

# Advanced Technologies to Detect and Minimize Multilayer Gauge Variation

Has Extrusion Technology Kept up  
with Gauging Techniques?

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## **Introduction**

This paper will include a quick review of the fundamental differences between on-line and off-line gauge measurement systems, including sampling methods. A more detailed comparison of off-line gauging technologies will be completed, focusing on the technologies used to examine samples reported in five case studies. The case studies will include examples of how data from high resolution gauge profiling equipment can be used to identify the root causes for the gauge variation. A copy of the slides shown during the presentation is attached to the end of this paper.

Thickness gauging technology can be broken down into two major categories: On-line thickness gauges and off-line or bench top laboratory thickness gauges.

## **On-line Thickness Gauging Characteristics**

On-line gauges provide immediate feedback that can be used for closed loop control, often referred to as automatic gauge control, when installed on extrusion lines. Full scan update time depends on nip or die oscillation speed. As a general rule, use system with faster response time and close to die if the process is dominated by short product runs or frequent product changeovers. Positioning the web sensor downstream is more suitable to optimize long steady runs where stability is goal.

Sensor technology limits capabilities in many cases. Capacitance sensors cannot measure EVOH or nylon because the dielectric constant of these polymers changes too much within the typical temperature range seen in film or sheet extrusion. Gamma backscatter and beta source sensors are not affected by temperature, but regulatory requirements make them difficult to maintain.

Single sided sensors such as capacitance and gamma backscatter can measure unsupported film, but bubble or web flutter often requires aggressive filtering. The long time constants used to filter the data make automatic gauging systems take longer to stabilize. Sensor heads that touch the bubble risk damaging the film.

Beta transmission sensors can only be used on a flat web because it is a see through sensor. Gamma backscatter and capacitance sensors are frequently placed downstream as well to avoid noise from bubble flutter. Although transmission sensors are more tolerant of web flutter which allows shorter time constants to be used, the response time is longer because they are positioned further away from the die than single sided sensors.

Where closed loop is used, be sure there is procedure to check calibration. All measuring devices suffer some drift over time; better units employ a compensating circuit. If not, set up a manual check system.

## **On-line Sampling Methods**

On line systems travel around the bubble when measuring tubing, or back and forth when measuring layflat tubing or sheet. Refer to slide 4 for details. The measurement path indicated by the solid and dashed line is a combination of transverse and machine direction measurements. The speed at which the sensor head travels depends on the required resolution, line speed and width of the sample to be measured.

Gamma backscatter, infrared and capacitance sensors can be used on blown film lines. Sensors that must transmit through the film such as beta sources, some capacitance, infrared and other optical transmission systems require both an emitter and sensor on opposite sides of the web. It is positioned near the edge, like in the machine direction technique shown in the previous slide. Blown film systems often oscillate the die or nip to randomize gauge variation. This movement will allow transmission sensor systems to estimate the overall gauge variation as long as the oscillation speed, layflat width and line speed are known.

The big drawback of on-line gauging systems is that the sensor must be large enough to capture sufficient data and the film distance from the sensor is not always stable. Significant filtering is required to supply data that can be useful, resulting in diminished resolution when compared to equivalent off-line systems.

## **Off-line Sampling Methods**

Both discrete point and continuous measurement off-line systems follow straight lines when measuring film thickness. Transverse direction measurement of layflat tubing follows a horizontal path around the circumference of the bubble, as indicated by the red lines on the left illustration. Usually only one path is selected. If more than one path is selected, the distance should be at least 10 feet or 2.5 meters apart. Distance between measuring points is usually not less than 1 inch or 2.5 cm for discrete systems.

Measuring several paths around the bubble is a time consuming way to determine to measure machine direction variation. A more common technique is to select a specific point on the circumference of the bubble and measure about 20 feet or 5 meters of film, as shown in the illustration on the right. Distance between points is usually not less than 6 inches or 15 centimeters for discrete systems. The same methodology is used when measuring cast film.

The major advantage of off-line systems is the potential for very precise resolution if high-end systems are employed. The major disadvantage is that the data cannot be used in a feedback loop to minimize gauge variation using automatic gauge control technology.

