

MEASUREMENT OF MULTI-LAYER STRUCTURES USING NON-CONTACT REFLECTIVE LIGHT TECHNOLOGY

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Abstract:

There are over ten thousand extrusion and coating lines that require some sort of gauging to determine the thickness of coatings, interlayer thickness or overall thickness. There are various methods currently available to provide this information but none provide the speed and accuracy desired by today's manufacturers of sophisticated material structures. Current technology, developed by Lumetrics, allows rapid on-line and off-line inspection of multi-layer translucent plastics and coating applications. Results of on-line and off-line testing and inspection systems will be presented.

Introduction:

Lumetrics, an optical measurement solutions company from Rochester, New York, developed a new interferometric device based on technology licensed from Eastman Kodak Company. The OPTIGAUGE™ Thickness Measurement System has applications in various markets measuring everything from multilayer plastics and films to tubing and glass of various types. A description of some of these capabilities will be described. Results will be presented of tests conducted in the measurement of single and multi-layer films both on a moving web and in lab testing. The tests were conducted during 2004 and 2005 numerous locations.

The tests showed consistent reading of films at various speeds on multiple lines with different materials. The off-line tests also showed consistent results in both a five layer medical film and in measuring a multi-layer blister pack.

Operation and test examples:

The technology used is a high-precision, fiber-optic, dual interferometer called the OPTIGAUGE Thickness Measurement System, which uses light reflections to provide extremely accurate and rapid measurement of multi-layer webs and coatings. The OPTIGAUGE uses two light sources, a simple light source (Light Emitting Diode or LED) to produce reflections from surfaces and a second light source (laser) to accurately calibrate those reflections.

Light is projected from an LED onto a surface. When light hits the surface some light is reflected back and some continues through the material. At each new surface or layer, more light is reflected back, and these light reflections are directed to the system. The index of refraction between the different layers must be different enough for the system to detect a difference but that difference is slight. Reflected light is directed to fiber stretchers, which cause the generation of interference patterns and measurement peaks.

The laser acts as an internal clock and reference to measure the distance between these peaks. A signal converter

and processor contain the specialized algorithms and application software that produce extremely accurate physical measurements. These calculations are all done in real time and then this information displays on the screen. Measurements can be transferred to software that controls the production process.

The OPTIGAUGE technology is useful in measuring many types of materials. The system has been used to test multilayer plastics, which will be described as part of this paper. The system is extremely flexible in what it can measure and also how it can measure. Multiple applications can be implemented because the probe can be remote from the dual-interferometer, and because various types of probes can be swapped in and out depending on the application. Some of these applications will be described below. Additionally, single layer and multilayer medical tubing can be measured with the OPTIGAUGE.

Single layer film:

Two tests of the OPTIGAUGE were conducted on a cast film line. The tests were made to prove the ability of the system to measure both single thickness and multi-surface plastics along a moving web. In the first test, single thickness polyester with a nominal thickness of approximately 54 microns was tested. The sample was measured; using a single probe, *Figure 1*, at rest and then at graduated speeds from 50 ft per minute to 600 ft per minute.

The OPTIGAUGE was consistently measuring 53 microns across all speeds. An adjustment was later discovered that accounted for the one-micron difference

in these sets of measurements. Additional measurements were taken and verified via micrometer gauges and a beta gauge.

The test results are indicated in the *Table 1* below. The optical thickness must be divided by the index of refraction for the material being tested to obtain the actual measurements. An average 1.5 index can be used for very accurate approximations.

The standard deviation of the material (4th and 5th column) showed that the film thickness was constant across the many feet of plastic sheet that went by. The averaging standard deviation of the machine (7th and 8th column), which shows the variation of the instantaneous measurement of thickness, was less than 1 micron. This shows that the OPTIGAUGE was producing precise data. The number of data points taken at 1 per second was actually an average of 30 per second and that average was recorded as a single data point by the system.

The results show that film speed seems to have little or no effect on the data. Based on this data, speeds of 2,000 ft per minute and above should show the same consistency of measurement. This test showed the efficacy of the OPTIGAUGE in measuring single thickness plastics across a moving web.

3 Layer label stock:

Improvements were made to the OPTIGAUGE because of the special challenges of measuring multi-layer plastics both in the lab and across a moving web. As a result a new electronic filtering and amplification circuit was added to make the

OPTIGAUGE more sensitive to multi-layer materials.

The second test was conducted on a three-layer sample of polyester, adhesive and polypropylene with nominal thicknesses of 32, 17, and 49 microns respectively.

