The Impact of Foil Pinholes and Flex Cracks on the Moisture and Oxygen Barrier of Flexible Packaging

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Abstract

High barrier packaging applications for medical, industrial and food packaging often include aluminum foil. Thinner gauges of aluminum foil contain pinholes through which moisture and oxygen from the environment can enter the package accelerating the degradation of the contents. This paper addresses the impact of foil pinholes and fractures on the oxygen and moisture barrier of multilayer flexible packaging, in theory and in practice using laser-produced pinholes. The analysis shows that the transmission through the pinholes is less that occurring through exposed sealant edges and much less than that which would take place through non-foil packaging of the same size.

Background

While metal and glass containers have a long history of high barrier protection of food and other products, the cost of metal containers and the fragility of glass containers make them less attractive than flexible packaging containing aluminum foil. The aluminum foil is usually the most expensive component of the flexible package and as such the economics drive the use of thinner gauges. Practical considerations of process capability to handle thin foils without excessive web breaks, wrinkles and other factors limit how thin a foil can be produced and converted into packaging. Aluminum foil is produced by one of several processes that include a rolling process where the thickness of one or two sheets is sequentially thinned under compression between rolls. The number and size of pinholes in a thin foil will depend on the filtration of inorganic materials from the molten aluminum, the casting process and the rolling conditions such as lubricant type and lubricant filtration[1]. The relationship between foil caliper and allowable number of pinholes can be seen in Table 1[2].

TABLE 1
Maximum Allowable Pinhole Count in One Square Meter

Foil Gauge	Foil Caliper, μ	Average	Maximum
28.5	7	423	1584
35	9	211	1056
50	13	85	528
75	18	21	106
100	25	0	0

Pinholes as Mask Windows
When a gas or moisture transmission rate measurement is needed of a very permeable film, a mask is used over the sample reducing the test area[3]. For example, users of transmission rate equipment with a 50 cm^2 sample area can use a mask covering all but 5 cm^2 of the sample to reduce the transmission flux to 1/10 the normal value. A foil pinhole may be considered a special case of a foil mask where the pinhole is considered the opening in a mask. When polymer layers cover the pinhole, one can compute the amount of moisture and oxygen coming through the pinhole, knowing the area of the foil pinhole and the transmission properties of the layers covering the pinhole. In the case of a PET film and LDPE extrusion laminate, Fick's law would govern this transmission as barriers for moisture and oxygen. The combined resistances (reciprocal of transmission) would be summed as referenced in the literature[4]

where: 1 / Combined Transmission Rate = Σ (layer thickness (i)/ layer transmission rate (i))

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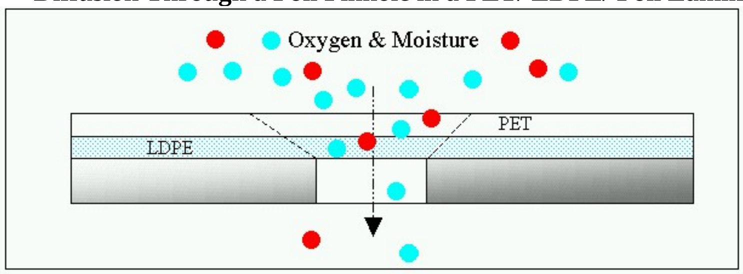
Math models were developed using this “masking” concept to quantify the impact of foil pinholes in packages for products sensitive to moisture and/or oxygen. The question is often asked; “Should the area be adjusted upward to recognize that moisture and oxygen diffuses to the pinhole from a larger area?” (See Figure 1)

FIGURE 1

Diffusion Through a Foil Pinhole in a PET/ LDPE/ Foil Lamination



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Computing Pinhole Transmission in Flexible Packaging vs. Non-Foil Films with Uniform Permeability

Because pinhole defects are non-uniformly distributed over the foil, the usual normalization to a rate per 1 m^2 or 100 in^2 need not be done. A typical test instrument having a 50 cm^2 test area (1/200 of a square meter) automatically applies a factor of 200 to the amount of moisture or oxygen being detected through the cell to compute the amount of moisture or oxygen coming through one square meter of non-foil packaging. When testing a whole container or a foil structure with a pinhole(s) that factor must be removed. In newer instruments, a software switch (flat film or package) is selected.

