Testing Methodology for VIPs

Avery Dennison Hanita

Table of Contents

Avery Dennison Hanita Testing Methodology for VIPs

Eddie Shufer, MSc Materials Science and Engineering, R&D, Avery Dennison Hanita

Abstract

The following methodology is used by Avery Dennison Hanita to assess the performance of Vacuum Insulation Panel (VIP) laminates, cores, and complete panels. Based on international film testing standards and integrating proprietary developments, the following test methods have been found by Avery Dennison Hanita to be the most effective and efficient ways to evaluate VIPs and their components.

Introduction

A Vacuum Insulation Panel (VIP), shown in Figure 1, is a composite unit comprising a core material (with an optional desiccant/getter) enclosed by an envelope that provides a barrier to the permeation of atmospheric gases. By evacuating the internal volume of the VIP, exceptionally high levels of thermal insulation can be reached.

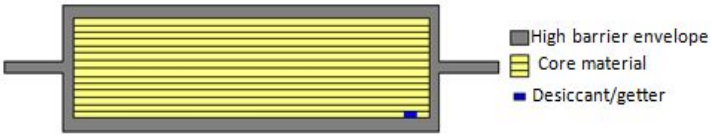

Figure 1: Structure of typical Vacuum Insulation Panel

How core material properties affect the long term performance of VIPs

Many organic and inorganic insulation materials with an open cell structure are available for use as VIP core material. When used in VIPs, the thermal conductivity of these materials rises as the internal pressure increases, [Ref. 1], as shown in Figure 2.

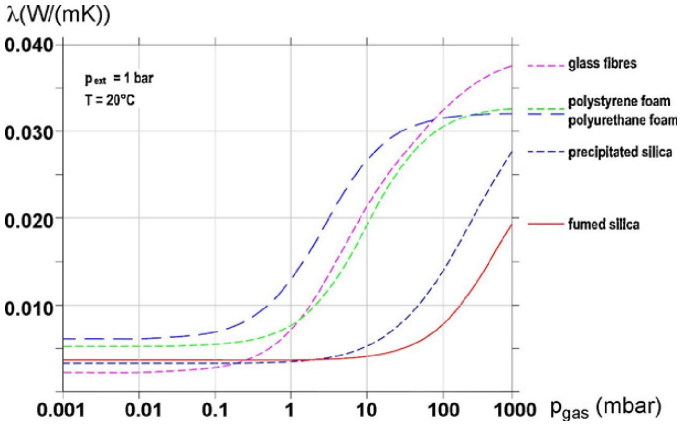

Figure 2: Thermal conductivity of several insulation materials as function of the internal pressure (redrawn from [Ref. 1])

The two most commonly used core materials are fiberglass and fumed silica. There are many types and suppliers of both core materials; each one has a different dependence of thermal conductivity as a function of internal pressure. The general theory of dependence of thermal conductivity on the internal pressure was published by U. Heinemann et al. [Ref. 1] and is described by Equation 1:

$$\lambda(P) = \lambda_0 + \frac{\lambda gas}{\left(1 + \frac{P_{1/2}}{P}\right)} + \frac{\lambda coupl}{\left(1 + \frac{P_{1/2} coupl}{P}\right)} \quad (1)$$

Where:

λ_0 is thermal conductivity at very low pressure such as $1 \times 10^{-2}\ mbar$, λ_0 is the sum of the two contributions of heat conducted by the solid porous core, and IR radiation.

λgas and $\lambda coupl$ are the thermal conductivity of air ($25.5\frac{mW}{m\cdot K}$) and thermal conductivity due to coupling effects between the skeleton of the core material and the gas molecules ($11\frac{mW}{m\cdot K}$) respectively.

$P_{1/2}$ and $P_{1/2}coupl$ (not relevant for fumed silica) are two specific pressure parameters that depend on the average pore size (d[μ], diameter) of the porous core material.

The relationship between the average pore size of core material in the value of its $P_{1/2}$ parameter is given by Equation 2:

$$d[\mu] = \frac{230}{P_{1/2}[mbar]} \quad (2)$$

According to Equation 1, any glass fiber core material can be fully characterized by the three specific parameters λ_0, $P_{1/2}$, and $P_{1/2}coupl$, while fumed silica core materials have only two relevant specific parameters: λ_0 and $P_{1/2}$. Since the longevity of the VIPs depends strongly on the specific properties of the core material used, Avery Dennison Hanita has recently developed a fast and easy method for its characterization, based on simultaneous measurement of internal pressure and thermal conductivity (Figure 3).