## **Elements of Thickness Variation**

The one sample illustration at the top of slide 6 appears to be within the acceptable control limits for this product. UCL refers to the upper control limit and LCL refers to the lower control limit.

Although the sample meets the gauge range specifications, the measurements indicate that “there are a whole lot of things going on”. Some fixed contributors can be identified if the resolution is sufficiently precise.

All automatic gauge control systems require the collection of multiple overlaid samples, as is shown in the middle illustration. The amount of data collected makes it difficult to distinguish fixed from random contributors. It is interesting to note that although a single sample may have been within the acceptable range tolerance, this may not be the case when several samples are measured.

Real time data collected from on-line sensors can be filtered to distinguish fixed from random contributors. An example is illustrated at the bottom, where the peak to peak and band width are used to generate a filtered, smoother curve. This trend curve can be used to modify the fixed contributors, and ultimately minimize the gauge variation. No automatic gauge control system will be able to eliminate the random contributors.

## **Off-line Thickness Gauge Characteristics**

Off-Line gauges measure static film samples either at the production line or in a QC lab. Off-Line gauges have no time constant averaging so they detect wider variation than on-line systems if the measuring device has sufficient resolution.

The simplest method is to weigh a known surface area of film and use an estimate of average film density to calculate the average thickness. This technique cannot be used to measure variation in film gauge.

Mechanical thickness gauges require that film be separated into individual sheets to obtain meaningful results. Almost every film factory uses these devices. They can be influenced by film irregularities such as gels, wrinkles, surface roughness or dust.

The digital readout Metutoyo displayed on the bottom left of slide 7 is a venerable industry standard that has a display resolution of  $\pm 0.05$  mils or 1.3 microns. It uses a light source to count the number of hatch marks etched into a glass cylinder as it moves up and down. It can be connected to firmware to capture data and calculate simple statistical information. These mechanisms are delicate and prone to bending of the piston.

A more robust system is shown in the middle picture on slide 7. It uses a linear variable displacement transducer (LVDT) technology that has a resolution of  $\pm 0.01$  mils or 0.02 microns.

One problem with both these units is that the film is repositioned manually for each reading. Slight changes in distance can have a significant effect on overall results, particularly if there are multiple thick and thin bands in the film sample.

The digital profiler with firmware shown on the right picture on slide 7 removes this problem by moving the sample strip to the left in equal distances each time a measurement is completed. The base system includes firmware for basic statistical reporting. It can also be connected to custom software that provides graphical displays to help when diagnosing gauge variation problems. Accurate displacement is critical when searching for fixed cycle disturbances.

Absorption gauges are indirect methods that utilize infrared or radiation sources to measure the percentage of the beam that did not pass through the film sample. Not all formulations are suitable for this type of technology.

Capacitance gauges are an indirect thickness measurement and are sensitive to density changes and should be calibrated regularly. A certain amount of filtering should be added to handle web wrinkles and creases. Almost any formulation that does not contain metals or voids such as foamed sheet or pearlized film, can be measured with this technology.

The capacitance profiler shown on slide 9 is model that used to measure gauge variation in examples 1 to 3 that will be reviewed later in this paper. It utilizes a patented AutoCal™ system to calibrate the capacitance sensor. A mechanical micrometer measures the film thickness and positions the film precisely to calibrate the capacitance sensor.

### **Off-line Layer Thickness Measurement Techniques**

Measuring layer thickness in a co-extrusion or laminated structure is time consuming and difficult to measure accurately. Cutting thin samples with microtomes and placing them under microscopes is a common technique. Refer to slide 11 for an example.

Less well known is the Michelson interferometer which was used to measure layer thicknesses in the in examples 4 and 5 described later in this paper. Michelson interferometer technology is based on the principle of projecting a narrow beam of white light through a sample, detecting the reflection from the boundary between layers and comparing it to a reference mirror. Refer to slide 12 for more details.