As an example of how a test is done for a film with a pinhole, consider the structure in Figure 1. The PET has a transmission rate of 76 cc • mils/m^2 · day for oxygen and 40 g · mils /m^2 · day for moisture while our generic LDPE has a transmission rate of 4000 cc · mils/m^2 · day for oxygen and 20 g · mils /m^2 · day for moisture. If these polymer films were laminated to aluminum foil with a single pinhole, we should be able to compute the impact of the pinhole. Assuming a pinhole diameter of 82μ, a value used in our tests and calculations, the computed transmission rate would be only 8 x 10^{-7} cc/ day (0.0008 μl/ day) and 1 x 10^{-7} g/ day (0.11 μg/ day) as shown in Table II. These low values would exceed the ability of our steady state test equipment to measure.

TABLE II

Transmission Through Film and Laminated Pinhole

Material	Standard WVTR	Standard O2TR	WVTR For 12μ Thickness	O2TR for 12μ Thickness
PET	40 g/ m^2 · day	76 cc/ m^2 · day	85 g/ m^2 · day	161 cc/ m^2 · day
LDPE	20 g/ m^2 · day	4000 cc/ m^2 · day	42 g/ m^2 · day	8470 cc/ m^2 · day
Combined			28 g/ m^2 · day	158 cc/ m^2 · day
The area for a 82μ diameter pinhole is 5.28 x 10^3 $μ^2$, 5.28 x 10^{-3} mm^2 or 5.28 x 10^{-9} m^2			0.15 μg/ day	0.00083 μl/ day

Experimental

In preparation for doing actual measurements on aluminum foil, pinholes were produced in foil using a laser in clusters of 1, 2, 4, 8, 16, 36 and 64 all within the 50 sq cm test area used by our oxygen and moisture test equipment. The size of the pinholes were characterized by microscopy and found to average 82μ in diameter. For sake of comparison, the theoretical pinholes discussed in this paper have been made that size. Based on conversations with a technical expert from a foil producer[5], these are large pinholes and is more typical of foil damage done while a converter handles the foil. Even so the pinholes require back lighting to be found.

Sheets of aluminum foil with pinholes were laminated in three steps.

1. 12μ PET/ Adhesive/ 38μ Al Foil
2. 12μ PET/ Adhesive/ 38μ Al Foil/ 12μ Acid Copolymer

3. 12µ PET/ Adhesive/ 38µ Al Foil/ 12µ Acid Copolymer/ 12µ LDPE

The aluminum sheets with pinholes were taped to the aluminum foil web moving through the adhesive laminator in the Step 1. These samples were cut out and turned over for the extrusion-coating step. Step 2 was prepared by temporarily turning off the LDPE extruder.

The laminated samples were removed from the web and tested on transmission rate equipment for moisture and oxygen. Tests were conducted in-house using older test equipment[6] as well as by newer, higher sensitivity test equipment[7]. The instrument manufacturer, knowing we were testing low transmitting samples, proposed the times used to reach to equilibrium:

Oxygen Testing

Oxygen Purge Zeros (nitrogen on both sides of sample)	100-150 Hours
Read Transmission Rates (Oxygen on one side of sample)	100-300 Hours
Water Vapor Testing	
Shim Stock in sample holder to measure system zero:	48 Hours
Testing of conditioned samples:	100-120 Hours

The results shown below in Tables III and IV show the transmission rates per pinhole. The rates are expressed in terms of microliters (µl) of Oxygen and micrograms (µg) per day per pinhole for moisture. These terms are 1 thousandth and 1 millionth of the commonly reported values respectively. To compute results for the numbers of pinholes allowed in a square meter, one would have to multiply by the counts in Table I.

TABLE III
Moisture Transmission Rate per Pinhole
(µg/day at 38°C, 90% RH)

Pinhole Count	Step 1	Step 2	Step 3
<8	No transmission detected		
8	18		
16	13	1	
36	Avg = 11, n=3, s = 3	5	<1
64	Avg = 23, n = 3, s = 1	2	1
Theoretical	<1	<1	<1

Note: 100 µg/day = 0.0001 g/day

TABLE IV
Oxygen Transmission Rate per Pinhole
(µL/day at 23°C, 1 Atmosphere)

Pinhole Count	Step 1	Step 2	Step 3
<8	No transmission detected		
8	Avg = 2, n = 4, s = 2		
16	Avg = 7, n = 3, s = 6	21	
36	Avg = 14, n = 5, s = 5	15	10
64	Avg = 28, n = 5, s = 4	19	4
Theoretical	<1	<1	<1

Note: 100 µl/day = 0.1 cc/day

One can see that higher transmission rates were found than expected. The two participating labs were showing a high level of variability from specimen to specimen. When it came to low pinhole counts, the sensitivity of the instruments might explain the unusual results. There have been a few theories to explain transmission rates much greater than the theoretical values.