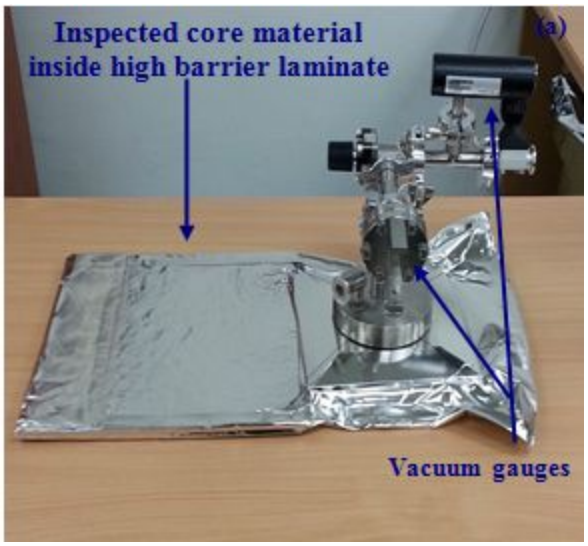

Figure 3(a): The λvs P system for core material experimental characterization

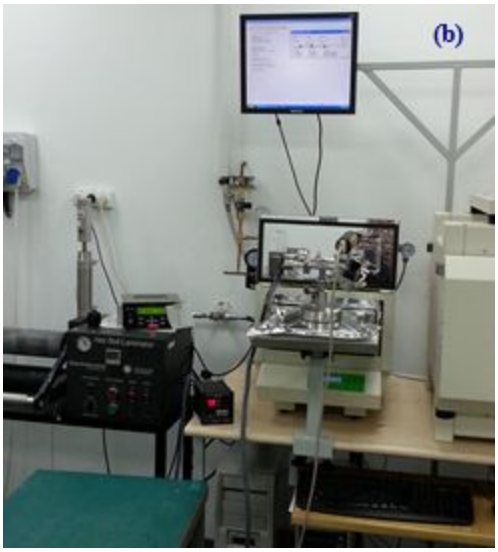

Figure 3(b): The λvs P system sample inside thermal conductivity measurement device (LaserComp FOX314)

At first, thermal conductivity is measured after evacuation of the system by a turbomolecular vacuum pump through a leak tight connector on the right side of Figure 3(a). Immediately after evacuation, the pressure inside the envelope is around 1×10^{-3} mbar, allowing the value of λ_0 for the inspected core material to be determined.

Later on, controlled amounts of gas are injected into the envelope through the leak tight connector. The thermal conductivity is measured after each air injection together with corresponding pressure measurement. Figure 4 shows the results of two tests made on two sheets of fiberglass taken from the same batch.

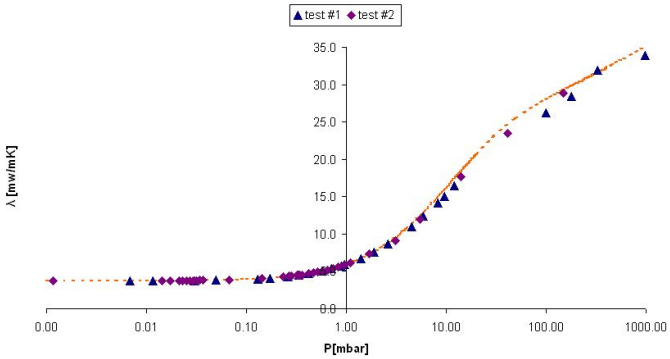

Figure 4: Thermal conductivity of two identical sheets of FG core as a function of internal pressure

The blue and purple dots represent the data points related to the two panels, while the orange line is the best fitted function of Equation 1. The best fitting parameters characterizing the tested FG core are shown in Table 1:

Table 1: FG Parameters

$\lambda_0[mW/mK]$	$\lambda gas[mW/mK]$	$P_{½}[mbar]$	$\lambda coupl[mW/mK]$	$P_{½_{coupl}}[mbar]$
3.7	25.5	10.5	11	800

As mentioned, the most important parameters for VIPs are λ_0, which determines the thermal conductivity at evacuated state (initial TC, lower the better), and $P_{½}$ (higher the better), which determines the degradation rate of the VIP due to permeation. As an example, the graph below shows the thermal conductivity as a function of the internal pressure of two types of fiberglass cores with different sets of characterizing parameters, as shown in Table 2.

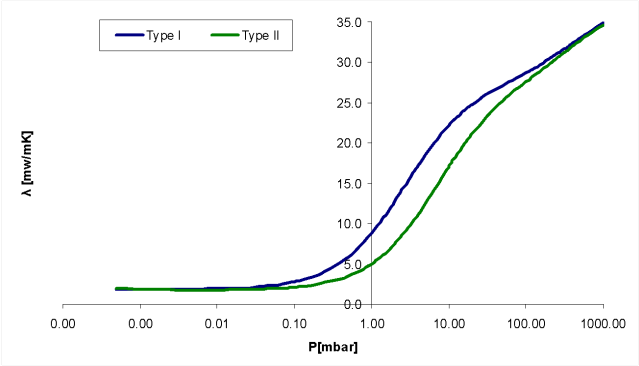

Figure 5: Thermal conductivity of two different glass fibers as a function of internal pressure

Table 2: FG Characterizing parameters

Fiber glass	λ_0 [mw/mK]	$P_{½}$ [mbar]	$P_{½}coupl$ [mbar]
Type I	1.85	2.7	450
Type II	2	7	450

The importance of $P_{½}$ can be demonstrated using this example of two identical VIPs (same envelope film and panel dimensions) but with different core material (Type I and Type II). For example, over time the internal pressure in both panels rose to 0.3 mbar due to air permeation. Although the internal pressure was identical in both panels, their thermal conductivity was very different: λ=4.4mW/mK for type I with P1/2=2.7mbar, and λ=2.8mW/mK for type II with P1/2=7mbar. This clearly shows the advantage of using cores with larger $P_{½}$ values.

