Flow Pattern Rating** vs. **Time After Roll Start (sec.)**
  - **Y-axis:** Flow Pattern Rating
  - **X-axis:** Time After Roll Start (sec.)

**Graph Details:**
- **Upper Graph:** Shows a linear increase in Flow Pattern Rating with time, peaking around 40 seconds and stabilizing afterward.
- **Lower Graph:** Depicts a sharp increase followed by a decrease in Sum of Peaks Below .10 mm^-1 (volts), with a notable peak around 40 seconds.
Sum of Peaks and Flow Pattern Rating vs. Lane #  Scan #1  Figure 24

Lane Number

1 (t)  2  3  4  5 (t)  6

Flow Pattern Rating

0  4  8  12  16  20  24

Sum of Peaks Below .10 mm-1 (volts)

0  4  8  12  16  20  24

Lane Number

1 (t)  2  3  4  5 (t)  6

(t) denotes transducer location
Figure 25

Sum of Peaks and Flow Pattern Rating vs. Lane # Scan #2

Lane Number

1(t) 2 3 4 5(t) 6

Lane (t) denotes transducer location

Flow Pattern Rating vs. Lane # Scan #2 Figure 25

1(t) 2 3 4 5(t) 6

Lane Number

Sum of Peaks Below 0.10 mm^-1 (volts)
Sum of Peaks and Flow Pattern Rating vs. Lane #  Scan #3  Figure 26

Lane Number

Lane Number (t) denotes transducer location
Sum of Peaks and Flow Pattern Rating vs. Lane # Scan #4 Figure 27

Lane Number (t) denotes transducer location
Sum of Peaks and Flow Pattern Rating vs. Lane # Scan #5 Figure 28

Lane Number (t) denotes transducer location.
Sum of Peaks and Flow Pattern Rating vs. Lane #  Scan #6  Figure 29

Lane Number

Scan #6

Figure 29

Lane Number

(t) denotes transducer location

Squares to the Inch
Sum of Peaks and Flow Pattern Rating vs. Lane # Scan #7 Figure 30

Lane Number

Flow Pattern Rating

Squares to the Inch

Lane Number (t) denotes transducer location
RMS Vacuum Pressure Fluctuation Level vs. Time and Vacuum Level (by UME flowport) Figure 31.

- Time after roll start (sec.): 120, 160, 200
- Reference Level (UME)
- Vacuum Level at Standard +16%
- Vacuum Level at Standard -16%

(10^-3 PSI)
DISCUSSION

Experiment One

The objective of this experiment was to correlate flow pattern occurrence to pressure fluctuations through comparison of Fourier Spectrum Analyses. In experiment One, the hypothesis was tested to determine if a correlation existed. Recordings made under standard conditions were analyzed. Results from this analysis are shown in figure Nine.

Refering to this figure, it is seen that the RMS pressure fluctuation level by the flow port (position 2) is significantly higher at the coating start as compared to the coating center. It is also seen to decay in time and approach the level observed at the coating center within the time of flow pattern occurrence. In contrast to this observation, the RMS pressure fluctuation by the end seal (position 1) is not significantly different at the coating start as compared to the coating center. There is no observeable decay and it appears that the random changes in the level are due to measurement error.

Refering to figure Ten, a similar observation is made comparing the ratio of the RMS level at the start to the level at the center. The importance of this comparison is shown in figure Eleven, the cut-off frequency versus time after coating start. This plot indicates that the energy contained in the pressure fluctuations by the flow port extends to approximately \(0.10 \text{ mm}^{-1}\) and decays to 0 frequency within the time of flow pattern occurrence. This information was obtained from the plots contained in Appendix A.

To summarize the measurements from experiment One, pressure fluctuations occurred by the flow port at the coating start that were 367\% greater than fluctuations at steady-state coating.
No significant difference was seen for pressure fluctuations seen by the end seal. The energy contained in the fluctuations by the flow port decayed in time corresponding to flow pattern occurrence. The frequency range of these fluctuations started at approximately 0.10 mm\(^{-1}\) and decayed to 0 mm\(^{-1}\) within the same time period. This information was used to provide the frequency range to be examined on sample density fluctuation spectra to check for similar decaying behavior. This would test the experimental hypothesis.

The samples obtained were cut into the sample matrix as discussed in the experimental section, and then visually rated for the flow pattern defect. Refering to Table One, the results from this rating are seen. In general, the flow pattern is seen to decay in time and to move toward the center of the web. Initially, the flow pattern is worse on the edge and then builds to peak levels, rated 8, on lane 3, scans 2 to 4. This level continues to move toward lane 6 and is decaying with time. Thus the visual observations of flow pattern occurrence trends on the film samples correlate to the general trends in pressure fluctuation measurements.