How the Delamination Around the Pinholes May Have Taken Place

Micrographs of the pinholes revealed that the laser had not vaporized the aluminum away from the point of focus, but had deposited the aluminum around the holes. These deposits resemble volcanoes as illustrated in Figure 2 and photographed in Figure 3. The adhesive coated polyester, being a somewhat stiff material, was held off the foil. This large area of delamination relative to the pinhole size delivered a larger amount of oxygen and moisture to the pinhole than would be have been delivered by the materials if they had been intimately bonded to the foil around the pinholes. Micrographs showing the delamination areas around the pinholes are shown in Figure 4.

FIGURE 2

Laser Pinhole In PET/ Adhesive/ Foil/ Acid Copolymer / LDPE Lamination

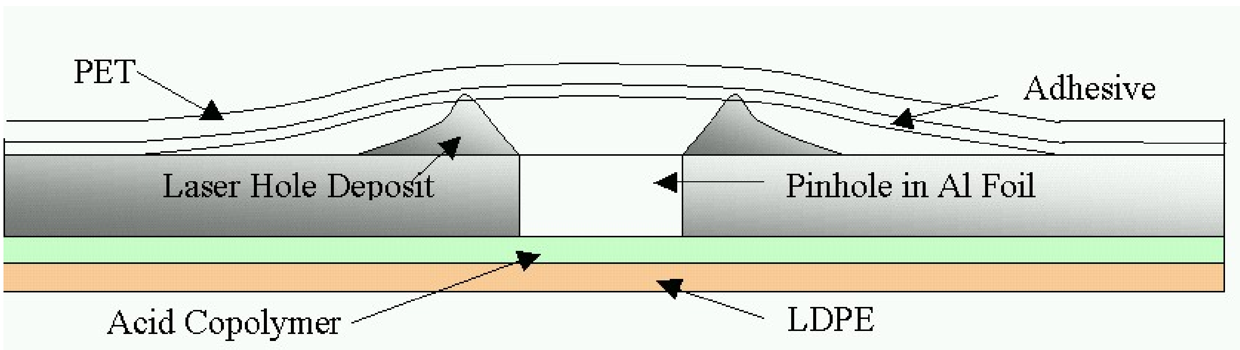
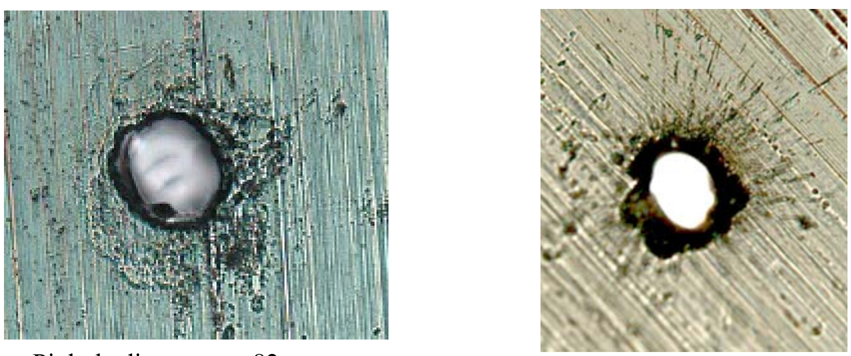