The parameters found using the above method are used to determine the barrier properties of a laminate, as described below.

Barrier Properties

In addition to the properties of the core material, the barrier properties of the film determine the longevity of the panel, limited by permeation of air and moisture vapor through the high barrier envelope.

Moisture permeation rate, which is also known as MVTR or WVTR, is measured by two different methods:

1. Measurement by Systech Water vapor permeation analyzer 7000 (Figure 6 & Figure 7), according to ASTM F-1249 on flat film (as produced, without any mechanical stress).

Figure 6: Systech Water vapor analyzer 7000 (credit:www.systechillinois.com)

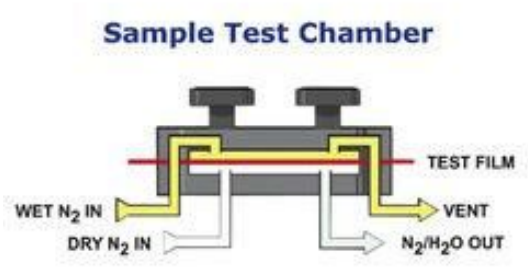

Figure 7: Schematic view of Systech Water vapor analyzer 7000 (credit:www.systechillinois.com)

The sample of flat barrier film to be tested is mounted in a two-compartment permeation cell. A constant water vapor pressure is maintained on one side of the sample to keep the cell at 90% relative humidity. On the other side (dry) the permeated water molecules through the sample are picked up by the extremely dry carrier gas (nitrogen N_2). The nitrogen then exits the cell and passes through P_2O_5 sensor. The amount of water vapor is measured by the sensor and the water transmission rate is calculated. The main drawback of this method is the fact that the measurement is performed on flat film without any of the mechanical stresses that occur when the film is converted to a panel. Figure 8 shows the test result of different Avery Dennison Hanita's laminate with MVTR as a function of measuring time on the Systech unit.

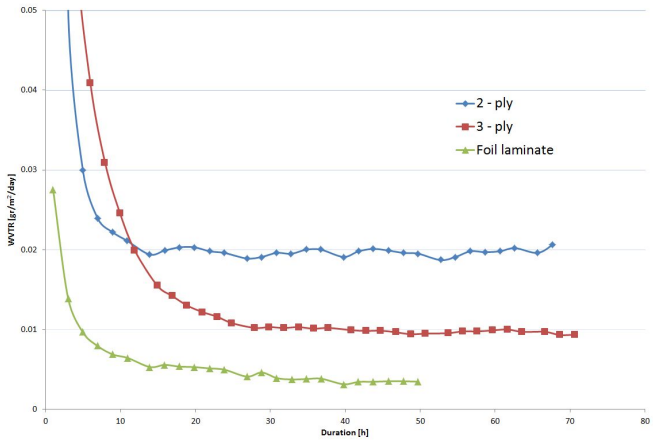

Figure 8: MVTR of Avery Dennison Hanita's laminates as function of measured time (Systech unit)

2. **The Water Intake (gravimetric) technique:** The procedure starts with the preparation of small FG panels (~15cm x 12cm) with desiccant inside using the inspected film. The panels are weighed using a micro-balance, and then held in a humidity oven at 40°C and 90%RH. Over the coming months, the panels are weighed once a week. The mass gain during this period is caused by the water molecules permeating and absorbed by the desiccant. Theoretically the water permeation rate is about 1000 faster than the permeation rate of air, therefore the contribution of the weight gain of air permeation is negligible. At the end of the test period, the WVTR of the panel (depending on the type of the envelope only) is calculated by dividing the

mass gain by test duration and the area of permeation.

Figure 9(a): small panel (~15cmx12cm) with glass fiber and desiccant (b): Weighing the panel with an analytic balance

The main advantages of the Water Intake (WI) technique over Systech unit testing its much more realistic values achieved due to testing at an application level. The results of WI tests made on three panels produced with standard Avery Dennison Hanita 3-ply laminate are presented in Figure 10 and Table 3.

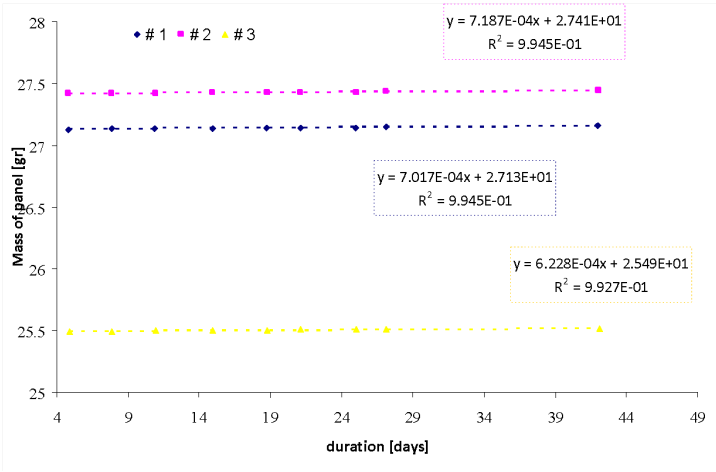

Figure 10: Weight of three panels produced with Avery Dennison Hanita 3-ply laminate as measured by the Water Intake test