Tests were taken while the sample was still and then at speeds of 50, 150, and 300 ft per minute. Faster speeds were not possible during this test because of the small sample roll size. For the static measurements, data was taken at a 30 Hz rate average; 30 samples thus give data points at 1 per second. *Table 2* shows the results of a data run at 300 fpm.

Medical film:

The third test was conducted on a live production line of a specialty film manufacturer. The tests involved setting up the probe over a line that was applying coating to a polyester base. A photograph of that setup is shown in *Figure 2*. Note, that the probe was installed with tie wraps to mount it and a nickel to adjust alignment. This was due to the temporary nature of the testing. Even with this crude arrangement the return signal from the probe was excellent and measurements were easily taken.

The data set consisted of logging the base-line adhesive thickness and then making changes to the line speed to induce a thickness increase as well as a thickness decrease. A base line was taken for about 30 minutes. Then the line speed was slowed at approximately 5:39 PM. This new thicker layer was left for about 5 minutes and then the line speed was increased at about 5:46 PM. This

thinner layer was run for approximately 5 minutes. The *Figure 3* graph shows a plot of this data run.

From the data on *Figure 3* the change in thickness is clearly evident as the line slowed down and was sped up. Additionally there is a wave pattern that can be seen which indicates a potential problem with a process or equipment. These types of issues might never be seen with standard measurement and inspection systems. If a standard manual inspection was done on this film at a single point, decisions would be made that do not reflect the true condition of the film.

Many materials can be scanned with a tabletop system that provides a cross sectional view of the material.

Lumetrics scanned a five layer medical film with the X / Y setup shown in *Figure 4*.

The data was dumped to an excel spreadsheet where calculations were performed to determine pass or failure of the film. The scanner system allows the data to be presented in any number of fashions. By examining this data the manufacturer is able to detect problems and issues both down and across the web. This also allows the manufacturer to examine layer consistency to ensure the correct amount of material is used.

Lid stock:

In addition to the medical film, a multi-layer, thermo-seal laminated, center foil lid stock was examined as well. The lid stock was manufactured for Company A and they were experiencing delamination problems with the film. Delamination of the lid stock's heat seal film from the

aluminum foil resulted in leaks that were detrimental to the sterile packaging properties of the Customer A's product.

Lumetrics used the OPTIGAUGE system to measure the thickness of the heat seal film/adhesive layer of a reference lid stock sample with an average thickness of 31.59 microns. Since the print thickness specification of the heat seal film is 25.4 microns, it was calculated that an additional adhesive layer of approximately 6 microns was present. These results confirmed the Lumetrics OPTIGAUGE system's ability to effectively determine if an adhesive layer was applied, and whether this adhesive layer was of an appropriate thickness for the lid stock. In operational use, this measurement would be accomplished using a two-probe OPTIGAUGE system to measure the thickness of the heat seal film before lamination, and the heat-seal side of the lid stock after lamination.

Test description:

Company A uses a thermo-seal aluminum foil lid stock, manufactured by "Supplier B", for its sterile packaging. *Figure 5* shows the print specifications describing this lid stock and their dimensions:

Company A provided Lumetrics with various samples of "Supplier B's" aluminum lid stock used for its sterile packaging. These samples were used to analyze the heat seal film side of the lid stock to determine if there was a difference between reference lid stock material and lid stock that had experienced unacceptable levels of delamination. The OPTIGAUGE Film Thickness Measurement system was

used to analyze the various aluminum lid stock thicknesses using a standardized measurement approach:

- Heat seal film with adhesive was measured at 10 distinct points.
- OPET film with adhesive was measured at 10 distinct points.
- Delaminated OPET film was measured at 10 distinct points.
- Delaminated OPET film and the total lid stock thickness were measured using a mechanical hand micrometer.

Test results:

Heat Seal Film Side

The thickness of the combined heat seal film/adhesive layer of the reference aluminum lid stock was measured at ten separate points. The data in *Table 3* shows an average thickness of 31.59 microns.

- Assuming a heat seal film thickness of 25.40 microns as given on the aluminum lid stock print, an adhesive layer is shown to be present with a calculated thickness of ≈ 6.19 microns.

The Lumetrics OPTIGAUGE system is designed to measure multiple film layers simultaneously with a minimum layer thickness of 12 microns needed to make an accurate measurement. Given this requirement, the OPTIGAUGE system cannot differentiate between the adhesive layer that was assumed to be approximately 6 microns and the heat seal film layer. Instead, it "sees" only one thicker layer and, as described above, can differentiate between the 25.40 microns thick heat seal film and a thicker (31.59 microns) layer comprised of the heat seal film *and* adhesive.