FIGURE 3

Laser Produced Foil Pinhole Showing Deposited Material on Backside of Hole

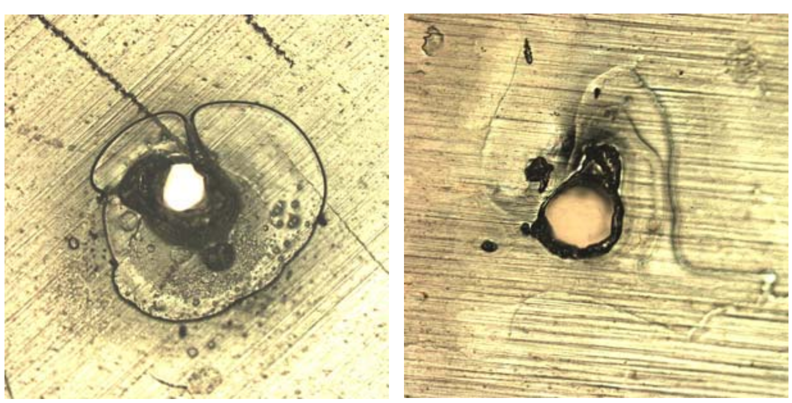


Pinhole diameters = 82 µm

FIGURE 4

Delamination Areas on Both Sides of Foil

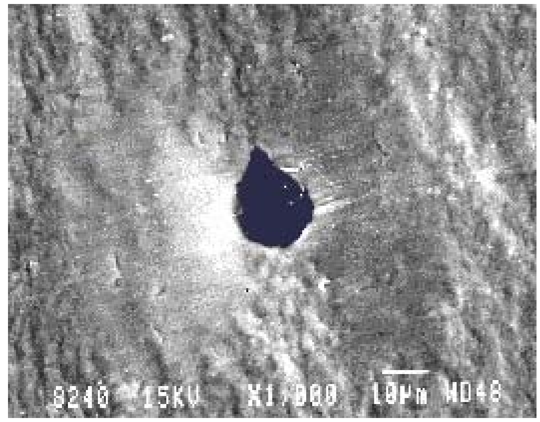
PET Side at 100X LDPE Side at 100X



By contrast, aluminum foil pinholes from natural sources are usually flat without protruding above the surface of the aluminum foil[8]. One exception is aluminum fine damage that can cause a puncture feature that can extend above the surface. SEM micrographs of pinholes in aluminum foil from normal causes are shown in Figure 5.

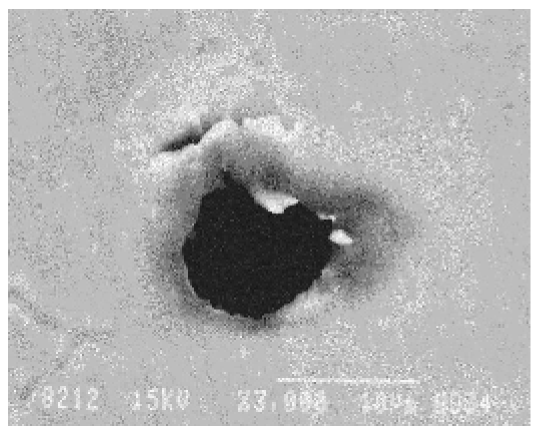
FIGURE 5
Examples of Typical Foil Pinholes

Pinholes from Al-Fe-Si Constituents



Pinhole from Depression in Foil

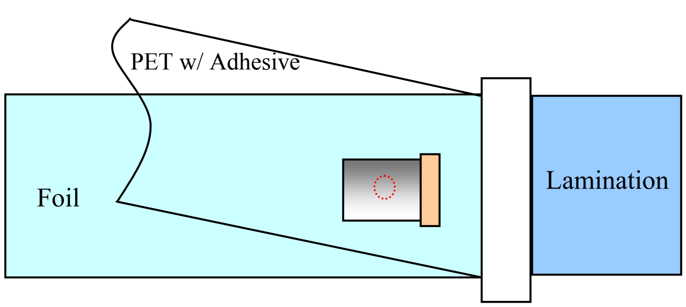
Pinhole from rolling Debris



Pinhole from Aluminum Fines Damage

In the first step of laminating polyester to the foil with adhesive, the web goes through a nip where two layers of foil are actually laminated. Of course, only the pinhole containing foil sheet is actually laminated to the PET. The air pressure under the sheet may be sufficient to hold the PET and adhesive off the foil around the pinhole.

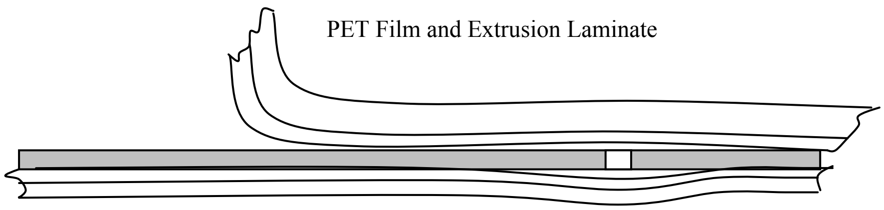
FIGURE 6
Lamination with Pinhole Containing Foil Piece Taped to Foil Web



In the Steps 2 and 3 of producing the test samples (the extrusion-lamination) the presence of delamination areas around the pinholes may result from the rapid expansion of trapped air in the pinhole and between the PET/LDPE and foil side when the sheet is hit with 316°C (600°F) resins during the extrusion coating operation.