Table 3: WI test results

#1		#2		#3	
A[cm²]	0.0403	A[cm²]	0.0416	A[cm2]	0.036
Δm [gr]	MVTR [gr/day m²]	Δm [gr]	MVTR [gr/day m²]	Δm [gr]	MVTR [gr/day m²]
$7.017x10^{-4}$	0.01741	$7.187x10^{-4}$	0.01728	$6.228x10^{-4}$	0.01730

As shown in Figure 1, each VIP FG panel contains a desiccant, usually calcium oxide. The amount of desiccant required is determined by the barrier properties of the film, and the temperature and relative humidity along the life time of the panel. When the desiccant is saturated, the pressure inside the panel starts to rise rapidly, causing a fast increase in thermal conductivity, and a loss of thermal insulation. Panels with fumed silica do not contain a desiccant, because the moisture molecules are absorbed efficiently by the core itself. Typically, the thermal conductivity of fumed silica increases due to moisture absorption at a rate of 0.5 mW/m·K per 1% of weight increase. [Ref. 2].

Gas (Air) permeation rate, which is also known as GTR:

The permeation rate of air through the envelope is measured using evacuated FG panels according to the following procedure:

1. Produce a 30cm x 30cm 3-seal bag using the sealing machine shown in Figure 11.

Figure 11: Heat sealing machine

2. Insert a FG core with a known dependency of thermal conductivity on pressure plus desiccant into the bag (after being baked at 120°C for a least one hour).

Figure 12: VIP before evacuation

3. Evacuate the panel to a pressure lower than 10^{-2}mbar, and seal the fourth edge (see Figure 12).

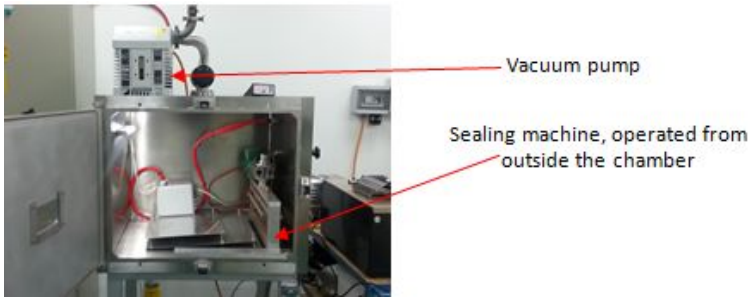

Figure 13: Vacuum chamber

4. After preparation, store panels at different temperature and relative humidity levels, and measure their thermal conductivity frequently using a LaserComp unit (see Figure 14), over a long period of time.

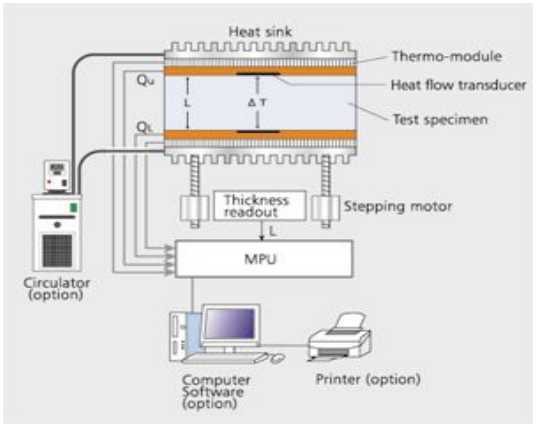

Figure 14: Thermal conductivity measurement device (credit: T Instruments LaserComp FOX314 www.tainstruments.com)

The thermal conductivity (λ) of a specimen is defined by the following equation:

$$\lambda = \frac{QU + QL}{2} \times \frac{L}{\Delta T} \qquad (3)$$

Where:

QU [W/m^2] is the output of the upper heat flux transducer, QL [W/m^2] is the output of the lower heat flux transducer, L is the thickness of the specimen, and ΔT is the temperature difference between the surfaces of the specimen.

Normally the temperature of the plates is 10°C and 35°C, unless otherwise required. In most cases, the panels are stored at a wide range of temperatures (4°C, 23°C, 40°C, 50°C/70RH, and 80°C). The thermal conductivity of each panel is measured over at least three months. Pressure is then calculated using the known TC vs. P from the measured values of thermal conductivity (as explained above), enabling a very accurate assessment of internal pressure increase along the storage time. In the final stage, the air permeability [cc(STP)/year m^2] of the laminate for a given temperature is calculated using the pressure increase rate and the panel dimensions (width, length, and thickness). This calculation will give the

amount of air (cc(stp)) permeating through the envelope of a 1m^2 panel in a year.

Figure 15 shows internal pressure as function of test duration for Avery Dennison Hanita 3-ply laminate at 50°C/70%RH.