This equipment must be able to see white light to work, so the film must be transparent or lightly colored. It can accommodate film samples between 2 microns and 2 mm (0.08 to 80 mils) thick. The software can handle up to 16 layers simultaneously. Adjacent materials must have different indexes of refraction to detect the boundary between layers.

The display screen from the Davinor brand of Michelson interferometer used to examine samples in examples 4 and 5 is shown in slide 13. The graph to the left shows the raw data. The table to the right displays the results of calculations that are used to determine the thickness of individual layers.

The graph displays 9 peaks for this 9 layer sample. The table shows the structure and layer thickness. The peaks between 0 and 1 on the left refer to layer 1, which happens to be a 6 micron thick layer of LDPE. It is displayed at the bottom of the table.

The layer structure of this sample is 6.0 microns of LDPE, 5.2 microns of HDPE, 2.2 microns of a tie layer, 1.7 microns of Nylon, 1.0 microns of EVOH, 1.5 microns of Nylon, 1.8 microns of a tie layer, 5.3 microns of HDPE, 5.3 microns of LDPE for a total film thickness of 30 microns.

Some interesting things to note about this test result are that there is no significant noise between layers to make the peaks difficult to identify. The sample was measured manually and it does take some care and skill to obtain this level of resolution.

Note that this equipment has sufficient resolution to detect layers as thin as 1 micron (0.04 mils).

### **Example 1 – Total TD Variation Below the Frost Line**

Transverse direction gauge variation was profiled for a blown film sample exhibiting thin and thick regions on opposite sides of the bubble. Refer to the linear plot on slide 15 for details.

This problem can be detected with several on-line or off-line thickness gauges. Continuous or discreet (point-to-point) sensors can be used as long as point spacing is relatively close if using point-to-point. When high resolution is required, discrete sensors take measurements as close as 1 inch apart. The fine-line capacitive sensor was capable of continuous cross-web measurement at high resolution to detect detail. Resolution with this unit was 0.001 mil (0.025 microns) at 50 data points per second. High spatial resolution, the ability to detect step changes with high resolution, was possible due to narrow sensor configuration.

The polar plot clearly displayed on slide 16 shows the magnitude of the problem. It would be readily visible to the operator. The target gauge for this blown film product was  $10 \pm 1$  mil ( $254 \pm 25$  microns). Gauge varied from a maximum of 14.6 mils (371 microns) to a minimum of 6.38 mils, (162 microns) which is unusually large for most film applications. Refer to slide 17 for details. An easier way to understand the variation is to compare the standard deviation and minimum to maximum range to the average thickness of the sample. The standard deviation was a reasonable 9.757%, but the minimum to maximum range was 61.875%, or approximately  $\pm 30\%$  of the average.

The Fourier series analysis for this sample exhibited 3 clusters of peaks, indicating that three fixed disturbances contributed to the majority of gauge variation measured in this sample. Refer to slide 23 for a summary. A cluster of peaks that is noticeably higher than neighbouring ones indicate a unusually strong impact on gauge variation.

The strongest set of peaks is 1 and 2. This is clearly visible from the polar plot, indicating the frost line is higher on one side and lower on the other side of the bubble. This pattern can be the result of a misaligned air ring. Just a small misalignment can produce the pattern observed in this sample. Check that the air ring is seated properly on top of the die by using the distance between the die gap and the air ring lip set as a reference. In this case, this was not the problem. Refer to slide 19 for details.

A similar pattern can be produced the air ring is tilted slightly. If the die oscillates or rotates, place a level indicator on the top of the air ring chamber and note the position of the air bubble. The air bubble will shift to the other side when the die turns 180° if the air ring or die and air ring are not level. In this case, this was not the problem either. Refer to slide 20 for details.

Dropping a plumb bob from the center on the primary nips to the top of the IBC stack clearly indicated that the stack was not centered to the collapsing frame. This will cause air currents to be faster on one side of the bubble, resulting in a tilted frost line. We had already ruled out a tilted die by placing a level on top of the air ring and observing that the air bubble did not shift significantly as the die rotated. Further examination was required to determine if the stack misalignment was caused by a bent stack or incorrect die position. Refer to slide 21 for details.