FIGURE 7

Air Expanding Under Pinhole Causing Extrudate to Lift-Off Foil



Comparison of Transmission Through Pinholes and Transmission Through Sealant Edges

A practical comparison is needed to compare the transmission through pinholes and the transmission through the sealant edge of a package between the foils under more realistic conditions. For this case an assumption of intimate contact to the foil will be made and a 90% RH and 38°C condition for the moisture transmission. For oxygen transmission, air will be assumed since packages are never stored in elevated levels of oxygen. We will also assume that the package area inside the seals will be a one square decimeter package (100 mm x 100 mm). We will compute cases of one, and a worst case, 32 pinholes - the maximum allowed for 7µ foil. A pinhole diameter of 82µ will also be assumed (again a worst case for pinholes). The seal depth is computed at 6 mm. The previous example model will be expanded to include a sealant. The calculations were discussed in the Background Section.

TABLE V

Moisture and Oxygen Transmission through pinholes vs. Edge Transmission

PET/ 12µ LDPE/ Aluminum foil with Pinhole(s)/ 12µ EAA/ 25µ LDPE

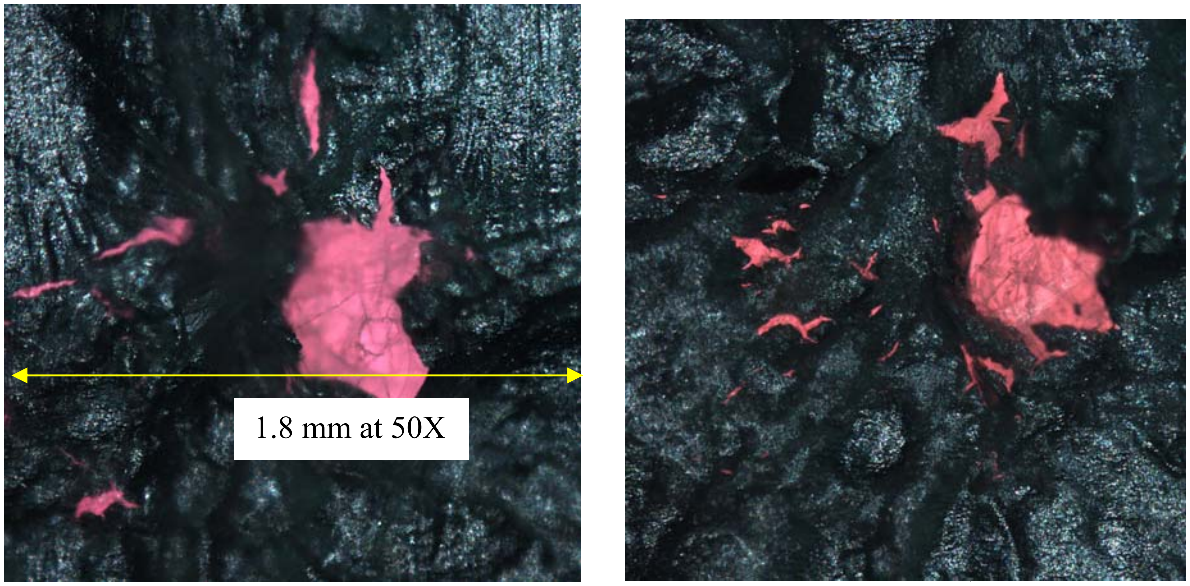
Source of transmission	Moisture at 90% RH and 38°C	21% Oxygen (air) at 23°C
Through 1 laminated Foil Pinhole	0.05µg/day	0.0002 µl/day
Through 32 laminated Foil Pinholes	1.6 µg/day	0.005 µl/day
Sealant Edge Transmission	2.5 µg/day	0.1 µl/day

Foil Fractures

A second application of the “mask” theory for computing the effect of disruptions in aluminum foil has been applied to foil fractures. Foil fractures can occur when a package is fabricated or when it is handled. In such cases the area of transmission increases dramatically and the possibility of poor performance increases. Figure 8 is a picture of foil fractures in a confectionary package resulting from the package fabrication (folding and sealing of the package around the product). The structure of the package is Paper/ 12µ LDPE/ Foil/ 12µ LDPE/ Cold Seal Adhesive.