Therefore, using the OPTIGAUGE system to measure the thickness of the heat seal film before and after the application of adhesive would ensure that adhesive is applied, and in the appropriate amount.

- This measurement process is demonstrated with the test results from the samples of the aluminum lid stock that exhibited delamination of the OPET film.

OPET Film Side

The thickness of the combined OPET film/adhesive layer of the reference aluminum lid stock was measured at ten distinct points with an average thickness of 18.01 microns. The thickness of the OPET film that was identified as having no adhesive applied was measured at 10 distinct points with an average thickness of 13.98 microns. See *Table 4*.

Therefore, an adhesive layer with a calculated thickness of 4.03 microns is assumed to be present on the reference aluminum lid stock.

The OPET film without adhesive was also measured using a mechanical hand micrometer, see *Table 5*. This measurement is compared, in the table below, to the average thickness of the OPET film measured by the OPTIGAUGE system and the print thickness specification.

Total Lid stock

The total thickness of the aluminum lid stock was calculated below using the measured thicknesses of the Heat Seal Film/Adhesive layer and the OPET Film/Adhesive layer, and the print thickness of the aluminum foil. This thickness was compared to the total thickness measured using a mechanical hand micrometer and the total thickness specification, *Table 6*.

Blister Pack:

Customer C was interested in mapping the wall thickness of blister packaging that uses its barrier film. The information provided from a wall thickness map was used to ensure that the performance qualities of the film were maintained after the blister forming process. Therefore, Customer C asked Lumetrics to use its OPTIGAUGE system to provide a thickness map of the blister sample.

The results of this thickness mapping analysis demonstrate the ability of the OPTIGAUGE system to measure the total wall thickness and the thickness of each individual film layer at the top, side, and bottom of a single blister. Because this system uses light delivered by a fiber-optic cable, it can measure small areas such as various portions of a single blister. And, the OPTIGAUGE system does a complete analysis by simultaneously measuring the thickness of all layers of the blister.

Test description:

- A total of 27 measurement points were analyzed for the thickness map.
 - 10 points around the top of the blister
 - 5 points along each side of the blister
 - 5 points along the bottom of the blister
 - 1 point on each end of the blister
- The blister used for the analysis was found to have three individual film layers. The thickness of each layer and total thickness were measured simultaneously at each test point.

- The data was consistent with the expected results. See *Figures 6-9*.

Conclusion:

The data from the three moving web trials shows consistent readings across the various speeds. Measurements were confirmed with micrometer readings where possible. The standard deviation of the data did not change as the speed was increased indicating that there was no degradation in the data quality. Expectations are that similar data with higher web speeds could easily be obtained. Testing also showed that the system performed best when the mounting system was designed to limit vibration.

Results for static, single layer, and multi-layer films showed that OPTIGAUGE technology is usable and extremely beneficial in the measurement and production of multi-layer plastics. This includes both simple flat films as well as more complex blister packages for medical applications. Additionally, multi-layer films with metal or opaque inner layers can also be measured using subtractive means during production. Systems can be effectively used on-line or off-line to verify layer and coating thicknesses.

There are numerous other applications that can use this technology because of its flexibility in setup and use. Online and offline applications are continuously being investigated with great results.

Figures:



Figure 1: Probe setup

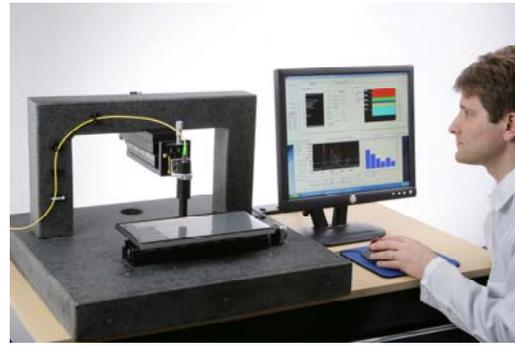


Figure 4 - Medical film station



Figure 2 - Film mfg test setup

F402 Heat Seal Film	0.0010"	1.0 mil	25.4 microns
F384 Adhesive	2.4 lb/3000 sq. ft.		
3003-0 Aluminum Foil	0.003"	3.0 mil	76.2 microns
F417 Adhesive	2.5 lb/3000 sq. ft.		
F265 OPET Film	48 GA	0.47 mil	12.0 microns