Upon removing the IBC stack and dropping a plumb bob closer to the die, we can observe that the die was not centered below the nips. The extruder and die were at operating temperature, so the effect of thermal expansion had already been taken into account. Since the collapsing frame should be symmetrical, it will push the bubble to one side, resulting in more effective cooling and lower frost line on the side closest to the air ring. This was clearly the main causes for peaks 1 and 2. Refer to slide 22 for details.

The second set of higher peaks was 7 to 9 times around the bubble. Since this air ring had 8 ports, there was reason to believe that the air pressure distribution within the air ring chamber is uneven. This was the cause of the 7 to 9 peak cluster. Cluster of peaks more than 16 times around the bubble may be too small to be significant. Refer to slide 23 for details.

There was a smaller cluster of peaks between 13 to 15 times around the bubble. The most likely causes for this were dirt in the air ring or port line effects caused by excessive melt temperature variation entering the die block. Dirt inside the air ring is difficult to detect.



A common cause is spraying silicon oil onto the air ring lip set to avoid tearing the bubble if it rubs against the lip set. Some of the oil can run down into the air ring lip gap. The high velocity air will freeze oil into a sticky residue that interferes with air flow through the lip. Blockages will cause thin spots around the bubble. A better approach is to spray the oil onto a rag and then wipe the rag onto the air ring lip set surfaces. A large amount of dirt was visible upon disassembling the air ring lip set. This was the cause of the 13 to 15 peak cluster.

### **Example 2 – Total TD Variation Above the Frost Line**

The same style of thickness gauge as used in Example 1 was used to measure this sample as well. The bubble was thicker on one side than the other. Other patterns were not readily apparent by looking at the linear or polar plots. Although the average gauge was within the specification, the range was double the acceptable standard. Refer to slide 25 for details.

The Fourier series analysis is displayed on slide 26. The large influence from columns 1 and 2 are due to the asymmetry of the bubble that is easily visible on both the linear and polar plots. The cluster around 4 and 5 requires more careful examination.

All the tests done for example revealed nothing unusual in this case. What was most revealing is that although the bubble appeared to be reasonably symmetrical below the cage, near the frost line, the bubble was rubbing against one side of the cage by the time it exited the top set of rollers. Examination of the collapsing frame revealed that that one panel was closer to the die centerline than the other. The slightly distorted bubble was rubbing against the both side stabilizers and one side of the collapsing frame. Columns 4 and 5 reflect the effect of the side stabilizers.

Alignment of collapsing frames can be difficult to measure, but they are critical to the overall gauge uniformity of the bubble. Some stretching will occur, but the film will usually recover as long as the stretching force is below the elastic modulus of the film. Refer to slide 27 for details.

The position of the bubble at the top of the cage can be caused by a tilted frost line or misalignments in the tower. If the contact points move with an oscillating nip, then check the alignment of collapsing surfaces. If it moves with the die, then checks for problems below the frost line. Refer to slide 28 for details.

Misalignments in the collapsing frame can happen at the top as well as the bottom. Check the alignment of the pivot points. The photograph shows how operators were unaware that one side of the collapsing frame was not even secured to the primary nip frame. Refer to slide 29 for details.

### **Example 3 – Total MD Variation When Extrusion Coating**

The same type of thickness gauge as selected for Examples 1 and 2 was used in this case. A high frequency cycle variation imbedded on a low frequency cycle variation was detected when profiling machine direction gauge variation from an extrusion coating line

The additional benefit of this type of thickness gauge is that the higher-end software capability included Fourier analysis can be used to determine the low frequency cycle as well as the embedded high frequency cycle. In an example like this, analyzing data visually would be difficult. Refer to slide 30 for details.

The strong cycles are on the odd columns is due to the length of the sample. It could just as easily been even numbers if the sample was just little shorter or longer. Although the client did not report the true root cause of these cycles, the larger low frequency cycles were probably due to draw resonance and the faster cycles were probably due to a misaligned bearing in the chill roll stack. Refer to slide 31 for details.