FIGURE 8

Foil Fractures in Confection Package



Moisture Transmission Through Multiple Foil Fractures in a Flexible Package

Photo	No. (left to right)	Width, mm	Length, mm	sq mm	μg H_20/day	μl O_2/day
Left	1	0.06	0.23	0.014	0.30	0.012
Left	2	0.06	0.19	0.011	0.25	0.010
Left	3	0.09	0.27	0.024	0.53	0.020
Left	4	0.06	0.09	0.005	0.12	0.005
Left	5	0.06	0.28	0.017	0.37	0.014
Left	6	0.40	0.69	0.276	6.07	0.232
Right	7	0.06	0.14	0.008	0.18	0.007
Right	8	0.21	0.21	0.044	0.97	0.037
Right	9	0.08	0.21	0.017	0.37	0.014
Right	10	0.09	0.11	0.010	0.22	0.008
Right	11	0.06	0.07	0.004	0.09	0.004
Right	12	0.06	0.06	0.004	0.08	0.003
Right	13	0.17	0.26	0.044	0.97	0.037
Right	14	0.38	0.57	0.217	4.77	0.182
Right	15	0.14	0.21	0.029	0.65	0.025
			Totals	0.725	15.95	0.609

If this package had been constructed using non-foil packaging, the barrier properties could be easily computed knowing the complete construction and the internal package area. For purposes of comparison, the package construction will be computed as 12μ PET/ print/ adhesive/ 12μ met. PET/ Cold Seal Adhesive. We will assume a 1 square decimeter package area and transmission rates as follows:

WVTR at 90% RH, 38°C: 0.7 g/m^2 · day (700,000 μg/m^2 · day)

O2TR at 1 Atm. Oxygen and 23°C: 0.5 cc/m^2 · day (500 μl/m^2 · day)

O2TR at 21% Oxygen and 23°C: 0.11 cc/$m^2 \cdot$ day (110 µl/$m^2 \cdot$ day)

TABLE VII
Comparison of Foil Structure with Pinholes and a Non-Foil Structure

Mode of Ingress	Moisture µg/day	Oxygen µl/day (from air)
Transport through foil fractures in example	16	0.61
Transport through non-foil metallized package	6,900	5.0

Summary

Aluminum foil provides a valuable high barrier in flexible packaging constructions. While pinholes are expected in aluminum foil with thin gauges, the barrier properties are not significantly compromised relative to the transport through the sealant edge of a flexible package. Similarly, foil fracturing can occur during the handling or transportation of a package. Except in extreme cases where all the layers of the package have been fractured, the loss of barrier, while significant, is less than that which would occur when using a high barrier non-foil package.

An attempt was made to test the theory regarding pinholes using steady state transmission rate equipment for moisture and oxygen. At low pinhole counts, no transmission was detected using steady state transmission rate test equipment. At high pinhole counts, delamination around the pinholes caused each affected pinhole to have a greater effect than predicted based on the area of the pinhole.

End Notes and References

[1] Ward, Chris; Rhenalu – American National Can technical Exchange Meeting Note June 10, 1998
[2] From Alcan/Pechiney General Raw Material Specification for Aluminum Foil # 14000G
[3] ASTM F1249-90 (Reapproved 2001) "Water Vapor Transmission Rate Through Plastic Film and Sheeting Using a Modulated Infrared Sensor", Section X1.4 Testing Poor Barriers using foil masks.
[4] Polymer Permeability, ed. J. Comyn, Elsevier Applier Science Publishers, 1985 edition. P. 291
[5] Ball, Mel D., Novelis Global Technology Centre, Novelis Inc., personal communications
[6] Neenah Technical Center determined water vapor transmission rates using a MOCON Model W600 Permatran-W while Oxygen Transmission Rates were determined by a MOCON Model 1050 OxTran modified by Ray Dorschner for higher sensitivity.
[7] MOCON Model 2/20 OxTran and model 3/30 PermaTran test instruments were utilized.
[8] Ball, Mel D.; Internal presentation on aluminum foil quality Novelis Global Technology Centre, Novelis Inc.

2005 PLACE Conference September 27-29 Las Vegas, Nevada

The Impact of Foil Pinholes and Flex Cracks on the Moisture and Oxygen Barrier of Flexible Packaging

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Reasons for Using Thin Aluminum Foil in Flexible Packaging

- Cost Effective
- Not Fragile
- Barrier performance is better than non-foil packaging

Process capability limits how thin a foil can be produced and converted into packaging.