Figures 12 to 17 show a quantitative analysis of the flow pattern density fluctuations. These indicate that the overall RMS fluctuation level for each lane as compared to the RMS fluctuation level for the reference sample. The density fluctuations decay to the reference level for each case except lane 2. Additionally, the general trend of peak density fluctuation is observed to move towards the center. The behavior of lane 2 is not consistent with visual observations and is considered a flyer. Every other lane compared exactly to the visual rating of flow pattern severity.

To correlate density fluctuations with pressure fluctuations, it was necessary to analyze density fluctuations at frequencies
below 0.10 mm$^{-1}$. This analysis is shown in figures 18 to 23, by lanes, and figures 24 to 30, by scans.

Refering to figures 18 to 23, the sum of all density fluctuation peaks below 0.10 mm$^{-1}$ versus time after roll start is shown. These numbers also reflect a correction for the noise estimate, determined with the same method as the pressure fluctuation noise estimate. Again the general trend of decay in time, and movement of the worst case flow pattern towards the center is seen. The peak flow pattern level is seen to occur in lane 3 which also has the greatest level of image density fluctuation at frequencies below 0.10 mm$^{-1}$. Again, each lane is seen to decay except lane 2. However, the density fluctuation level at scan 7, lane 2 is 7 volts versus 22 volts for the location of level 8 flow pattern.

Refering to figures 24 to 30, the behavior of density fluctuations on each scan is compared between lanes. Scan 3 to 5 in lanes 3 to 5 show the highest level of density fluctuation, 10 to 22 volts, and the highest judgemental rating of flow pattern. Again, the trend of decay in time is seen and the movement of peak density fluctuations towards the center occurs.

To summarize experiment One, pressure fluctuations seen at the start of coating are 367% greater than during steady-state coating, and decay in approximately 100 seconds to the level seen at steady-state. The frequencies observed that were significantly different from reference recordings ranged from 0.10 mm$^{-1}$ at the start to 0 within the decay period. Correlating to these pressure fluctuations are image density fluctuations on flow pattern samples. In the same frequency range, the image density fluctuations are seen to decay within approximately 80 seconds to reference levels, and to move
towards the center of the vacuum chamber. Thus the experimental hypothesis is accepted and vacuum chamber pressure fluctuations seen at the coating start correlate to flow pattern density fluctuations.

Experiment Two

The objective of this experiment was to compare pressure fluctuations between end seals and flow ports of the vacuum chamber. Additionally, the effect of vacuum pressure level on pressure fluctuation behavior was measured.

Refering to figure 31, the pressure fluctuation by the UME port is compared to the ME port, at the standard vacuum level. The ME port is seen to decay in 100 seconds as compared to the 280 second decay period of the UME port fluctuations. The reference level for the UME port is $8.3 \times 10^{-3}$ psi versus $3.3 \times 10^{-3}$ psi for the ME port. Comparing general decay behavior, the ME port fluctuations decay in an exponential fashion, while the UME port fluctuations cycle between decaying and increasing during the overall period of decay.

Comparing the three vacuum levels, the references are 8.1, 8.3 and 7.6 ($x10^{-3}$ psi) for S.P.±16%, S.P. and S.P.±16% respectively. It is believed that there is no significant difference between these levels. Comparing the decay rates, each plot tends toward the reference level in 280 to 300 seconds after the coating start. The decay behavior is similar between each vacuum level, with cycles between decaying and increasing during the decay period. There appears to be no significant effect on the decay rate at different vacuum levels for the vacuum range tested. However, there is a significant difference between the UME and the ME flow port pressure fluctuation decay period and between the reference levels. A general comparison of physical quality evaluation samples
shows that the UME side of the web still has flow pattern after the ME side has reached steady-state.

Refering to figure 32, the vacuum level trial for end seal fluctuation is seen. There is no real decaying observed during the measurement period for each of the vacuum levels. Refering to the reference levels, they are 13.8, 18.8 and 19.8 ($10^{-3}$psi) for S.P. -16%, S.P., and S.P. +16% respectively. This follows logical progression considering that a higher vacuum would require higher airflow through the given end seal gap and this would produce a noisier spectrum. What is interesting in this series of plots is that both the S.P. -16% and the S.P. +16% are higher than the reference level until approximately 220 seconds. However, the S.P. level fluctuation moves randomly about its reference level for the entire measurement period. Comparing the running control samples, the sample at S.P. -16% is the worst for flow pattern on the UME side, while the S.P. and S.P. +16% are about the same. This suggests a correlation between end seal fluctuation and flow pattern severity. However, an exact quantification of this correlation is beyond the scope of this project.

