## **Barrier Development and Evaluation Methodology**

# D.S. Musgrave<sup>1</sup> <sup>1</sup>Thermal Visions, Inc., Granville, USA

#### 1. Introduction:

Vacuum insulation is now a rapidly expanding market and technical advances are being made quickly. One of the most important components of vacuum insulation is the barrier film. The function of the barrier and associated heat seals is to slow the increase in panel internal pressure which is directly related to the panel's thermal performance. A better barrier opens up markets that require slower decrease in performance (longer life) and potentially high temperature applications. Over three years were spent developing a more advanced barrier film and it has been used on hundreds of thousands of panels. From this development and use experience, many of the important things learned are presented in this paper.

2. Variables That Affect Performance Versus Time:

The temperature and humidity on each side of the panel versus time during the transportation, storage, product manufacture and end use affect the atmospheric gas diffusion rate and the water vapor transmission rate. It should also be remembered that the diffusion through the heat seals should be determined and incorporated into the calculations. The panel dimensions need to be considered. The panel dimensions affect the seal length to panel area. The most important panel dimension is the panel thickness. The change in panel performance is linearly related to the panel thickness. A panel with twice the thickness will have half the rate of decrease in performance since the diffusing gases will go into twice the volume so the pressure rise is half. The last item is the potential damage to the barrier that can occur at any stage of the panel's life from panel manufacture through the end of the panel's useful life.

### 3. Accelerated Life Testing:

Barriers have been for sometime at the performance where producing actual panels and testing them versus time to determine the degradation rate requires very long time and therefore is not very useful in barrier development. It might take half a year or more before being able to get some idea if a new barrier design is beneficial. This approach was particularly a problem in the new barrier development since the research was to develop a barrier that was many times superior to the existing barriers and would take many times longer.

a. Mocon Oxygen Transmission Testing:

Years ago, MOCON developed a Super OX-TRAN test that was stated to test down to  $0.0005 \text{ cc/m}^2$ -day-bar. One of the problems is that virtually all the barrier films for vacuum insulation measure below the minimum that the MOCON test

can measure. Thus it is not able to give any feedback to say if a new barrier design is improved. Some of the other issues are:

- i. MOCON measures only oxygen, other gases such as nitrogen, water vapor, and out gassing from the barrier or core is not measured.
- ii. The Super OX-TRAN test is a steady state test. Thus it will miss transient effects that might occur immediately after evacuation. It also means that there is an inherent fixed sensitivity. Running the test longer does not increase sensitivity.
- iii. The Super OX-TRAN test also does not measure the diffusion through the panel heat seals.
- b. Accelerated High Temperature and High Humidity Testing:

Some organizations have used a high temperature (85° C) and high humidity (85% RH) test of full thickness test panels. If the actual application is at high temperature and humidity, this test may be a good test but most vacuum panels are used at low temperature and many are used in refrigerator or freezer walls where the relative humidity is, by design, very low. In most cases it is best to not think in terms of relative humidity but to think in terms of vapor pressure. Relative humidity is the water vapor weight in the air divided by the maximum amount the air could hold. This water vapor weight changes drastically with temperature and therefore also the vapor pressure changes drastically with temperature. The water vapor pressure at 85° C and 85% relative humidity is about 49400 Pa. The water vapor pressure in the wall of a typical refrigerator is less than 94 Pa. This is over 500 times higher. In addition to this, the barrier diffusion rate will also increase with temperature. The exact rate is unknown but it may double for every 10° C. Between 25° C and 85° C there are six 10° increments so there might be a 64 times increase in the diffusion rate. Multiplying both together results in about 33,000 times increase. One week of this condition could be equivalent to about 634 years in a refrigerator wall. Therefore, this test was not very beneficial in the barrier film development.

c. Minimum Volume Accelerated Barrier Test:

For the above reasons, the researchers began developing an accelerated barrier test. It was not desired to accelerate the diffusion or out gassing due to higher temperature or higher external pressure since the diffusion and out gassing mechanisms might change. The approach chosen was to utilize an extremely small internal volume (very thin panel) incorporating the panel's normal surface area and seal length. It was also decided not to try to measure the gas diffusion rate but to directly measure the panel thermal performance change (by ASTM standard conductivity testing) since this is the actual property of interest. This test captures all the degradation mechanisms that result in decrease in panel performance. It also has one more large advantage. As the test proceeds, the effect of any out gassing can be seen and the longer the test runs the more accurate the prediction. A few months of testing can predict the panel performance 10 or 15 years in the future and longer testing can provide an even longer forecast. This test allowed the evaluation of many different barrier designs. Without this test, the development would not have been possible.