The graph shows two different linear segments (a and b), each described by a trend line, whereby the slope represents the pressure increase rate.

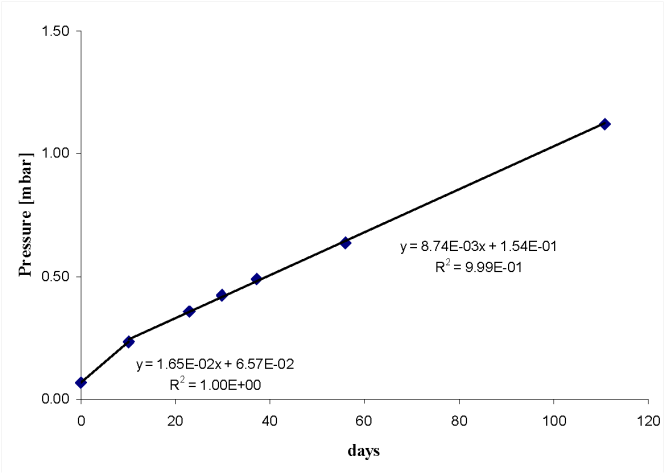

Figure 15: Internal pressure as a function of time for Avery Dennison Hanita 3-ply laminate stored at 50°C/70RH

It is clearly shown in Figure 15 that the pressure increase rate at the initial stage is larger by a factor of ~ 2. This phenomenon refers to outgassing, which contributes substantially to the pressure increase rate in the first few weeks after panel production. This initial period of time should be excluded from the permeability calculation. Outgassing occurs at all temperatures; its duration is longer at a lower storage temperature. At ambient, it may take about two months before the contribution of outgassing to the pressure increase rate becomes insignificant and can be ignored. The pressure increase rate should be determined solely by the steady state permeation rate of air through the envelope. In general the permeability should be calculated using only the pressure increase after steady state has been reached, and the outgassing effect negated. The origin of outgassing can be analyzed using an RGA (Residual Gas Analyzer), and is explained below.

Outgassing and steady state permeation

Evaluation after a short time period may lead to incorrect conclusions regarding the barrier properties of laminates, as presented in the following example. Figure 16 shows thermal conductivity increase at 23°C as a function of time for two VIPs with identical dimensions and different envelope laminates.

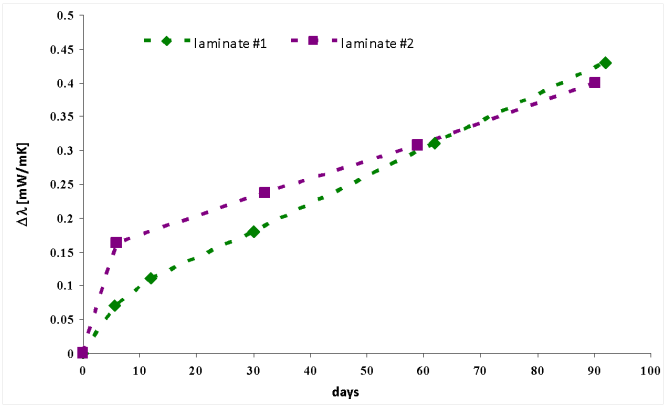

Figure 16: Thermal conductivity increase of two FG VIPs with same dimensions but different envelopes

Over the first few weeks, the thermal conductivity of FG panels changes much faster due to air outgassing from the laminates, with the amount of outgassing varying for each laminate. If the barrier properties of the laminates were to be determined after 30 days, it would wrongly be concluded (presented in Figure 17) that laminate #1 has better barrier properties than laminate #2.

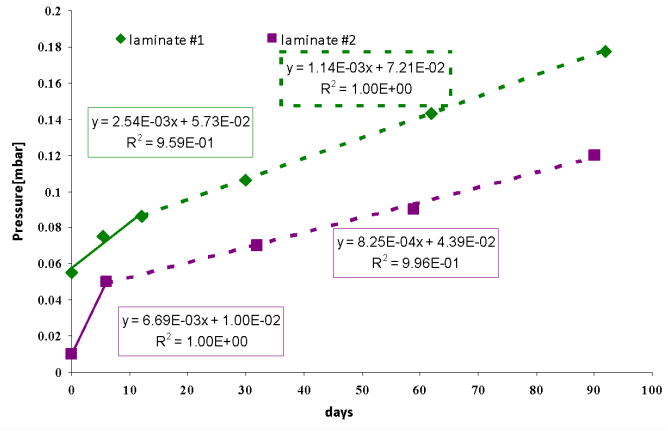

Figure 17: Internal pressure of 2 VIPs with same dimensions and different envelopes

From Figure 17 it can be clearly seen that the outgassing in laminate #2 is much greater than the outgassing in laminate #1 (by factor of ~2.6), meaning that in the initial stage, the internal pressure increase of laminate #2 is larger, and can mistakenly be assumed to be an inferior gas barrier compared to laminate #1. As the evaluation time is extended and steady state (the point at which pressure increase is constant in time) is reached after few weeks, it can be clearly seen from Figure 17 that laminate #2 actually has better barrier to gas than laminate #1 by a factor of ~1.5.