### **Example 4 – Measuring Layer Thickness in 7 Layer Film**

Refer to slide 32 to view the 7 layer blown film sample. The structure is symmetrical, containing an outer jacket, tie layer, copolymer layer, Saran barrier layer, copolymer layer, tie layer and inner jacket. The film has a nominal gauge of 3.25 mils or 83 microns.

Lines are clearly visible in the machine direction after staining. The lighter bands are regions where it was suspected that the Saran barrier layer was missing. The position of these bands relative to the die lips did not change as the film was extruded.

The stained region of the sample was examined first using the Davinor brand Michelson interferometer. Refer to slide 33 for details. Seven layers were detected, as is shown by the graph that revealed 7 peaks of interest. Each peak represents the relative intensity of light reflection of an interface between layers. Only the higher and more distinct peaks are significant. The distance between the peaks is the optical distance between layer interfaces and must be corrected by using the refractive index of that layer to calculate the actual layer thickness.

With sufficient adjustment to the resolution of this instrument, it is possible to measure the individual layers as precisely as with a microscope, but it is much quicker. In this sample, the distance between peaks 0-1 can be correlated to the thickness of the outside jacket. Similarly, peaks 1 to 2 refer to the outer tie layer. Peaks 2 to 3 refer to the outer copolymer layer. Peaks 3 to 4 refer to the Saran barrier layer. Peaks 4 to 5 refer to the inner copolymer layer. Peaks 5 to 6 refer to the inner tie layer and peaks 6 to 7 refer to the inner jacket layer.

### **Example 5 – Confirming Missing Layer in 7 Layer Film**

Refer to slide 34 for details. The light reflection curve in the drop out regions will be different than where Saran is present. This graph has been adjusted to focus on the inner part of the film structure.

The peaks of interest are numbered inside the red box. There is no Saran present in this sample, so there are only 5 peaks of interest. The distance between peaks 0 and 2 represents the outer jacket and tie layers combined and peaks 2 to 3 represent the outer copolymer layer. Peaks 3 to 4 represent the inner copolymer layer and Peaks 4 to 5 represent the inner tie and jacket layers combined.

With sufficient adjustment to the resolution of this instrument, it is possible to measure the individual layers as precisely as with a microscope, but it is much quicker. Although permeation testing may detect the loss of Saran in portions of the film structure, it cannot distinguish the variations in thickness that identifies patterns. Patterns are used to diagnose the cause of the problem.

The target thickness was 3.25 mils (82.6 microns). Actual gauge range was 3.19 to 3.41 mils (81.0 to 86.6 microns) in the stained region. Where the Saran barrier layer was missing, total thickness range was only 2.81 to 3.06 mils (71.4 to 77.7 microns). The explanation of the root cause for this problem is illustrated on slide 35. This illustration demonstrates how layers are merged together in typical co-extrusion dies. Layers are joined together in stages to minimize the risk of interfacial instability as shown in the illustration on the left.

This gauge reduction drop out bands where the Saran layer is missing is more than would be predicted if Saran layer was just removed. The most likely cause is build-up of burnt Saran in these regions restricting the flow of the layers that were merged together before reaching the Saran merge point. This is shown in the illustration the right.

The same unit can run in profiling mode and the 0.2 mm diameter light beam can be programmed to take measurements as close as 1 millimetre apart. It takes time to collect the quantity of data to record an accurate profile for a typical film sample, but there really is no faster alternative to collect information with 1 micron thick resolution. Refer to slide 36 for an example of the profiling graphics displayed by the Davinor brand Michelson interferometer in.

## Conclusions

1. Gauging technology is not far behind extrusion technology.
2. It is currently not possible to measure individual layer thickness at 8 microns or less with an in-line thickness gauge with high resolution in an A-B-C-B-A structure.
3. When measuring 1 micron or fewer thicknesses, or high spatial resolution cases, manual (point-to-point) operation on an off-line profiling Interferometric gauge is effective.
4. Off-line interferometric profilers provide much faster measurements than microscopic analysis.

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**Presentation Slides**