4. Additional Development Tools:

To rapidly investigate the many barrier design options, the barrier diffusion was modeled using computer Finite Element Analysis (FEA). With this technique it is not possible to predict the exact diffusion rate but it provided valuable insight.

## 5. Additional Considerations:

Currently there are two major approaches to barrier films. The barrier can contain an aluminum foil layer or the barrier contains no aluminum foil.

a. Barrier with Aluminum Foil:

The aluminum foil is an outstanding barrier but it can have pin holes created in the manufacturing rolling process or pin holes can be created sometimes when the barrier is folded over to tape the seal edges or other manipulation. The pin holes can lead to a catastrophic loss of vacuum but most barriers incorporating aluminum foil also incorporate a metalized film laminated to the foil. This mitigates most of the issue with the pin holes since it is no longer a through path. However there is still one major disadvantage to barriers containing aluminum foil. Heat flows along the aluminum foil and travels around the edge of the panel to the other side. This heat effectively by-passes the vacuum insulation. On very large panels this reduction in panel performance could be 20 to 25%. However on narrow or small panels the performance reduction of the vacuum panel can be about 30% to 55% or even more in extreme situations.

b. Barrier with No Aluminum Foil:

Barriers with no aluminum foil have a difficult task to create an outstanding barrier. Typically multiple vacuum metalized layers are laminated together. Sometimes chemical barrier materials are added. It is difficult but not impossible to arrive at a design that has outstanding performance and because there is no foil layer, there is no edge heat transfer that would reduce the panel performance.

6. Panel Failure:

During the final evaluation and real world trial use, it is important to investigate any panel failures and determine where, why, and how the failure occurred. Once the cause is understood, a design to mitigate the problem can be developed and tested.

a. Helium Leak Testing:

The first objective is to find the failure location on the panel. Often the panel is pressurized with helium and a "sniffer" is slowly moved around the panel until the helium is found. The "sniffer" can be based on a mass spectrometer or at less cost a detector based on the substantially different thermal conductivity of helium is used. It is a time consuming process to find the leak or in some cases multiple leaks. The areas of the leak are circled with a marker for further investigation.

b. Backlight Dark Room Approach:

An alternate approach to finding the leaks is to remove the panel core and light the interior of the barrier pouch while in a dark room. A pin hole can sometimes be spotted. This approach is not as sensitive as the helium leak test but it is usually faster and provides the location of the leak.

c. Optical Microscope:

Once the failure area has been found, an optical microscope can be used to try to better understand the failure. Fig. 1 is an example of the optical microscope approach. The optical microscope was of marginal benefit in the research.



Fig. 1. Optical microscope (back lit) of a barrier where there was a physical hole.

d. Scanning Electron Microscope (SEM):

The scanning electron microscope can be the most revealing method of understanding a failure. It can be used at very high magnification but most often it was used at magnifications about the same as could be achieved with an optical microscope. The great advantage of the scanning electron microscope was the very large depth of field (everything was in clear focus). Fig. 2 is an example of a failure location. The detail is so great that the researcher can start to build an understanding of the situation that must have caused the failure. In this case you can see the laminate layers and make assumptions as to which layers failed first.



Fig. 2 is a scanning electron microscope picture of a failure.

Vacuum panel barriers can fail for many reasons. The most common reason is the barrier comes into contact with something sharp such as the corner of a steel cabinet. Generally vacuum insulation barriers should be viewed as fragile. Anything sharp will cause failure. Other failures can be caused by high stress areas such as corners and folds of the barrier. Still other panels may fail caused by items that are not really barrier failures. Moisture still left in the panel can cause reduced performance. The panel may be out of the acceptable dimensions. The panel may be damaged in shipment to the customer.

7. Conclusions:

The technology to produce improved vacuum insulation barriers is increasing every day. This author expects that barrier performance will significantly improve over the next ten years. Also the abuse resistance of the barriers will improve. The better barriers will result in opening new markets and expanding existing markets. The industry is at a very steep part of the sales and technology curve and barrier technology will lead the way to success.