To summarize experiment Two, it was seen that vacuum pressure fluctuations by the UME flow port decayed in 300 seconds as compared to 100 seconds for the ME flow port. The reference level seen by the UME port was 2.5 times the level seen by the ME port. Comparing performance by the UME flow port at varying vacuum levels, no significant effects were seen. For the end seal fluctuation, again the UME reference level was higher than the ME, 19.8 vs. 3.7 ($10^{-3}$ psi), however no significant decay patterns were observed. One interesting effect was seen in the vacuum series trial in which both S.P. -16% and S.P. +16% pressure fluctuation levels were seen
to randomly vary above the reference level, while the S.P. vacuum pressure fluctuation varied randomly about its reference level. To conclude experiment Two, the pressure fluctuations by the UME flow port are at a higher level and decay at a slower rate than fluctuations by the ME port. This phenomena was confirmed by visual inspection of physical quality evaluation samples which showed higher flow pattern severity on the UME side as compared to the ME side. No decay rate was effected by the adjustment of vacuum level. For pressure fluctuations by the end seal, the UME side was seen to be 435% greater than the ME side at the reference level. An interesting phenomena was observed where the S.P.-16% and the S.P.+16% vacuum levels had pressure fluctuations that varied randomly above their respective reference levels during the measurement period, while the S.P. level fluctuation varied about its reference level.
CONCLUSION

The objective of this thesis research was to correlate pressure fluctuation frequency spectra to flow pattern density fluctuation spectra. This objective has been achieved and the experimental hypothesis accepted. The second hypothesis, that vacuum level will affect the decay rate of pressure fluctuations seen in the first experiment has been rejected for the vacuum range tested.

For general comparisons, the UME side of the vacuum chamber decay rate is one-third the rate of the ME side, and the overall noise indicated by reference levels is 2.5 times the ME side.

This information provides a basis for further work that can be done to minimize the effect of these fluctuations on coated material. Several items can be examined which may eliminate some of the fluctuations. The first is to examine the bar-coating roller geometry to ensure that each time the bar and roller are separated the geometry returns to its setpoints when the bead is re-established. It is believed that the repeatability of the geometry on the UME side is much worse than the ME side. Overall, however, it is felt that the fundamental problem lies in the vacuum system's inability to reach steady-state in a short time span. Factors that affect this performance are vacuum chamber port design, vacuum pump size and hose size. Each of these parameters needs to be investigated to enable a smoother vacuum system to be obtained.

In general, the work completed on this thesis project indicates that the vacuum system is the major cause of flow pattern and that various vacuum system parameters need to be investigated to eliminate the defect.
REFERENCES


APPENDIX A

This Appendix contains the series of pressure fluctuation frequency plots that were used to generate figure #10. The plots are numbered successively from the roll start by odd numbers, with plot #49 being used for error estimation. The plots represent the ratio of spectrum "A" in the FFT analyzer memory to spectrum "B". The plot is scaled to a reference of 1.00 so that the line marked 1.00 would reflect an equal power at the coating start and the coating center. Spectrum "A" consists of 8 spectral averages and spectrum "B" of 32 averages. The reason for 8 averages is that it represents 11.2 seconds of voltage measurements and is felt to provide the best noise level without averaging out too much data. Wherever frequencies rise above the line marked +E, the power at that point is larger than at steady-state coating. Values that fall below the line -E are less than the power at steady-state. These points are considered to be transient noise and are not considered in the analysis since they represent a condition of less fluctuation and therefore, greater vacuum stability. These points are not believed to be causing any coating disturbances since they tend toward a more uniform steady-state condition.
APPENDIX B

Equipment Used

Digital Spectrum Analyzer:
   Nicolet Scientific Model 444A Real Time Spectrum Analyzer

Pressure Transducers
   PCB Model 106B50 piezoelectric dynamic pressure transducer with PCB amplifier to provide 1X, 10X, or 100X gain

FM Recorder
   Racal Store-4 DS four channel FM tape recorder with Dual Standard FM electronics

Digital Plotter
   Tektronix 4660 single pen plotter connected to the spectrum analyzer via RS-232 interface.

Scanning Densitometer
   The scanning densitometer is a proprietary piece of equipment owned by duPont Co., Inc.
APPENDIX C

Flow Pattern Sample

coating direction

Flow Pattern
VITA

The author, who is married and has one child, was born on 1/25/58 in Niagara Falls, New York. He has lived in the Upstate New York area since then. He attended Fairport Senior High School, graduating in 1976 with a Regent's Diploma with Honors. He began attendance at the Rochester Institute of Technology in 1976, originally in the Photographic Illustration Department. After graduating with an A.A.S. with High Honors in 1978, he transferred to the Photographic Science and Instrumentation Division. He received an A.A.S. degree with High Honors in 1980 and will complete his degree requirements in 1982, for the Bachelor of Science degree.

The author began working for the E.I. duPont deNemours and Co., Inc., in 1979 as a summer employee in the Technical Coating and Finishing Group. He joined the company full-time in 1981, where his main responsibilities have been in the area of Coating. His main career interests lie in the photographic manufacturing area, which he will continue to pursue upon graduation.