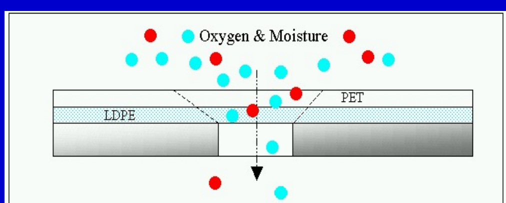
Maximum Pinhole Count for Aluminum Foil

Foil Gauge	Foil Caliper, µ	Average/ sq meter	Maximum/ sq meter
28.5	7	423	1584
35	9	211	1056
50	13	85	528
75	18	21	106
100	25	0	0

Causes of Pinholes

- Insoluble contaminates in aluminum matrix
- Filtration of particles from lubricants and oils
- Casting process
- Rolling conditions

Pinholes as Masks



- Pinholes are a special case of a mask.
- Fick's laws still apply but the sample area is reduced.
- Combined Transmission Rate

= 1/ Σ (layer thickness (i)/ layer transmission rate (i))

Masking Samples

Transmission through Pinhole

Material	Standard WVTR	Standard O2TR	WVTR For 12µ Thickness (each)	O2TR for 12µ Thickness (each)
PET	40 g · mil / m^2 · day	76cc · mil / m^2 · day	85 g/ m^2 · day	160 cc/ m^2 · day
LDPE	20 g · mil / m^2 · day	4000 cc · mil / m^2 · day	42 g/ m^2 · day	8500 cc/ m^2 · day
Combining Both Layers			28 g/ m^2 · day	160 cc/ m^2 · day
The area for a 82µ diameter pinhole is 5.3 x 10^3 $µ^2$, 5.3 x 10^{-3} mm^2 or 5.3 x 10^{-9} m^2			0.15 µg/ day	0.00083 µl/ day

Experimental Confirmation

- Sheets of aluminum foil with 1-64 pinholes were laminated in three steps.

1. 12µ PET/ Adhesive/ 38µ Al Foil*
2. 12µ PET/ Adhesive/ 38µ Al Foil*/ 12µ Acid Copolymer
3. 12µ PET/ Adhesive/ 38µ Al Foil*/ 12µ Acid Copolymer/ 12µ LDPE

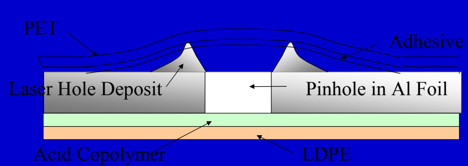
Moisture Transmission (µg / Pinhole)

Count	Step 1	Step 2	Step 3
<8	No transmission detected		
8	18		
16	13	1	
36	11	5	<1
64	23	2	1
Theoretical	<1	<1	<1

Oxygen Transmission (µl /pinhole)

Count	Step 1	Step 2	Step 3
<8	No transmission detected		
8	2		
16	7	21	
36	14	15	10
64	28	19	4
Theoretical	<1	<1	<1

Laser Hole Deposits



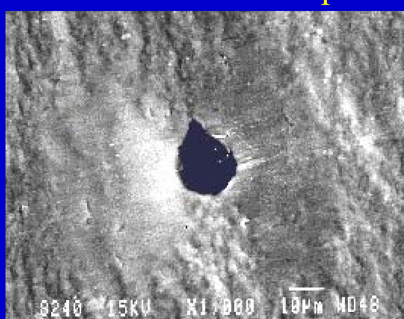
Pinholes Before and After Lamination

PET Side of Lamination

LDPE Side of Lamination

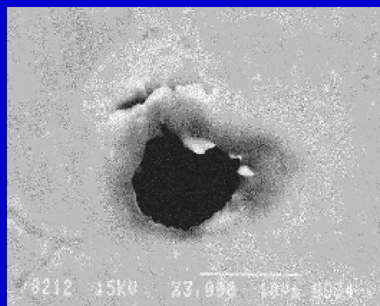
Pinholes from Al-Fe-Si Constituents

Pinhole from Thin Spots

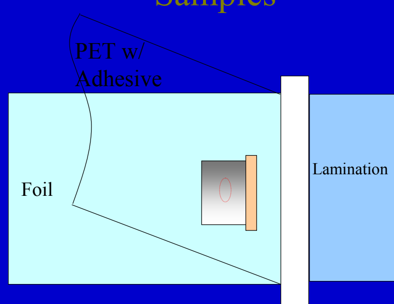


Pinhole from Rolling Debris

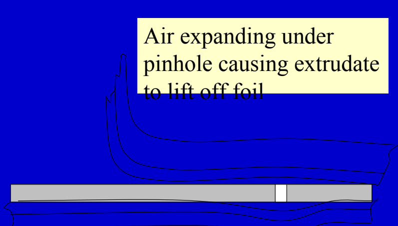
Damage from Aluminum Fines



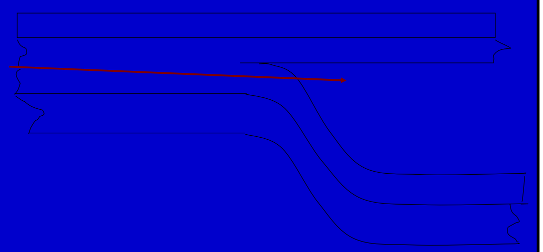
Laminating Laser Pinhole Samples



Extrusion Coating/Laminating



Transmission Through Sealed Edges



Transmission Modes Compared

- Package size = 100 x 100 mm
- Foil has 1 and 42 pinholes (max allowable)
- Pinhole size is 82µ in diameter
- Structure:

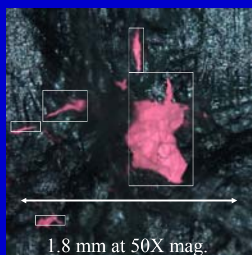
PET/ 12µ LDPE/ 7µ Aluminum foil with Pinhole(s)/ 12µ EAA/ 25µ LDPE

Transmission Modes Compared

100 x 100 mm pouch, 6 mm deep seals

Source of Transmission	WVTrans at 90% RH 38°C	O2Trans at 21% and 38°F
Through 1 Laminated PH	0.05 µg/day	0.0002 µl/day
Through 42 Laminated PH	1.6 µg/day	0.005 µl/day
Sealant Edge Transmission	2.5 µg/day	.1 µl/day

Application of Method to Fractured Foil Packages



- Foil Fractures are usually irregular
- Masking analogy can be applied knowing total area involved
- Polymeric layers must be intact

Summing Pinhole Areas and Permeation

W, mm	L, mm	sq mm	µg H_20/day	µl O_2/day
0.06	0.23	0.014	0.30	0.0012
0.06	0.19	0.011	0.25	0.0010
0.09	0.27	0.024	0.53	0.020
0.40	0.69	0.28	6.07	0.23
0.21	0.21	0.044	0.97	0.037
0.09	0.11	0.010	0.22	0.008
0.38	0.57	0.217	4.77	0.182
	Totals:	0.725	15.95	0.609

Not all lines from Table VI in Paper have been shown

Foil Package with Flex Cracks (0.72 $mm^{2)}$ vs. Non- Foil Pouch (1 dm^2) 0.7 g/m^2 day & 0.11 cc/m^2 day

Paper/ 24µ LDPE/ Foil/ 24µ LDPE/ Cold Seal Adh.
vs.
12µ PET/ print/ adhesive/ 12µ met. PET/ Cold Seal Adh.

Mode of Ingress	Moisture µg / day	Oxygen µl / day
Through Fractures	16	0.61
Through non-foil metallized Pkg.	6,900	5

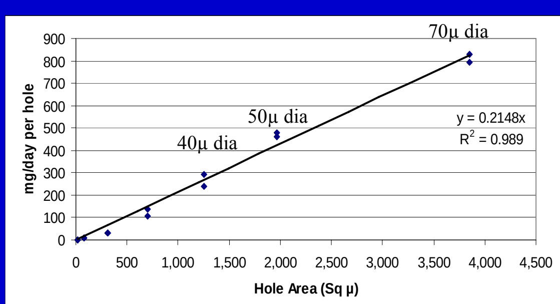
SUMMARY 1- Pinholes

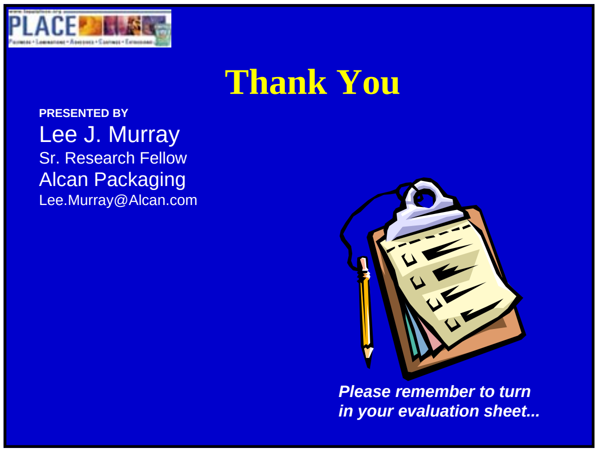
- Al foil - a valuable barrier for flex packaging applications
- Pinholes are expected in thin gauges
- Transmission through PH are often very low as part of a multilayer flexible packaging compared to edge transmission or non-foil packaging.

SUMMARY 2 - Foil Fractures

- Foil fractures happen during packaging and handling operations.
- Process limitations dictate how thin a foil can be used in a structure.
- Only in cases or when the whole structure is compromised is flex cracking worst than non-foil packaging.

Water Vapor Transmission through PH in Bare Foil – Mocon 3/31

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