Thickness 0.0048" ± 10%, 121.92 μm ± 10%

Figure 5 - "Company A" foil lid stock print specifications

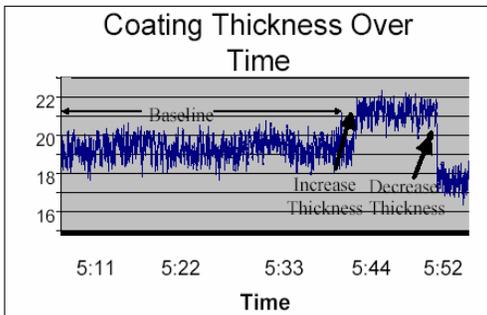


Figure 3 - Coating thickness

Test results:

Total Thickness (micrometers)

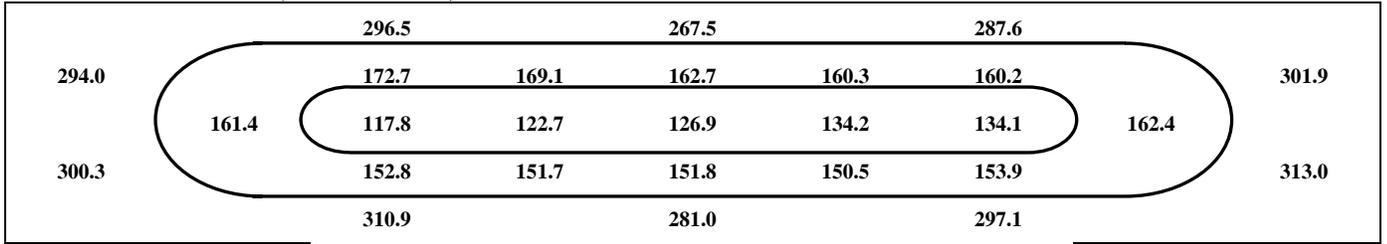


Figure 6 – Total blister thickness

Outer-Wall Thickness (micrometers)

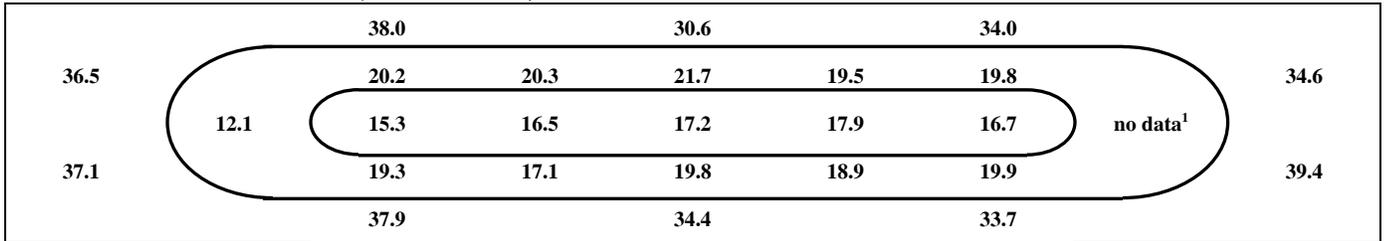


Figure 7 – Outer wall blister thickness

¹ Could not detect two layers, but only one layer with a thickness of 19.4 microns. The thickness of each individual layer (< 10 microns) is less than the minimum thickness required by the OPTIGAUGE system.

Middle-Layer Thickness (micrometers)

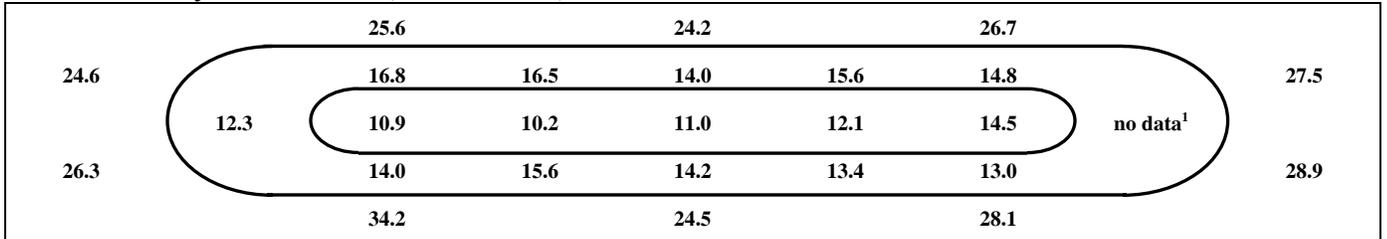


Figure 8 – Middle wall blister thickness

¹ Could not detect two layers, but only one layer with a thickness of 19.4 microns. The thickness of each individual layer (< 10 microns) is less than the minimum thickness required by the OPTIGAUGE system.