Dependence of permeability on storage temperature

It was found that the Arrhenius equation (Equation 4) can be used to model the dependence of permeability on temperature.

$$P(T) = P_0 \exp\left(-\frac{E_A}{RT}\right) \quad (4)$$

Where:

P_0 is permeability at infinite temperature, $E_A\,[J]$ is activation energy and $R = 8.314\frac{J}{mol\,K}$ is ideal gas constant.

Example: Test results of the permeability of a Avery Dennison Hanita 3-ply laminate measured at various temperatures:

T [°C]	T [K]	1000/T [K-1]	P [cc(STP)/year m²]	Ln(P)
4	277	3.61	2.6	0.96
23	295	3.39	6.75	1.91
40	313	3.19	20.7	3.03
50	323	3.1	28.1	3.34
80	353	2.83	80	4.38

Table 4: Permeability of Avery Dennison Hanita 3-ply laminate measured at various temperatures

A plot of Ln(P) as a function of 1000/T is presented in Figure 18.

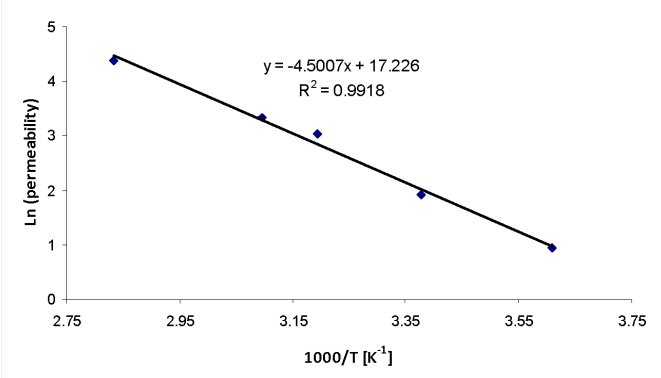

Figure 18: Plot of Ln (Permeability) as a function of 1000/T

The Activation energy E_A and P_0 are calculated from equation of the trend line:

$$E_A = -4.5007 \cdot 1000 \cdot 8.314 = 37.4 \times 10^3\,J$$

$$P_0 = \exp(17.226) = 3.03 \times 10^7\ \text{cc(STP)/year m}^2$$

Figure 19 describes the permeability of Avery Dennison Hanita 3-ply laminate over a wide range of temperatures.

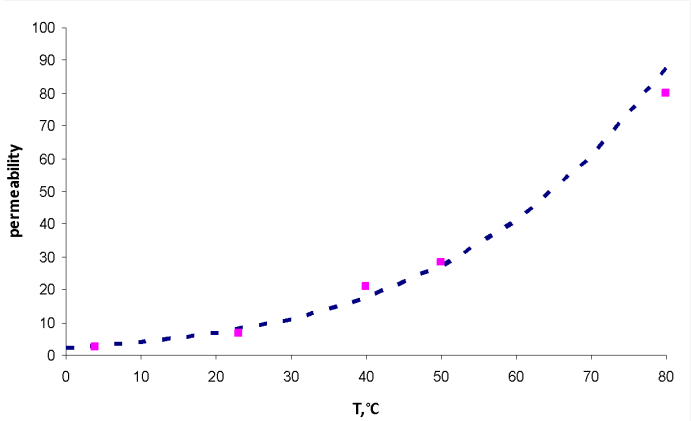

Figure 19: Permeability of Avery Dennison Hanita 3-ply laminate as function of temperature. The blue dashed line is a plot of Equation 4 and the pink dots are the measured values.

Normally, all GTR values at Avery Dennison Hanita are measured under three conditions:

1. Low temperature measurement: room temperature (~23°C),
2. Intermediate temperature and high relative humidity: 50°C/70RH (humidity is added because the barrier properties of some laminates are affected by the amount of water absorbed),
3. High temperature measurements: 80°C. Some GTR tests are performed at higher temperatures of up to 150°C.

ALT - Accelerated Life Testing

During accelerated life testing, VIPs are stored in a climate oven with periodically cycling conditions. During the test the panels are exposed to extreme conditions**:**

1. 2.5h at -30°C, dry.
2. 5.5h at 80°C and 65% relative humidity.
3. Back to 1st condition.

Each cycle lasts nine days, and the test consists of 4-5 cycles, with thermal conductivity measured after each cycle.

The results of three ALT tests on several types of laminates are presented in Figure 20.

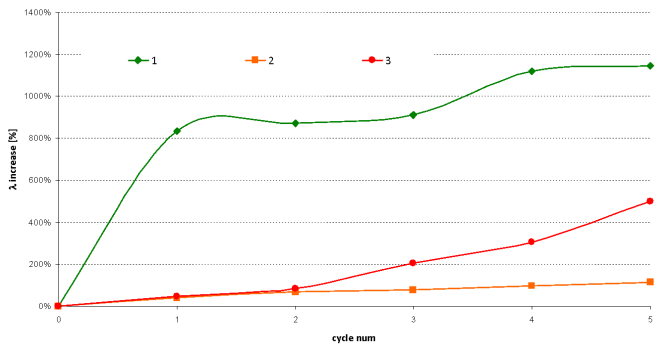

Figure 20: Thermal conductivity increase of three different laminates as function of cycle number of ALT tests

This test enables a comparison between different laminates. For example, Figure 20 leads to the following conclusions:

1. Laminate 1 fails almost immediately: the failure mechanism is probably due to barrier degradation caused by high temperature and RH. This result correlates with high permeability at 50°C/70RH.
2. Laminate 3 fails after two cycles: such a failure is typically due to degradation of the adhesive layer resulting from exposure to the combination of elevated temperature and high levels of humidity.
3. Laminate 2 stands the test quite well: thermal conductivity increase is due gas permeation only, and all the laminate films and the adhesive layer survived the test well.