Inner-Wall Thickness (micrometers)

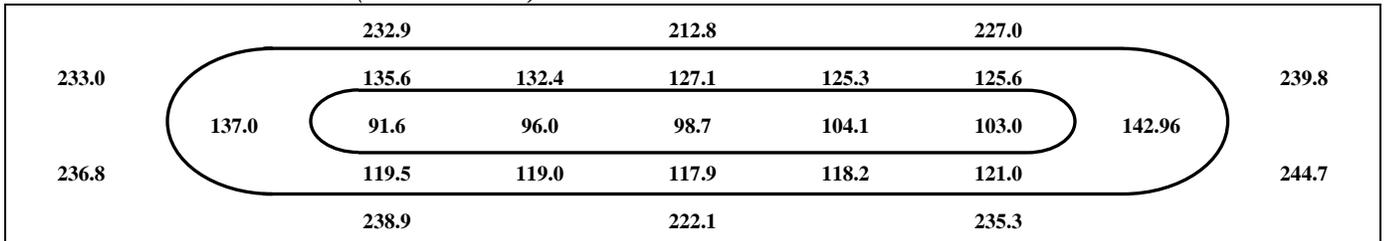


Figure 9 – Inner-wall blister thickness

Summary of Data

Material: Polyester Film

Web Speed (ft/min)	Average Optical Thickness		Standard Deviation		Number of Data points taken at 1 per second (30 Hz. Averaging)	Averaging StDev	
	(Microns)	(mils)	(Microns)	(mils)		(Microns)	(mils)
50	80.290	3.161	0.321	0.013	193	0.625	0.025
100	80.320	3.162	0.311	0.012	162	0.948	0.037
150	80.444	3.167	0.299	0.012	165	0.952	0.037
200	80.348	3.163	0.253	0.010	115	0.943	0.037
250	80.357	3.164	0.238	0.009	128	0.899	0.035
350	80.405	3.166	0.268	0.011	121	0.939	0.037
450	80.202	3.158	0.223	0.009	103	0.918	0.036
550	80.250	3.159	0.250	0.01	153	0.872	0.034
600	80.329	3.163	0.210	0.008	156	0.854	0.034

Table 1 – Single layer thickness

Test 2: Web speed 300 fpm				
	Avg. Thick	Std Dev	Max	Min
Total thickness	100	0.78	101.67	99.42
Polypropylene with adhesive	67.71	0.87	68.97	66.82
Polypropylene	50.13	0.58	50.75	49.22
Adhesive (calculated)	17.58	NA	18.22	17.6
Polyester	32.4	0.32	32.75	31.82

Table 2 Multi-layer thickness measurement

In microns	Heat Seal Film/ Adhesive Thickness	Heat Seal Film Print Thickness	Calculated Adhesive Thickness
1	31.72	25.40	6.32
2	31.40	25.40	6.00
3	31.39	25.40	5.99
4	31.74	25.40	6.34
5	31.65	25.40	6.25
6	31.56	25.40	6.16
7	31.50	25.40	6.10
8	31.75	25.40	6.35
9	31.64	25.40	6.24
10	31.56	25.40	6.16

Std Dev	0.13	N/A	0.13
Median	31.60	25.40	6.20
Average	31.59	25.40	6.19

Table 3 – Heat Seal Film + Adhesive Thickness Measurement

micron	OPET Film/Adhesive Thickness	OPET Film Thickness	Calculated Adhesive Thickness
1	17.75	14.69	3.06
2	18.27	13.90	4.37
3	18.09	13.19	4.90
4	18.35	14.16	4.19
5	18.30	14.29	4.01
6	18.06	14.56	3.50
7	18.21	13.39	4.82
8	17.77	13.85	3.92
9	17.60	13.94	3.66
10	17.65	13.79	3.86

Std Dev	0.29	0.47	0.57
Median	18.08	13.92	3.97
Ave	18.01	13.98	4.03

Table 4 - OPET film + adhesive thickness measurements

	OPET Film		
Measure Method	OPTIGAUGE	Micrometer	Print Specification
Thickness	13.98 microns	12.7 microns	12. microns

Table 5 - OPET Film Without Adhesive Thickness Measurements

Heat Seal Film/Adhesive Thickness	OPET Film/Adhesive Thickness	Aluminum Foil Print Thickness	Lid stock Thickness (calculated)	Lid stock Thickness (micrometer)	Lid stock Thickness (print)
31.59 microns	18.01 microns	76.2 microns	125.8 microns	126.5 microns	121.9 microns

Table 6 - Total Lid stock Material Thickness Measurements