Effective thermal conductivity of a VIP

When the VIP is used in its application, the effective thermal conductivity λeff should be taken into account in the performance calculations. λeff combines the two relevant mechanisms of heat conductance by the panels:

a) Heat transmittance via the core material (which changes with time), as measured in the center of the panel (λcop) using a LaserComp, and b) Heat transmittance via the envelope.

The phenomenon of heat conducted by the envelope is called thermal bridge. The effective thermal conductivity can be calculated by the following equation [Ref. 3]:

$$\lambda eff = \lambda cop + \Psi * d * \frac{A}{V} \quad (5)$$

The linear heat conducted by the envelope Ψ is highly dependent on the thickness of the Aluminium used in the laminate. With Al foil based laminates, the thickness of the Al layers is 50 to 100 times thicker than with metallized films, and the effect of the thermal bridge on the overall heat conductance from the warm side to the cold side of the panel is considerable. In many cases when Al foil is used, the heat flow through the envelope (the thermal bridge, represented by the second part of the equation above) can be substantially larger than that through the core (represented by λcop), causing a substantial reduction in the insulation performance of the panel.

The pictures below demonstrate the differences in the thermal bridge when using a two-sides-Al foil envelope for the VIP, a hybrid envelope (one side metallized and one side Al foil), or a two-sides-metallized envelope:

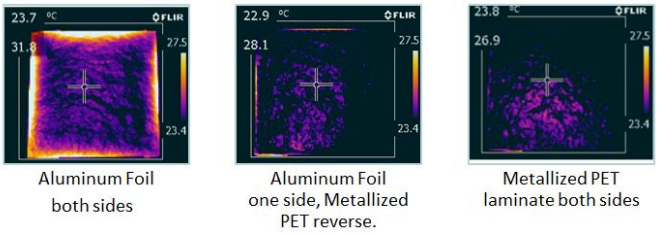

Figure 21: Thermal imaging of the thermal bridge effect

The above picture shows that thermal conductivity is higher around the panel edges, which can cause condensation when a VIP is installed inside a refrigerator.

RGA – Residual Gas Analyzer

A residual gas analyzer (RGA) is a small mass spectrometer (Figure 22), typically designed for process control and contamination monitoring in vacuum systems.

Figure 22 (a)

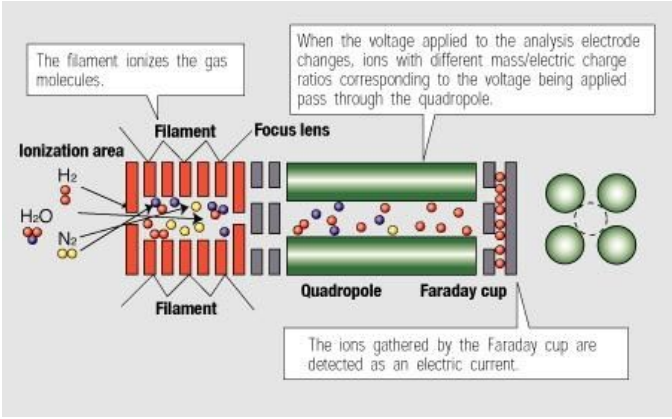

Figure 22(a): RGA unit, (b): schematic view of RGA (credit:www.mksinst.com)

Operating principle:

1. A small amount of gas is inserted into RGA inlet, which is continuously evacuated by a turbo-molecular pump.
2. The gas molecules entering pass through a filament and are ionized.
3. The ionized gas molecules pass through a quadrupole which separates each ion (type of gas) according to its molecular mass.
4. The ions are gathered by a detector (detected as an electric current) and are presented on a chart – partial pressure as a function of molecular mass. Avery Dennison Hanita's RGA can detect molecules with an atomic weight of up to 200amu.

Setup:

Figure 23 shows setup for analyzing the residual solvents in the film:

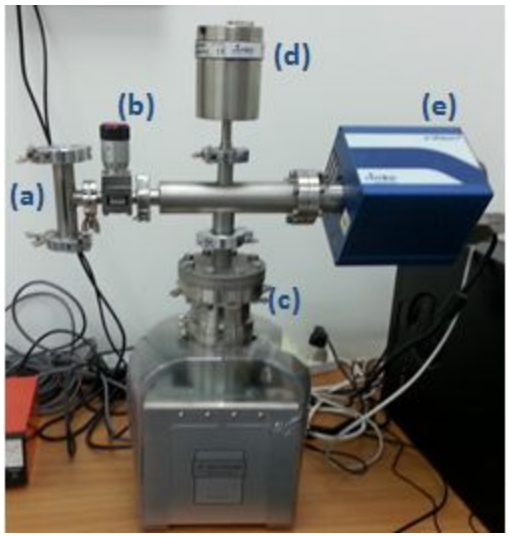

Figure 23: RGA setup

a) Small cavity with inspected film and small bag of CaO desiccant (optional).
b) Vacuum needle valve.
c) Vacuum turbo molecular pump.
d) Vacuum gauge for monitoring the amount of gas entering the RGA unit.
e) RGA unit with connection to computer.

Test procedure:

1. The whole system without the inspected film (closed vacuum valve) is pumped down to a low pressure of 10^{-4}mbar. The system then records the background partial pressure of all molecular weights from 1amu to 200amu.
2. The cavity with the film inside is stored at 80°C for 3h to encourage outgassing from the film to the cavity.
3. The cavity is then connected to the RGA setup.
4. A small amount of gas from (a) is inserted into the RGA by opening the vacuum valve. The scan is recorded as a film test.
5. At the final stage, the difference between the two scans is calculated for each molecular weight.
6. The differences between the two scans for each molecular weight are the measured partial pressures of the molecules with the different molecular weight (the partial pressures inside the RGA SYSTEM). Figure 24 shows such an RGA scan.

Typical RGA analysis:

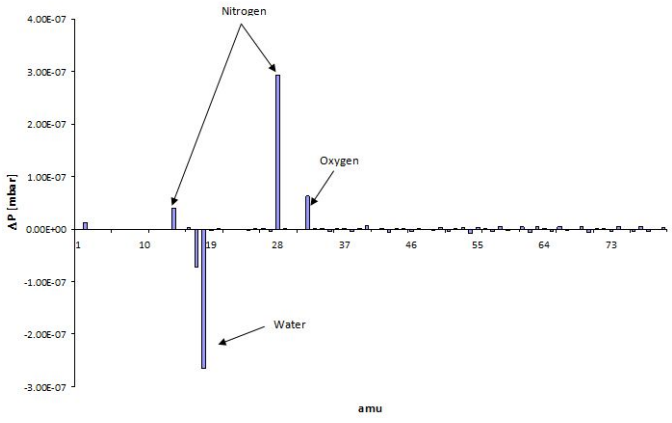

Figure 24: RGA scan

The spectrum above in Figure 24 presents typical results for an RGA analysis, revealing that no residual solvents exist in the film, as shown by the absence of peaks at high amu. The only gas

detected was air (nitrogen + oxygen); the amount of water decreases below the background level due to absorption by desiccants.

It is important to mention that this measurement does not provide information on the absolute value of the partial pressure of the different molecules inside the tested compartment. It does, however, provide very accurate information about the relative concentration of the different molecules in the tested compartment.

5. Mechanical Properties

All mechanical properties of the laminates are tested using a Lloyd Tensiometer Unit, as shown in Figure 25.

Figure 25: Lloyd Tensiometer

The following mechanical properties are normally tested:

1. Lamination strength – tested on each production roll at each lamination stage.
2. Tensile strength and elongation at break point - tested on final product, similar to ASTM D882.
3. Puncture resistance- tested on final product, similar to FTMS 101C 2065 or ASTM D4833M.
4. Sealing strength - tested on final product, similar ASTM F88M.

6 Flame retardant (FR) properties - DIN 4102

The flame retardant properties of a laminate are determined by exposing a small VIP to fire for a period of time, as dictated by the standard, and measuring the height that the flame reaches. For example, to achieve rating B2 in DIN 4102, the flame should not pass a certain distance over a certain period of time.

Figure 26: FR measurement setup

Conclusion

The above methodology for testing VIPs is successfully used by Avery Dennison Hanita to assess and evaluate the performance of Avery Dennison Hanita-manufactured, competitor and customer-provided laminates, cores, and panels. Although many international film testing standards are commonly used as a basis, the VIP industry is still united in its need to foster specific standards for evaluation of the VIP. Avery Dennison Hanita's developments pioneer this process, and together with customers and organizations such as VIPA and CEN, are on the threshold of testing standardization.

References

[1] G. Reichenauer*, U. Heinemann, H. P. Ebert, Relationship between pore size and the gas pressure dependence of the gaseous thermal conductivity, Colloids and Surfaces A: Physicochemical and Engineering Aspects, 300 (2007) 204-210.

[2] A. Beck, F. Oswald, M. Binder, Influence of water content on the thermal conductivity of vacuum panels with fumed silica kernels, in: A. Beck, et al. (Eds.), Proceedings of the 8th International Vacuum Insulation Symposium, ZAE Bayern/UniWue, Wurzburg, September 18–19, 2007, (2007), pp. 1–9.

[3] Effective thermal conductivity of vacuum insulation panels, K. Ghazi Wakili, R. Bundi, and B. Binder, Laboratory for Applied Physics in Building, Swiss Federal Laboratories for Materials Testing and Research (EMPA),CH-8600 Duebendorf, Switzerland.